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Fundamentals of the Method for Analysis of Polynuclear Aromatic Hydrocarbons (PAHs) in Method 0010 Train Samples

MM5 Train Configuration for Polynuclear Aromatic Hydrocarbons (PAHs)

The train configuration of the MM5 sampling train will be used to collect stack gas for an assessment of the PAH compounds listed in Table 1, using the procedures outlined in SW-846 Methods 0010, 3542, and 8290. This train may also be used to measure dioxin and furan compound concentrations found in the stack gas using SW-846 Method 0023A, semivolatile organics, and PCBs by Method 1668.

During each CPT run, the MM5 train will be assembled and leak-checked before sampling commences. A minimum of 3 dry standard cubic meters of stack gas will be sampled during each sampling run. At the end of each run, the sampling train will be disassembled, and all train samples will be collected.

In the field, the front-half solvent rinses of the filter holder, the probe, and nozzle will be collected by conducting three separate and thorough rinses each of acetone and methylene chloride in that order. When the same MM5 train handles the PAHs and the dioxins and furans, the toluene probe rinses should be collected in a separate sample bottle from those of the acetone and methylene chloride probe rinses. In the analytical scheme, toluene will be handled in such a way as to introduce the toluene only into the dioxin and furan fraction. Toluene blow-down for extract volume reductions are significantly more difficult than the more volatile acetone and methylene chloride solvents. PAH or semivolatile losses during the extract volume reductions are avoided if toluene is prevented from mixing with the semivolatile fraction preparation.

The particulate filter and front-half rinses (acetone and methylene chloride, only) will be Soxhlet extracted using methylene chloride for eighteen (18) hours (Method 3540C). PAH isotope dilution internal standards will be added to the samples at this stage of the sample preparation. The PAH sampling surrogates (and PCB, dioxin, and furan sampling surrogates when a combined train is used) will also be added to the particulate samples at this time. (A subsequent Soxhlet extraction will be conducted using toluene for dioxins and furans, at which time the toluene probe rinse will be added to the sample.) The extracts will be set aside for analysis by Method 8290 for PAH compounds.

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TABLE 1

SUMMARY OF PAH COMPOUNDS FOR ANALYSIS

Analyte	CAS Registry Number
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Biphenyl	92-52-4
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Benzo(ghi)perylene	191-24-2
Benzo(a)pyrene	50-32-8
Benzo(e)pyrene	192-97-2
2,6-Dimethylnaphthalene	581-42-0
Fluoranthene	206-44-0
Fluorene	86-73-7
Indeno(1,2,3-cd)pyrene	193-39-5
2-Methylnaphthalene	91-57-6
1-Methylnaphthalene	90-12-0
1-Methylphenanthrene	832-69-9
Naphthalene	91-20-3
Perylene	198-55-0
Phenanthrene	85-01-8
Pyrene	129-00-0
2,3,5-Trimethylnaphthalene	2245-38-7

The XAD-2 resin tube samples and the solvent rinses of the back-half filter holder, and the coil condenser samples will be handled in the same way as the front half samples, except that they will be prepared separately and analyzed as a separate sample. These samples will also be extracted sequentially using methylene chloride and followed by toluene when a combined train is used. Extractions will be conducted using Soxhlet extraction apparatus (Method 3540C), and the extracts will be combined for analysis in the same way as the front half fractions. Sampling surrogates will not be added to these samples during preparation since they will be applied to the XAD-2 resin prior to field sampling.

The condensate impinger catch samples will consist of a composite of the deionized water impinger condensate catches. The impinger catches will be combined in the field and measured volumetrically to within plus or minus 1 mL using a graduated cylinder and also will be weighed gravimetrically to within plus or minus 0.5 grams using an analytical balance.

