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CCP-TP-187

Revision 1

CCP Sample Preparation for Semivolatile Organic Compounds

EFFECTIVE DATE: 11/18/2008

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PRINTED NAME

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 delete compounds that are not needed in the spiking solutions for Waste Isolation Pilot Plant (WIPP) analyses.

1. ABSTRACT

This Central Characterization Project (CCP) procedure describes the procedures for preparing samples for analysis of semivolatile organic compounds by solvent extraction. This method also includes procedures for extract cleanup to remove interfering contaminants or radionuclides prior to analysis. Steps are included in this procedure for preparing extracts for radiological screening.

2. APPLICABILITY

This method is applicable to samples collected for characterization per the requirements of the Waste Isolation Pilot Plant (WIPP) Waste Analysis Plan (WAP). This method implements SW-846 Method 3550B, "Ultrasonic Extraction."

The target analyte list is found in the determinative method CCP-TP-185, *CCP Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry*."

This procedure functions as an 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 analyzed are radioactive solids. Reduced sample sizes from those recommended by EPA SW-846 (e.g., 5 g vs. 30 g for solids) are used as the best compromise between low method detection levels and minimal radioactivity exposure to laboratory personnel (ALARA).

This method uses ultrasonic extraction. An appropriate solvent or solvent mixture is added to a measured amount of sample and sonicated to extract the organic compounds. A gentle stream of nitrogen is used to concentrate the extracts.

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.”

4.1.5 Handle standards containing carcinogens per MCP-2703, “Carcinogens.”

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 Ergonomic Hazardsl

4.3.1 Take periodic breaks when performing repetitive tasks.

4.4 Waste Disposition and Pollution Prevention

4.4.1 Manage all waste generated per ACLP-0.40, “Analytical Laboratories Waste Management.”

5. APPARATUS AND REAGENTS

5.1 Apparatus

5.1.1 Analytical balance, capable of weighing 0–200 g with 0.01 g readability

5.1.2 Oven, Thermolyne 62700 or equivalent

5.1.3 Solvent evaporation apparatus (custom-made), consisting of a nitrogen gas manifold with sample tube holder

5.1.4 Ultrasonic processor, a 3/4 or 1/2-inch standard horn-type sonicator equipped with a titanium or equivalent tip and having a minimum power wattage of 300 watts with pulsing capability

5.1.5 Vortex mixer, Barnstead/Thermolyne Maxi Mix II or equivalent

5.1.6 Beaker, glass, various sizes

5.1.7 Bottle, glass, various sizes, with Teflon-lined screw cap

5.1.8 Bottle, wash, Teflon FEP, various sizes

5.1.9 Concentrator tube, glass

5.1.10 Cylinder, graduated, glass or Teflon, various sizes

5.1.11 Desiccator, glass

5.1.12 Flask, Erlenmeyer, glass, various sizes

5.1.13 Flask, volumetric, glass, class A, various sizes with ground glass or Teflon stoppers

5.1.14 Filter paper, Whatman No. 41 or equivalent, various sizes

5.1.15 Funnel, glass, narrow stemmed, various sizes

5.1.16 Pipet, disposable Pasteur, glass, or equivalent

5.1.17 Pipet, volumetric, various sizes

5.1.18 Planchette, stainless steel

5.1.19 Separatory funnel, Teflon or glass, various sizes

5.1.20 Spatula, stainless steel or Teflon

5.1.21 Stirring rod, glass

5.1.22 Syringe, glass with luer-lock, various sizes

5.1.23 Syringe, glass, various sizes

5.1.24 Transfer container for planchette

5.1.25 Vial, autosampler, glass, 2-ml, with Teflon-lined screw cap or equivalent

5.1.26 Vial, scintillation, glass, 20-mL, with Teflon-lined screw cap or equivalent

5.1.27 Syringe filter, 0.22-µm, nylon

5.2 Reagents

Use Analytical Reagent Grade chemicals and ASTM Type II conductivity or better water for preparation of all reagents, unless otherwise specified. Unless

otherwise indicated, store reagents in glass to prevent leaching contaminants from plastic containers.

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

Use all purchased standards within the manufacturer-specified shelf life, or within one year of the supplier's vial being opened, whichever occurs first. Use all prepared standards within six months, or whenever routine QC indicates a problem.

5.2.1 Methanol (CH_3OH), pesticide quality or equivalent

5.2.2 Methylene chloride (CH_2Cl_2), pesticide quality or equivalent

5.2.3 Nitrogen gas, ultra high purity (UHP) or better

5.2.4 Sand, organic-free. Purchase sea sand, Fisher catalog # S25 or equivalent. Bake the sand for a minimum of four hours at 400 °C, cool in a desiccator, and store in a tightly closed glass container.

