

SVOCs absorbed on the sample media. We recommend spiking the XAD-2 resin with a surrogate standard before testing with a carbon-13 or hydrogen-2 isotopically labeled standard for each of the class of analytes targeted for analysis. Perform this spiking as follows:

(1) Insert the lower PUF plug into the bottom of the sorbent module.

(2) Add half of one portion of XAD-2 resin to the module and spike the XAD-2 in the module with the standard.

(3) Wait 1 hour for the solvent from the standard(s) to evaporate, add the remaining 20 g of the XAD-2 resin to the module, and then insert a PUF plug in the top of the sorbent module.

(4) Cover the inlet and outlet of the sorbent module with pre-cleaned aluminum foil.

(d) *Sampling system assembly.* After preparing the sample media and the sampler, assemble the condensate trap, cooling coil, filter holder with filter, sample probe, and sorbent module, then lower the assembly into the reservoir. Cover the probe inlet with pre-cleaned aluminum foil.

#### § 1065.1109 Post-test sampler disassembly and sample extraction.

This section describes the process for disassembling and rinsing the sampling system and extracting and cleaning up the sample.

(a) *Sampling system disassembly.* Disassemble the sampling system in a clean environment as follows after the test:

(1) Remove the PM filter, PUF plugs, and all the XAD-2 from the sampling system and place them into a Soxhlet extraction thimble. Store them at or below 37 °C until analysis.

(2) Rinse sampling system wetted surfaces upstream of the condensate trap with acetone followed by toluene (or a comparable solvent system), ensuring that all the solvent remaining in liquid phase is collected (note that a fraction of the acetone and toluene will likely be lost to evaporation during mixing). Rinse with solvent volumes that are sufficient to cover all the surfaces exposed to the sample during testing. We recommend three fresh solvent rinses with acetone and two with

toluene. We recommend rinse volumes of 60 ml per rinse for all sampling system components except the condenser coil, of which you should use 200 ml per rinse. Keep the acetone rinsate separate from the toluene rinsate to the extent practicable. Rinsate fractions should be stored separately in glass bottles that have been pre-rinsed with acetone, hexane, and toluene (or purchase pre-cleaned bottles).

(3) Use good engineering judgment to determine if you should analyze the aqueous condensate phase for SVOCs. If you determine that analysis is necessary, use toluene to perform a liquid-liquid extraction of the SVOCs from the collected aqueous condensate using a separatory funnel or an equivalent method. Add the toluene from this aqueous extraction to the toluene rinsate fraction described in paragraph (a)(2) of this section.

(4) Reduce rinsate solvent volumes as needed using a Kuderna-Danish concentrator or rotary evaporator and retain these rinse solvents for reuse during sample media extraction for the same test. Be careful to avoid loss of low molecular weight analytes when concentrating with rotary evaporation.

(b) *Sample extraction.* Extract the SVOCs from the sorbent using Soxhlet extraction as described in this paragraph (b). Two 16 hour extractions are necessary to accommodate the Soxhlet extractions of all SVOCs from a single sample. This reduces the possibility of losing low molecular weight SVOCs and promotes water removal. We recommend performing the first extraction with acetone/hexane and the second using toluene (or an equivalent solvent system). You may alternatively use an equivalent method such as an automated solvent extractor.

(1) We recommend equipping the Soxhlet extractor with a Dean-Stark trap to facilitate removal of residual water from the sampling system rinse. The Soxhlet apparatus must be large enough to allow extraction of the PUF, XAD-2, and filter in a single batch. Include in the extractor setup a glass thimble with a coarse or extra coarse sintered glass bottom. Pre-clean the extractor using proper glass-cleaning procedures. We recommend that the Soxhlet apparatus be cleaned with a (4

to 8) hour Soxhlet extraction with methylene chloride at a cycling rate of three cycles per hour. Discard the solvent used for pre-cleaning (no analysis is necessary).

(2) Load the extractor thimble before placing it in the extractor by first rolling the PM filter around the inner circumference of the thimble, with the sampled side facing in. Push one PUF plug down into the bottom of the thimble, add approximately half of the XAD-2, and then spike the XAD-2 in the thimble with the isotopically labeled extraction standards of known mass. Target the center of the XAD-2 bed for delivering the extraction standard. We recommend using multiple isotopically labeled extraction standards that cover the range of target analytes. This generally means that you should use isotopically labeled standards at least for the lowest and highest molecular weight analytes for each category of compounds (such as PAHs and dioxins). These extraction standards monitor the efficiency of the extraction and are also used to determine analyte concentrations after analysis. Upon completion of spiking, add the remaining XAD-2 to the thimble, insert the remaining PUF plug, and place the thimble into the extractor. Note that if you are collecting and analyzing for SVOCs in gas and particle phases, perform separate extractions for the filter and XAD-2.