The impinger condensate composite includes the contents of MM5 impingers 1, 2, and 3. The impingers and connecting glassware receive acetone and methylene chloride solvent rinses. The solvent rinses are collected in separate sample container. A 1-L portion of the impinger condensate composite will be combined with the glassware solvent rinse sample, and a continuous liquid-liquid extraction will be carried out using Method 3520 using methylene chloride, only. The PAH extracts will be reduced to a volume of one (1) milliliter for the analyses of PAHs. Note that the total impinger condensate composite volume must be recorded before extraction commences. The reported analyte concentrations will be delivered from the analyst in micrograms per liter (µg/L), which will be multiplied by the total condensate composite volume to obtain the total analyte contribution from the condensate samples.

A spiking program will be applied to the MM5 trains that will allow for complete assessment of the sampling and analytical process regarding the overall method accuracy. Spiked compounds will be placed on the components of the train at the different stages of the sampling and analytical program so that the efficiency of the method's performance can be measured quantitatively. By assuming that the spiking compounds have chemical characteristics that are identical to the PAH target compounds, the overall method efficiency can be assessed. Four types of spiking materials will be applied to the MM5 train samples. These types are defined as follows:

- Sampling Surrogate Spikes—These compounds are spiked directly onto the XAD-2 resin at the laboratory during resin tube preparation and prior to any field handling or sampling. The final recovery of these compounds gives the most comprehensive indication that the determination of native compounds using the MM5 methodology is accurate. Good recovery of these compounds will reflect the XAD-2 resin's ability to capture and retain semivolatiles, PCBs, and the various isomers of dioxins and furans.

- PAH Isotope Dilution Internal Standard Spikes—These compounds are placed directly onto the sample just prior to the preparation and extraction steps. The final recovery efficiency of these compounds reflects the overall accuracy of the sample's laboratory handling and analysis. Accordingly, these compounds are used to generate data that indicate the relative accuracy of the analytical methods.

- PAH Recovery Standards—These compounds are applied to the sample extracts just before the extracts are introduced onto the GC/MS instrument injection ports. These compounds are precisely applied at this step in the analytical scheme and provide the actual relative response factors that are used to calculate analyte concentrations.

- Matrix Spike Compounds (back half and spiked resin blanks only)—These compounds are spiked onto separately prepared aliquots of the MM5 train condensate samples or XAD-2 resins before analysis. The spiked aliquots are then analyzed, and the spike recovery is calculated. Recovery of these spikes provides an independent indicator of method accuracy relative to the sample matrix.

The analysis of the extracts for PAHs will be analyzed by selective ion monitoring (SIM) GC/MS.

Method 0010 for Polynuclear Aromatic Hydrocarbons in Stack Gas Emissions

SAMPLE PRESERVATION AND HOLDING TIMES

All samples requiring refrigeration should be placed on ice (when required for preservation) in coolers during and after sampling and will be stored at a temperature of approximately 4°C until analyzed. In addition to cooling all samples that require low temperature preservation, chemical preservatives should be used, as required, in samples for specific analyses according to EPA protocols.

The holding times and preservation techniques are either those recommended in Title 40 CFR Section 136.3, Table 11, "Required Containers, Preservation Techniques, and Holding Times," or those presented by EPA in Table 3-1 of the *Handbook - Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration* (EPA-625/6-89-023).

Measurement	Matrix	Preservation[a]	Holding Time[b]
Polynuclear Aromatic Hydrocarbons (PAHs)	Particulate Filter and Front-Half Filter Holder and Probe Solvent Rinses	Chill with ice 4 °C ±2°C	14 days to extraction, 40 days from extraction to analysis
	XAD-2 Resin and Back Half of the Filter Holder Solvent Rinses	Chill with ice 4 °C ±2°C	14 days to extraction, 40 days from extraction to analysis
	Impinger Condensate Composite and the Glassware Solvent Rinses	Chill with ice 4 °C ±2°C	14 days to extraction, 40 days from extraction to analysis

[a] CPT samples requiring refrigeration will be preserved on ice from the time of collection through delivery to the analytical laboratory.

[b] Holding times are calculated from the date of collection.