5.2.5 Sodium sulfate (Na_2SO_4), anhydrous, granular. Purify by heating to 400° C for four hours in a shallow tray. Cool in a desiccator and store in glass bottle with a Teflon-lined screw cap, or preclean per Step 7.2.2.

5.2.6 Stock Acid Matrix Spiking Solution. Purchase a certified, commercially prepared solution of pentachlorophenol and phenol at 1000–5000 µg/mL in methylene chloride or methanol that is second-source to CCP-TP-185 calibration standards.

5.2.7 Stock Base/Neutral Matrix Spiking Solution. Purchase a certified, commercially prepared solution of 2,4-dinitrotoluene and 1,4-dichlorobenzene at 1000–5000 µg/mL in methylene chloride or methanol that is second-source to CCP-TP-185 calibration standards.

5.2.8 Stock Matrix Spiking Solution. Purchase a certified, commercially prepared solution of pentachlorophenol, phenol, 2,4-dinitrotoluene, and 1,4-dichlorobenzene at 1000– 5000 µg/mL in methylene chloride or methanol that is second-source to the CCP-TP-185 calibration standards.

5.2.9 Stock Acid Surrogate Spiking Solution. Purchase a certified, commercially prepared solution of phenol-d_5, 2,4,6-tribromophenol, and 2-fluorophenol at 1000–5000 µg/mL in methylene chloride or methanol.

5.2.10 Stock Base/Neutral Surrogate Spiking Solution. Purchase a certified, commercially prepared solution of 2-fluorobiphenyl, nitrobenzene-d_5,

and terphenyl-d_{14} at 1000–5000 µg/mL in methylene chloride or methanol.

5.2.11 Stock Surrogate Spiking Solution. Purchase a certified, commercially prepared solution of 2-fluorobiphenyl, nitrobenzene-d_5, terphenyl-d_{14}, phenol-d_5, 2,4,6-tribromophenol, and 2-fluorophenol at 1000–5000 µg/mL in methylene chloride or methanol.

5.2.12 Acid Matrix Spiking Solution. Prepare a 50 µg/mL solution in methanol by diluting the Stock Acid Matrix Spiking Solution (Item 5.2.6).

5.2.13 Base/Neutral Matrix Spiking Solution. Prepare a 25 µg/mL solution in methanol by diluting the Stock Base/Neutral Matrix Spiking Solution (Item 5.2.7).

5.2.14 Matrix Spiking Solution. Prepare a solution in methanol by diluting the Stock Matrix Spiking Solution (Item 5.2.8) and (if needed) the Stock Acid Matrix Spiking Solution (Item 5.2.6) so that the spiking solution final concentrations are 50 µg/mL for the acids and 25 µg/mL for the base-neutral compounds.

5.2.15 Acid Surrogate Spiking Solution. Prepare a 50 µg/mL solution in methanol by diluting the Stock Acid Surrogate Spiking Solution (Item 5.2.9).

5.2.16 Base/Neutral Surrogate Spiking Solution. Prepare a 25 µg/mL solution in methanol by diluting the Stock Base/Neutral Surrogate Spiking Solution (Item 5.2.10).

5.2.17 Surrogate Spiking Solution. Prepare a solution in methanol by diluting the Stock Surrogate Spiking Solution (Item 5.2.11) and (if needed) the Stock Acid Surrogate Spiking Solution (Item 5.2.9) so that the spiking solution final concentrations are 50 µg/mL for the acids and 25 µg/mL for the base-neutral compounds.

5.2.18 LCS Spiking Solutions. Use the Acid Matrix Spiking Solution (Item 5.2.12) and the Base/Neutral Matrix Spiking Solution (Item 5.2.13) or the Matrix Spiking Solution (Item 5.2.14) as the spike solutions for the laboratory control sample (LCS).

5.2.19 MDL and P&A Standards

NOTE: *CCP-TP-185 specifies required numbers of MDL and P&A standards.*

5.2.19.1 For each standard required, add ~2 mL of appropriate solvent (e.g., methanol) to a 20-mL vial. Add 25 µL of a 200 µg/mL stock standard solution containing all target analytes and surrogates (e.g., CCP-TP-185 calibration standards) and cap the vial. Process 5 g aliquots of organic-free sand through the extraction procedure (Step 7.1) as samples, using the prepared spiking vials in place of those specified in Step 7.1.2.