(3) For the initial extraction, combine the concentrated acetone rinses (from the sampling system in paragraph (a) of this section) with enough hexane to bring the solvent volume up to the target level of 700 ml. Assemble the extractor and turn on the heating controls and cooling water. Allow the sample to reflux for 16 hours with the rheostat adjusted to cycle the extraction at a rate of  $(3.0 \pm 0.5)$  cycles per hour. Drain the water from the Dean-Stark trap as it accumulates by opening the stopcock on the trap. Set aside the water for analysis or discard it. In most cases, any water present will be removed within approximately 2 hours after starting the extraction.

(4) After completing the initial extraction, remove the solvent and concentrate it to  $(4.0 \pm 0.5)$  ml using a Kuderna-Danish concentrator that in-

cludes a condenser such as a three-ball Snyder column with venting dimples and a graduated collection tube. Using this concentrator will minimize evaporative loss of analytes with lower molecular weight.

(i) Rinse the round bottom flask of the extractor with (60 to 100) ml of hexane and add the rinsate to this concentrated extract.

(ii) Concentrate the mixture to  $(4 \pm 0.5)$  ml using a Kuderna-Danish concentrator or similar apparatus.

(iii) Repeat the steps in paragraphs (b)(4)(i) and (ii) of this section three times, or as necessary to remove all the residual solvent from the round bottom flask of the extractor, concentrating the final rinsate to  $(4 \pm 0.5)$  ml.

(5) For the second extraction, combine the toluene rinses (from the sampling system in paragraph (a) of this section) with any additional toluene needed to bring the solvent volume up to the target level of 700 ml. As noted in paragraph (a) of this section, you may need to concentrate the rinsate before adding it to the extraction apparatus if the rinsate solvent volume is too large. Allow the sample to reflux for 16 hours with the rheostat adjusted to cycle the extraction at a rate of  $(3.0 \pm 0.5)$  cycles per hour. Check the Dean-Stark trap for water during the first 2 hours of the extraction (though little or no water should be present during this stage).

(6) Upon completion of the second extraction, remove the solvent and concentrate it to  $(4 \pm 0.5)$  ml as described in paragraph (b)(4) of this section. Using hexane from paragraph (b)(4) of this section as the rinse solvent effectively performs a solvent exchange of toluene with hexane.

(7) Combine the concentrated extract from paragraph (b)(4) of this section with the concentrated extract from paragraph (b)(6) of this section. Divide the extract into a number of fractions based on the number of analyses you need to perform. Perform the separate sample clean-up described in paragraph (c) of this section as needed for each fraction.

(c) *Sample clean-up.* This paragraph (c) describes how to perform sample cleaning to remove from the sample extract any solids and any SVOCs that

will not be analyzed. This process, known as "sample clean-up", reduces the potential for interference or co-elution of peaks during analytical analysis. Before performing the sample clean-up, spike the extract with an alternate standard that contains a known mass of isotopically labeled compounds that are identical to the target analytes (except for the labeling). The category of the target analyte compounds (such as PAHs or dioxin) will determine the number of compounds that make up the standard. For example, PAHs require the use of four compounds in the alternate standard to cover the four basic ring structures of PAHs (2-ring, 3-ring, 4-ring, and 5-ring structures). These alternate standards are used to monitor the efficiency of the clean-up procedure. Before sample clean-up, concentrate the fractionated sample to about 2 ml with a Kuderna-Danish concentrator or rotary evaporator, and then transfer the extract to an 8 ml test tube with hexane rinse. Concentrate it to a volume of about 1 ml using a Kuderna-Danish concentrator. Use good engineering judgment to select an appropriate column chromatographic clean-up option for your target analytes. Note that these clean-up techniques generally remove compounds based on their polarity. The following procedures are examples of clean-up techniques for PAHs and nPAHs.

(1) *PAH clean-up*. The following method is appropriate for clean-up of extracts intended for analysis of PAHs:

(i) Pack a glass gravity column (250 mm x 10 mm recommended) by inserting a clean glass wool plug into the bottom of the column and add 10 g of activated silica gel in methylene chloride. Tap the column to settle the silica gel and then add a 1 cm layer of anhydrous sodium sulfate. Verify the volume of solvent required to completely elute all the PAHs and adjust the weight of the silica gel accordingly to account for variations among batches of silica gel that may affect the elution volume of the various PAHs.

(ii) Elute the column with 40 ml of hexane. The rate for all elutions should be about 2 ml/min. You may increase the elution rate by using dry air or nitrogen to maintain the headspace

slightly above atmospheric pressure. Discard the eluate just before exposing the sodium sulfate layer to the air or nitrogen and transfer the 1 ml sample extract onto the column using two additional 2 ml rinses of hexane. Just before exposing the sodium sulfate layer to the air or nitrogen, begin elution of the column with 25 ml of hexane followed by 25 ml of 40 volume % methylene chloride in hexane. Collect the entire eluate and concentrate it to about 5 ml using the Kuderna-Danish concentrator or a rotary evaporator. Make sure not to evaporate all the solvent from the extract during the concentration process. Transfer the eluate to a small sample vial using a hexane rinse and concentrate it to 100 µl using a stream of nitrogen without violently disturbing the solvent. Store the extracts in a refrigerator at or below 4 °C, and away from light.