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Method 0010

PAHs in Stack Gas

Method 0010 PAH Recommended Quality Measurements for a 3 Run CPT

Analytical Parameter (Analysis)	Sample Name or Type	Total No. of Field Samples	Analytical Procedure Description (Method)	Laboratory QC Measurement Type	Frequency of Applied QC Measurement Type	Total No. of Laboratory QC Measurements	Field QC Measurement Type	Total No. of Field QC Samples	Total No. of Laboratory Analyses
Polynuclear Aromatic Hydrocarbons (PAHs)	MM5 Train (Particulate Filter and Front-Half Filter Holder and Probe Solvent Rinses)	3	Soxhlet extraction, GC/MS (SW-3540C, SW-3542, SW-8290, CARB 429)	PAH isotope dilution internal standard spike[a]	Every filter and solvent combined sample	4	Blank train	1	4
				Carbon-13-labeled sampling surrogate spike	Every filter and solvent combined sample	4			
	MM5 Train (XAD-2 Resin and Back Half of the Filter Holder Solvent Rinses)	3	Soxhlet extraction, GC/MS (SW-3540C, SW-3542, SW-8290, CARB 429)	Spiked resin blank	Two resin tubes per test condition	2	Trip blank	1	7
				Carbon-13-labeled sampling surrogate spike	Every XAD-2 resin tube including blanks	7	Blank train	1	
				PAH isotope dilution internal standard spike[a]	Every XAD-2 resin tube including blanks	7			
	MM5 Train (Impinger Condensate Composite and the Glassware Solvent Rinses)	3	Liquid-liquid extraction, GC/MS (SW-3510C, SW-3542, SW-8290, CARB 429)	PAH isotope dilution internal standard spike[a]	Every impinger composite including blanks	4	Blank train	1	4

[a] Surrogate spikes will be applied to all samples including matrix spikes, duplicates, and blank analytical aliquots.

Analytical Quality Control Checks, Frequencies, Target Acceptance Criteria, and Corrective Action

Parameter/Method	QC Check	Frequency	Target Criteria	Corrective Action
Polynuclear Aromatic Hydrocarbons (PAHs) by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry-Selected Ion Monitoring (HRGC/LRMS-SIM)	Mass scale calibration (tuning) using DFTPP	Prior to initial calibration, before each 12 hour shift	Ion abundance within ranges specified in Method 8270C	Make necessary adjustments until conditions are met
	GC column performance (resolution check)	Prior to initial calibration, before each 12 hour shift	Resolution of anthracene from phenanthrene, and benzo (e)pyrene from benzo(a)pyrene • %Valley ≤ 25%	Make necessary adjustments until conditions are met
	Initial Calibration (ICAL) (linearity check at five concentration levels and retention time window verification)	Prior to analysis, repeat as needed	Relative Response Factors (RRF): • %RSD ± 30% for unlabeled standards • %RSD ± 30% for internal standards Other criteria • S/N ratios (of primary ions) ≥ 10	(1) Make necessary adjustments (2) Repeat linearity check
	Continuing Calibration	Beginning and end of each 12-hour shift	%Recovery of standard compounds • %Recovery: 70 – 130% for labeled standards • %Recovery: 70 – 130% for unlabeled standards • Mass scale calibration within specifications • S/N ratios ≥ 10	(1) Perform corrective action, then repeat single point check in duplicate (2) If either single-point check is unacceptable, perform multi-point calibration
	Laboratory Method Blanks	Once per sample batch (maximum 20 samples) Analyze after calibration standard and before the first sample	Target compound concentrations • Concentration < lower quantitation level	(1) Flag data associated with method blanks (2) Discuss in final report
	Laboratory Control Sample	Once per sample batch (maximum 20 samples) Analyze after calibration standard and before the first sample	%Recovery of IDIS and Target Analytes • %Recovery: 60 - 140 for internal standards • %Recovery: 60 - 140 for target analytes	(1) Flag data (2) Discuss in final report
	Internal Standard Spike	Every sample (including method blanks and all QC samples)	%Recovery of internal standards • Naphthalene-d_8: 15 to 120% • Acenapthylene-d_8: 20 to 120% • 2-Methylnapthylene-d_{10}: 20 to 120% • All other labeled IDIS: 30 to 120%	(1) Flag data (2) Discuss in final report
	Laboratory Surrogate and Sampling Surrogate spike recovery	Optional	%Recovery of surrogates • 40 to 160% recovery	(1) Flag data (2) Discuss in final report