5.2.20 Titanium chloride ($TiCl_3$), 5% solution. Purchase commercially or prepare by 4-fold dilution of a 20% solution with water.

5.2.21 Oxalic acid, 1 M. Prepare by dissolving 12.6 grams of oxalic acid dihydrate ($HO_2CCO_2H{\cdot}2H_2O$) in 100 mL water. Apply heat as necessary to aid dissolution and cool to room temperature before use.

6. SAMPLE HANDLING

6.1 Take care to avoid sample contamination from laboratory sources.

6.1.1 Use glass or Teflon containers with Teflon-lined screw-caps.

NOTE: *Use of other plastic containers or lids can cause sample contamination from phthalate esters and other hydrocarbons present in plastic.*

CAUTION

Allowing the sample or extract to contact gloves may cause contamination of the sample.

6.1.2 Ensure gloves do not contact sample or extract when filling sample containers.

6.2 Store all samples at 4 °C.

NOTE: *SW-846 allows a range of ± 2 °C around the specified storage temperature.*

6.3 Extract all samples within 14 days of sample collection.

6.4 Store extracts at ≤-10 °C.

6.5 Prepare 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 by the laboratory within 14 days of validated time of sample receipt (VTSR) of the first sample in the batch.

7. PROCEDURES

If multiple groups of samples are prepared within an analytical batch, prepare an LB with each group of samples. Refer to Section 8, Quality Control Requirements, for more information.

NOTE: *Steps are performed sequentially for each sample. Within an analytical batch, individual samples may be at different steps in the process.*

7.1 Sonication Extraction in a Hood or Glove Box

NOTE: *Steps 7.1.1 through 7.1.4 may be performed simultaneously or at any time as required by the process and determined by the analyst.*

7.1.1 IF sonicator power supply is manually tunable,
THEN review the maintenance log to verify that sonicator and horn have been tuned according to manufacturer instructions.

7.1.2 Prepare a labeled 20-mL vial containing spiking solutions for each sample (including LB, LCS, MS and MSD).

7.1.2.1 Add surrogate spiking solutions to all 20-mL vials.

7.1.2.1.1 Add 1.0 mL of Surrogate Spiking Solution (Item 5.2.17) OR add 1.0 mL of Acid Surrogate Spiking Solution (Item 5.2.15) and 1.0 mL of Base/Neutral Surrogate Spiking Solution (Item 5.2.16).

7.1.2.2 Add matrix spiking solutions to the 20-mL vials prepared for the LCS, MS and MSD.

7.1.2.2.1 Add 1.0 mL of Matrix Spiking Solution (Item 5.2.14) OR add 1.0 mL of the Acid Matrix Spiking Solution (Item 5.2.12) and 1.0 mL of the Base/Neutral Matrix Spiking Solution (Item 5.2.13).

7.1.3 IF working in a CA,
THEN transfer a sufficient amount of the appropriate matrix material (Item 5.2.4) into a small container to use for the LCS and LB.

7.1.4 Label a 125-mL glass bottle for each sample (including LB, LCS, MS and MSD) to use as an extract collection vessel.

7.1.5 IF working in a CA,
THEN transfer the spiking solution vials, sand, sample containers, 125-mL collection vessels, and any needed reagents or apparatus to the workplace specified by the RWP and hazard index (see Section 4.2).

7.1.6 Prepare the sample for extraction, as appropriate for the sample matrix.

7.1.6.1 Discard any foreign objects as directed by supervision.

7.1.6.2 Mix the sample thoroughly.

7.1.6.3 Consult with supervision to determine if particle size reduction is necessary.

7.1.6.3.1 Grind or otherwise subdivide dry waste amenable to grinding so that the fragments appear to be less than 1 mm in size.

7.1.6.3.2 Cut, shred, or otherwise reduce size of gummy, fibrous, or oily materials not amenable to grinding in order to allow mixing and maximum exposure of the sample surfaces during the extraction.

7.1.7 Process all samples in the analytical batch as follows.

7.1.7.1 Weigh an aliquot into a 100-mL glass beaker.

NOTE: *The sample size may vary depending on the sample matrix, sample availability, and the sample radioactivity. Normally 5 g of sample are used.*

7.1.7.1.1 IF aliquoting the LB or LCS,
THEN weigh organic-free sand to the size of the associated samples.

7.1.7.1.2 Weigh a 5 g aliquot for samples, MS, or MSD.

7.1.7.1.3 Record the weight to the nearest 0.01 g.

7.1.7.2 IF the sample is nonporous or wet (gummy or clay type) and does not have a free-flowing sandy texture,
THEN add and mix in a sufficient amount of anhydrous Na_2SO_4 (Item 5.2.5) to make the sample free-flowing.