(2) *nPAH clean up*. The following procedure, adapted from "Determination and Comparison of Nitrated-Polycyclic Aromatic Hydrocarbons Measured in Air and Diesel Particulate Reference Materials" (Bamford, H.A., *et al*, *Chemosphere*, Vol. 50, Issue 5, pages 575-587), is an appropriate method to clean up extracts intended for analysis of nPAHs:

(i) Condition an aminopropyl solid phase extraction (SPE) cartridge by eluting it with 20 ml of 20 volume % methylene chloride in hexane. Transfer the extract quantitatively to the SPE cartridge with at least two methylene chloride rinses. Elute the extract through the SPE cartridge by using 40 ml of 20 volume % methylene chloride in hexane to minimize potential interference of polar constituents, and then reduce the extract to 0.5 ml in hexane and subject it to normal-phase liquid chromatography using a pre-prepared 9.6 mm x 25 cm semi-preparative Chromegabond® amino/cyano column (5 µm particle size) to isolate the nPAH fraction. The mobile phase is 20 volume % methylene chloride in hexane at a constant flow rate of 5 ml per minute. Back-flash the column with 60 ml of methylene chloride and then condition it with 200 ml of 20 volume % methylene chloride in hexane before each injection. Collect the effluent and concentrate it to about 2 ml using the

## § 1065.1111

Kuderna-Danish concentrator or a rotary evaporator. Transfer it to a minivial using a hexane rinse and concentrate it to 100 µl using a gentle stream of nitrogen. Store the extracts at or below 4 °C, and away from light.

(ii) [Reserved]

### § 1065.1111 Sample analysis.

This subpart does not specify chromatographic or analytical methods to analyze extracts, because the appropriateness of such methods is highly dependent on the nature of the target analytes. However, we recommend that you spike the extract with an injection standard that contains a known mass of an isotopically labeled compound that is identical to one of the target analytes (except for labeling). This injection standard allows you to monitor the efficiency of the analytical process by verifying the volume of sample injected for analysis.

## PART 1066—VEHICLE-TESTING PROCEDURES

### Subpart A—Applicability and General Provisions

Sec.

- 1066.1 Applicability.
- 1066.2 Submitting information to EPA under this part.
- 1066.5 Overview of this part 1066 and its relationship to the standard-setting part.
- 1066.10 Other procedures.
- 1066.15 Overview of test procedures.
- 1066.20 Units of measure and overview of calculations.
- 1066.25 Recordkeeping.

### Subpart B—Equipment, Measurement Instruments, Fuel, and Analytical Gas Specifications

- 1066.101 Overview.
- 1066.105 Ambient controls and vehicle cooling fans.
- 1066.110 Equipment specifications for emission sampling systems.
- 1066.120 Measurement instruments.
- 1066.125 Data updating, recording, and control.
- 1066.130 Measurement instrument calibrations and verifications.
- 1066.135 Linearity verification.
- 1066.140 Diluted exhaust flow calibration.
- 1066.145 Test fuel, engine fluids, analytical gases, and other calibration standards.
- 1066.150 Analyzer interference and quench verification limit.

## 40 CFR Ch. I (7–1–14 Edition)

### Subpart C—Dynamometer Specifications

- 1066.201 Dynamometer overview.
- 1066.210 Dynamometers.
- 1066.215 Summary of verification procedures for chassis dynamometers.
- 1066.220 Linearity verification for chassis dynamometer systems.
- 1066.225 Roll runout and diameter verification procedure.
- 1066.230 Time verification procedure.
- 1066.235 Speed verification procedure.
- 1066.240 Torque transducer verification.
- 1066.245 Response time verification.
- 1066.250 Base inertia verification.
- 1066.255 Parasitic loss verification.
- 1066.260 Parasitic friction compensation evaluation.
- 1066.265 Acceleration and deceleration verification.
- 1066.270 Unloaded coastdown verification.
- 1066.275 Daily dynamometer readiness verification.
- 1066.290 Verification of speed accuracy for the driver's aid.

### Subpart D—Coastdown

- 1066.301 Overview of coastdown procedures.
- 1066.305 Coastdown procedures for motor vehicles at or below 14,000 pounds GVWR.
- 1066.310 Coastdown procedures for vehicles above 14,000 pounds GVWR.
- 1066.315 Dynamometer road-load setting.

### Subpart E—Preparing Vehicles and Running an Exhaust Emission Test

- 1066.401 Overview.
- 1066.405 Vehicle preparation and preconditioning.
- 1066.410 Dynamometer test procedure.
- 1066.415 Vehicle operation.
- 1066.420 Test preparation.
- 1066.425 Performing emission tests.

### Subpart F—Electric Vehicles and Hybrid Electric Vehicles

- 1066.501 Overview.

### Subpart G—Calculations

- 1066.601 Overview.
- 1066.605 Mass-based and molar-based exhaust emission calculations.
- 1066.610 Dilution air background correction.
- 1066.615 NO<sub>x</sub> intake-air humidity correction.
- 1066.625 Flow meter calibration calculations.
- 1066.630 PDP, SSV, and CFV flow rate calculations.
- 1066.635 NMOG determination.
- 1066.695 Data requirements.