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TestAmerica Summary of Field Quality Control Sample Requirements for a 3 Run CPT

Sample[a]	QC Sample Type	Frequency	QC Sample Total
MM5 Train Polynuclear Aromatic Hydrocarbons (PAHs) (Methods 3542, 0010, and CARB 429)	Trip Blanks	One per CPT sample shipment	1 to 3 XAD-2 resin tubes
	Field Blanks	One per CPT	1 XAD-2 resin tube
	Blank Train	One blank train per CPT: Particulate filter and front-half of the filter holder and probe solvent rinses, XAD-2 resin and solvent rinses of the back-half filter holder and coil condenser, impinger condensate composite and solvent rinses	1 set of train samples per test
	Spiked Resin Blanks	Two per CPT	2 XAD-2 resin tubes

Method 0010/CARB 429 SPIKE Compounds and Quantity Spiked

Spike Type	Quantity Spiked
PAH Isotope Dilution Internal Standard Compounds (applied at commencement of sample prep)	
d_8-Naphthalene	100 ηg
d_{10}-2-Methylnaphthalene	100 ηg
d_{10}-1-Methylnaphthalene	100 ηg
d_8-Acenaphthylene	100 ηg
d_{10}-Phenanthrene	100 ηg
d_{12}-2,6-dimethylnaphthalene	100 ηg
d_{10}-Fluoranthene	100 ηg
d_{12}-Benz(a)anthracene	100 ηg
d_{12}-Chrysene	100 ηg
d_{12}-Benzo(b)fluoranthene	100 ηg
d_{12}-Benzo(k)fluoranthene	100 ηg
d_{12}-Benzo(a)pyrene	100 ηg
d_{12}-Perylene	100 ηg
d_{12}-Indeno(1,2,3-cd)pyrene	100 ηg
d_{14}-dibenz(a,h)anthracene	100 ηg
d_{12}-Benzo(ghi)perylene	100 ηg
D_{10}-Anthracene (Alternate standard; used only when combining condensate extracts and XAD/filter extracts)	100 ηg
PAH Sampling Surrogate Compounds (applied to XAD-2 before field sampling)	
$^{13}C_6$-Fluorene	250 ηg
d_{10}-Fluorene	250 ηg
d_{14}-p-Terphenyl	250 ηg

Notes:

ηg Nanogram
µg Microgram
mL Milliliter

Summary of PAH Surrogate and Internal Standard Target Recovery Ranges

PAH Sampling Surrogate Compounds	Target Percent Recovery Range
$^{13}C_6$-Fluorene	30-120%
d_{10}-Fluorene	30-120%
d_{14}-p-Terphenyl	30-120%
PAH Isotope Dilution Internal Standard Compounds	
d_8-Naphthalene	30-120%
d_{10}-2-Methylnaphthalene	30-120%
d_{10}-1-Methylnaphthalene	30-120%
d_8-Acenaphthylene	30-120%
d_{10}-Phenanthrene	30-120%
d_{12}-2,6-dimethylnaphthalene	30-120%
d_{10}-Fluoranthene	30-120%
d_{12}-Benz(a)anthracene	30-120%
d_{12}-Chrysene	30-120%
d_{12}-Benzo(b)fluoranthene	30-120%
d_{12}-Benzo(k)fluoranthene	30-120%
d_{12}-Benzo(a)pyrene	30-120%
d_{12}-Perylene	30-120%
d_{12}-Indeno(1,2,3-cd)pyrene	30-120%
d_{14}-dibenz(a,h)anthracene	30-120%
d_{12}-Benzo(ghi)perylene	30-120%
D_{10}-Anthracene	30-120%

Method 0010 for Polynuclear Aromatic Hydrocarbons in Stack Gas Emissions

Method 0010 Recommended Sample Collection Methods, Frequency, and Containers for a 3 Run CPT