7.1.7.3 Add the spiking solution in the labeled vial to the corresponding sample aliquot in the beaker.

7.1.7.4 Add 10–20 mL methylene chloride to the spiking solution vial, swirl, and add the methylene chloride to the sample beaker.

7.1.7.5 Place the disrupter horn in the sample beaker such that the bottom surface of the tip is just above the sediment layer.

7.1.7.6 Adjust the sonicator setting to achieve very active mixing of the sample by pulsed energy.

NOTE: *Output control affects agitation of the beaker contents; percent duty cycle affects agitation of the sample.*

7.1.7.7 Sonicate the sample for three minutes.

7.1.7.8 Decant the methylene chloride extract into the labeled collection bottle, retaining the sample in the beaker.

7.1.7.9 Repeat the extraction Steps 7.1.7.4 through 7.1.7.8 two more times using fresh methylene chloride.

7.1.7.10 Discard the solid sample residue and the beaker.

7.1.7.11 Clean the disrupter horn with methylene chloride.

7.1.8 IF this extraction is performed in a glove box,
THEN contact an RCT to determine if the extracts are within RWP limits.

7.1.8.1 IF the radiation level is within RWP limits
AND with RCT concurrence,
THEN transfer the extracts to a radiological hood and GO TO Section 7.2.

7.1.8.2 IF the radiation level is NOT within RWP limits,
THEN perform one or more of the following steps (listed in order of preference), repeating as necessary, until the radiation level of the extract is reduced to within RWP limits.

7.1.8.2.1 Clean the exterior surfaces of the bottle.

7.1.8.2.2 Transfer the extract to a clean bottle.

NOTE: *Contamination on the outside of the bottle can make it impossible to accurately survey the extract.*

7.1.8.3 IF the extract radiation level still can NOT be reduced to within RWP limits,
THEN obtain proper RWP coverage before proceeding with the extraction process.

7.1.9 IF it is desirable to further reduce the extract radiation level before further processing of the extract occurs,
THEN refrigerate the extract overnight at 4 ± 2 °C.

7.2 Solvent Evaporation by Nitrogen Blow Down

7.2.1 Calibrate a concentrator tube for each sample extract.

7.2.1.1 Pipet 1 mL methylene chloride into the concentrator tube.

7.2.1.2 Mark or etch the level of the meniscus on the outside of the tube.

7.2.1.3 Discard the methylene chloride.

7.2.1.4 Add 10 mL methylene chloride into the concentrator tube.

7.2.1.5 Mark or etch the level of the meniscus on the outside of the tube.

7.2.1.6 Discard the methylene chloride.

7.2.1.7 Place the concentrator tube into the nitrogen evaporation apparatus.

7.2.2 Prepare Na_2SO_4 (Item 5.2.5) for drying the extract.

7.2.2.1 Place filter paper in a glass funnel and add Na_2SO_4 until funnel is approximately half full.

7.2.2.2 Rinse the filter paper and Na_2SO_4 three times with methylene chloride and discard the methylene chloride.

7.2.3 Filter the methylene chloride extract through the Na_2SO_4 to dry the extract.

7.2.3.1 Collect the extract in a calibrated concentrator tube.

7.2.3.2 IF the Na_2SO_4 becomes saturated with water, THEN replace the filter paper and Na_2SO_4.

7.2.3.3 Rinse the extract collection bottle three times with methylene chloride and filter through the Na_2SO_4, adding the rinses to the concentrator tube.

7.2.3.4 Rinse the Na_2SO_4 three times with methylene chloride and add the rinses to the concentrator tube.

7.2.3.5 Discard the filter and Na_2SO_4.

7.2.4 Evaporate the extracts to the 10-mL calibration mark using a gentle stream of nitrogen.

7.2.4.1 Place a Pasteur pipet into the tubing of the evaporation apparatus so that the tip of the pipet is above the liquid level.

7.2.4.2 Rinse the internal wall of the tube several times with methylene chloride during the evaporation process.

7.2.5 IF the extract was prepared in a radiological hood, glove box, or hot cell, THEN prepare an aliquot of the extract for radiological screening.

WARNING

Some sample extracts are potentially alpha contaminated.

7.2.5.1 Pipet 0.1 mL of the extract onto a stainless steel planchette.

7.2.5.2 Air-dry the planchette.

7.2.5.3 IF a heavy residue remains after drying, THEN check with the technical lead before proceeding.

7.2.5.4 Place the planchette in a transfer container.

7.2.5.5 Notify the RCT that the sample is ready for gross alpha/beta counting.

7.2.6 IF the contamination levels are ≤ 6 cpm α AND ≤ 30 cpm β/γ, THEN GO TO Step 7.2.12 to further concentrate the extract.

7.2.7 IF the contamination levels are >6 cpm α
OR >30 cpm β/γ,
THEN transfer the extract to a refrigerator and cool to 4 ± 2° C (normally overnight).

7.2.7.1 Repeat Step 7.2.5 to redetermine the extract radioactivity.

7.2.7.2 IF the contamination levels are ≤6 cpm α
AND ≤30 cpm β/γ,
THEN GO TO Step 7.2.12 to further concentrate the extract.

7.2.8 IF the contamination levels are >6 cpm α
OR >30 cpm β/γ,
THEN in consultation with the technical lead, RadCon, and the ALD QA Officer, as appropriate, perform one or more of the following decontamination steps as necessary, and document steps taken in the Sample Preparation Logbook.

A. Repeat Step 7.2.7 (overnight refrigeration)

B. Repeat Steps 7.2.3 through 7.2.6 (filtration through anhydrous sodium sulfate)

C. Filter the extract through a 0.22-µm filter (GO TO Step 7.2.9)

D. Perform alpha-decontamination using titanium chloride and oxalic acid (GO TO Step 7.2.10)

E. Dilute the extract with methylene chloride (GO TO Step 7.2.11).

NOTE: *The decontamination options are listed in order of general preferred use. Option C is useful to remove radionuclide contamination associated with particulate matter. Option D, decontamination using titanium chloride/oxalic acid, is useful to remove organic-soluble Pu^{+4} species. Option E, dilution of the extract, will result in decreased detection capabilities (elevated sample-specific MDLs). Options may be repeated, if necessary.*

7.2.9 IF determined necessary in Step 7.2.8,
THEN filter the extract through a 0.22-µm filter.

7.2.9.1 Triple-rinse an appropriately sized glass syringe fitted with a 0.22-µm nylon filter with ~1 mL aliquots of methylene chloride and discard the rinsate.

7.2.9.2 Quantitatively transfer the extract from the concentrator tube to the syringe.

7.2.9.3 Filter the extract into a clean concentrator tube and GO TO Step 7.2.4.

7.2.10 IF determined necessary in Step 7.2.8,
THEN decontaminate the extract using titanium chloride and oxalic acid.

7.2.10.1 Quantitatively transfer the methylene chloride extract to a small Teflon separatory funnel.

7.2.10.2 Add 10 mL of 5% $TiCl_3$ solution (Item 5.2.20) and 200 µL methanol to the separatory funnel.

7.2.10.3 Seal and gently swirl the separatory funnel.

7.2.10.3.1 Vent the separatory funnel into the back of the hood, away from anything that may deflect any solution or vapors out of the hood.

7.2.10.3.2 Repeat this until the pressure is not excessive.

7.2.10.4 Shake the separatory funnel vigorously for five minutes, with periodic venting.

7.2.10.5 Let the separatory funnel stand for ~15 minutes to allow phase separation.

7.2.10.6 Drain the entire methylene chloride phase into a new Teflon separatory funnel.

NOTE: *It is important to drain the entire methylene chloride phase into the new separatory funnel. Inclusion of small amounts of aqueous phase is not a problem.*

7.2.10.6.1 Add ~2 mL of methylene chloride to the original separatory funnel so that the methylene chloride falls along the inside walls.

7.2.10.6.2 Swirl the separatory funnel and allow the phases to separate.

7.2.10.6.3 Drain the methylene chloride into the separatory funnel containing the original extract.

7.2.10.6.4 Repeat Steps 7.2.10.6.1 through 7.2.10.6.3 twice for a total of three rinses.

7.2.10.6.5 Discard the remaining aqueous phase.

7.2.10.7 Add 10 mL of 1 M oxalic acid (Item 5.2.21) to the separatory funnel containing the methylene chloride.

7.2.10.8 Seal and gently swirl the separatory funnel.

7.2.10.8.1 Vent the separatory funnel into the back of the hood, away from anything that may deflect any solution or vapors out of the hood.