Sample Name (Matrix)	Analysis	Type of Container(s)	Sampling Method	Sampling Frequency	Test Samples	Field QC Samples	Total Field Samples Collected
MM5 Train Front-Half Composite (Particulate Filter and Front-Half Filter Holder and Probe Solvent Rinses)	Polynuclear Aromatic Hydrocarbons (PAHs)	Petri Dishes, 250-ml Boston-Round Amber Glass	Method 3542[a] Method 0010[b] Modified CARB 429[c]	Collect 3 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 blank train front half composite	4
MM5 Train Back-Half Composite (XAD-2 Resin Tube and Back-Half of the Filter Holder And Coil Condenser Solvent Rinses)	Polynuclear Aromatic Hydrocarbons (PAHs)	XAD-2 Resin Tubes, 250-ml Boston-Round Amber Glass	Method 3542[a] Method 0010[b] Modified CARB 429[c]	Collect 3 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 blank train back half composite, 1 field blank, 1 trip blank	6
MM5 Train (Impinger Composite and Glassware Solvent Rinses)	Polynuclear Aromatic Hydrocarbons (PAHs)	1-Gallon Amber Wheaton Glass, 250-mL Boston Round Amber Glass	Method 3542[a] Method 0010[b] Modified CARB 429[c]	Collect 3 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 blank train impinger composite	4

[a] Method 3542 is appropriate for sampling for semivolatile analytes. Taken from "Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5) Sampling Train", *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996). U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. 20460.

[b] Method 0010 is appropriate for sampling stack gas for semivolatile organic compounds. Taken from "Modified Method 5 Sampling Train," *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, (SW-846), Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996). U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. 20460.

[c] CARB 429 is appropriate for sampling stack gases for polynuclear aromatic hydrocarbons (PAHs). Taken from "Determination of Polycyclic Aromatic Hydrocarbon (PAH) Emissions from Stationary Sources", State of California Air Resources Board Method 429, Adopted September 12, 1989.

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Method 0010

Method for Determining Polynuclear Aromatic Hydrocarbons (PAHs) in Stack Gas

Sampling and Field Procedure for Polynuclear Aromatic Hydrocarbons (PAHs) in Stack Gas

Sample Name:	Modified Method 5 (MM-5) Polynuclear Aromatic Hydrocarbon (PAH) Sampling Train
Sampler:	Stack Sampling Engineer
Process Sample Location:	Stack Sampling Platform
Sampling & Health & Safety Equipment:	Sampling and safety equipment is as follows: ▪ U.S. EPA Method 0010 (MM-5) Sampling Train ▪ Organic-free DI water ▪ Aluminum foil ▪ 250 mL amber Boston Round - acetone probe rinse and the methylene chloride probe rinse ▪ Glass Petri Dish - particulate filter ▪ XAD-2 Resin Tube ▪ 250 mL amber Boston Round - backhalf rinse of the filter holder behind the particulate filter, coil condenser and connecting glassware with acetone and methylene chloride ▪ 1 Liter amber Boston Round - condensate and impinger contents of impingers #1, #2, and #3 ▪ 250 mL amber Boston Round - acetone and methylene chloride rinse of the impingers and connecting glassware ▪ Squirt bottles for acetone and methylene chloride ▪ Graduated cylinder ▪ Safety glasses or face shield ▪ Gloves and other safety equipment as required
Sample Collection Frequency:	Continuously for approximately 4 hours until at least 3 m^3 of stack sample is collected for each run; sampling rate will be ≤ 0.75 m^3 per hour. Three runs will constitute a test.
Sampling Procedures:	XAD-2 Tube Preparation - The laboratory will prepare the XAD-2 resin tubes and deliver them to the sampling team for use during the project. During the resin preparation, 250 ηg each of $^{13}C_6$-fluorene, p-terphenyl-d_{14}, and fluorene-d_{10}

sampling surrogates will be applied to each XAD-2 resin tube. These labeled spikes will serve as sampling surrogates to indicate analyte loss due to the sampling process. The procedures for preparing, handling, storing, and analyzing the tubes are those described in the U.S. EPA SW-846 Methods 0010 referenced below. Pre-cleaned XAD-2 resin is commercially available (Supelco® or Restek®) and will be used to prepare the resin tubes. Two XAD-2 resin tubes using the purchased resin will be spiked with surrogates and isotope dilution internal standards and analyzed as laboratory resin blanks (Spiked Resin Blanks) to confirm that the resin is free from significant background contamination and to assess the recovery capabilities of the analytes from the resin batch.