7.2.10.8.2 Repeat this until the pressure is not excessive.

7.2.10.9 Shake the separatory funnel vigorously for five minutes, with periodic venting.

7.2.10.10 Let the separatory funnel stand for ~15 minutes to allow phase separation.

7.2.10.11 Prepare anhydrous Na_2SO_4 for drying the extract per Step 7.2.2 while waiting for the phases to separate.

7.2.10.12 Triple-rinse an appropriately sized glass syringe fitted with a 0.22-µm nylon filter using ~1 mL aliquots of methylene chloride and discard the rinsate.

7.2.10.13 Drain the entire methylene chloride layer into the syringe.

7.2.10.14 Rinse the separatory funnel with methylene chloride.

7.2.10.14.1 Add ~2 mL of methylene chloride to the separatory funnel so that the methylene chloride falls along the inside walls.

7.2.10.14.2 Swirl the separatory funnel and allow the phases to separate.

7.2.10.14.3 Drain the methylene chloride rinse into the same syringe containing the extract.

7.2.10.14.4 Repeat Steps 7.2.10.14.1 through 7.2.10.14.3 twice for a total of three rinses.

7.2.10.14.5 Discard the remaining aqueous phase.

7.2.10.15 GO TO Step 7.2.3, substituting “syringe” for “extract collection bottle” in Step 7.2.3.3.

7.2.11 IF determined necessary in Step 7.2.8,
THEN dilute the extract with methylene chloride.

7.2.11.1 Dilute the extract with methylene chloride until radionuclide levels of ≤6 cpm alpha and ≤30 cpm beta/gamma are achieved.

7.2.11.2 Record the final volume of the extract in the Sample Preparation Logbook.

7.2.11.3 Fill an autosampler vial with extract.

7.2.11.4 Discard the excess extract.

7.2.11.5 GO TO Step 7.2.15.

7.2.12 Ensure the concentrator tube containing the extract is in the nitrogen evaporation apparatus.

7.2.13 Evaporate the extracts below the 1-mL calibration mark using a gentle stream of nitrogen.

CAUTION

Analytes may be lost if the volume of solvent is evaporated below 0.5 mL.

7.2.13.1 Rinse the internal wall of the tube several times with methylene chloride during the evaporation process.

7.2.14 Quantitatively transfer the extract to a labeled graduated autosampler vial (Teflon-lined screw cap) and dilute to the 1-mL graduation mark with methylene chloride.

7.2.15 Store the extract vials at ≤-10 ^{o}C until analysis.

8. QUALITY CONTROL REQUIREMENTS

8.1 Analytical Batch QC

NOTE: *Acceptance criteria for analytical batch QC samples are provided in the determinative method CCP-TP-185.*

8.1.1 Prepare a minimum of one laboratory blank (LB) per analytical batch. If samples in an analytical batch are prepared in multiple preparation groups, prepare a LB with each group of sample preparations.

8.1.2 Prepare a minimum of one laboratory control sample (LCS) per analytical batch.

8.1.3 Prepare a minimum of one matrix spike (MS) per analytical batch.

8.1.4 Prepare a minimum of one matrix spike duplicate (MSD) per analytical batch.

8.2 System Monitoring with Surrogate Compounds

8.2.1 Add acid and base/neutral surrogate compounds specified in Item 5.2.11or Items 5.2.9 and 5.2.10 to each sample and batch QC sample.

8.3 Method Performance Demonstration

Method performance demonstrations consist of method detection limit (MDL) determination and method precision and accuracy (P&A) demonstrations.

8.3.1 Demonstrate initial method performance for the sample preparation and determinative method combination. Refer to the determinative method CCP-TP-185 for required number of measurements, frequency, and acceptable accuracy, precision, and MDL criteria for the target analytes.

8.3.2 Demonstrate continuing method performance for the sample preparation and determinative method combination. Refer to the determinative method CCP-TP-185 for required number of measurements, frequency, and acceptable accuracy, precision, and MDL criteria for the target analytes.

9. CALCULATIONS

None

10. RECORDS

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 Sample Preparation Logbook

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

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

11.6 CCP-TP-185, *CCP Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry*

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

11.8 EPA SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," Third Edition, Method 3500B, "Organic Extraction and Sample Preparation," U.S. Environmental Protection Agency, 1986

11.9 EPA SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," Third Edition, Method 3550B, "Ultrasonic Extraction," U.S. Environmental Protection Agency, 1986

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

11.11 MCP-7, "Radiological Work Permit"

11.12 MCP-2703, "Carcinogens"

11.13 MCP-3635, "Chemical Hygiene Plan"