For storage and transport to the field, the resin tubes will have their ends sealed with Teflon® tape, wrapped in aluminum foil, sealed in Ziploc® bags, and packed in a clean sample cooler. In the field, the cooler will be stored in the sample recovery trailer and resin tubes are removed only when ready for labeling and installation in the sampling train.

Before each sampling run, the Sampling Coordinator will supply a XAD-2 resin tube and a field blank tube, if required, to the Stack Sampling Engineer who will direct the operation of the MM-5 train. At the end of each run, the Sample Coordinator will recover the resin tubes and other train components and complete the preparation of the sample documentation. The MM-5 stack samples will be stored on ice at approximately 4^{o}C in insulated coolers in a storage area away from sources of fugitive contamination.

MM-5 Train Operation - The MM-5 train components will be provided by the Stack Sampling team. With the exception of the necessary modification for installing and recovering the resin tubes, the sampling procedures will be as specified in U.S. EPA Methods 1 and 2 for stack flow measurements, and Method 4 and 5 for moisture content and particulate. An initial traverse is made with a pitot tube at each sample point following U.S. EPA Methods 1 and 2 to establish the stack velocity profile, temperature, and flow rate, and to check for cyclonic air flow. Sample point location will be in accordance with U.S. EPA Method 1. The sampling team will record the data as recommended in Method 5.

The sampling equipment will be calibrated before and after the test. The pretest calibrations will be available for agency review before testing commences.

The first impinger (Impinger #1) will be an empty condensate knockout impinger. The MM-5 train will be charged with 100 ml of organic-free DI water in the second (Impinger #2) and third (Impinger #3) impingers. The fourth impinger will contain indicating silica gel which is tare weighed to the nearest 0.5 gram.

The sampling train will be leak tested according to U.S. EPA Method 5 protocols. A Teflon® plug or a sampler's thumb covered with Teflon® tape will be placed over the end of the nozzle to ensure that no contaminants are transferred to the train during nozzle leak checks.

MM-5 Train Sample Recovery - The six (6) sample fractions that will be separately recovered from the MM-5 train are as follows:

- Particulate Filter - Will be removed from its holder and carefully placed in its original, labeled Petri dish, sealed with Teflon® tape, and sealed in a Ziploc® plastic bag for shipment to the laboratory.

- Solvent Probe Rinse - The nozzle, probe, the front-half of the filter holder will be brushed and rinsed three times with acetone followed by brushing and rinsing three times with methylene chloride. The rinses will be combined and placed in a 250 mL amber labeled Boston Round sample collection bottle with a Teflon®-lined lid.

- XAD-2 Resin Tube - The XAD-2 resin tube will be removed from the sampling train, its ends capped or sealed with Teflon® tape, wrapped in aluminum foil, sealed in a Ziploc® bag, and stored on ice for shipment to the laboratory.

- Back half of the Filter Holder and Coil Condenser solvent glassware rinses - The back half of the filter holder, coil condenser, and connecting glassware will be rinsed three times with acetone and methylene chloride. The rinses will be combined and placed in a 250 mL amber Boston Round sample bottle with Teflon®-lined lid.

- Condensate (Impinger #1) and Impinger Contents of Impingers #2 and #3 - The aqueous contents of each individual impinger (1-3) will be volumetrically measured to the nearest milliliter, recorded separately for moisture calculations, and then combined into a 1 gallon Wheaton jug with a Teflon®-lined lid. All three impingers and connecting glassware are rinsed three times with DI water. The rinses are then added to the sample bottle.

- Condensate and Impinger Contents of Impingers #2 and #3 Solvent Glassware Rinses - Rinse Impingers #1 - #3 three times with acetone followed by three times with methylene chloride. Place these solvent rinses in a labeled sample collection bottle with a Teflon®-lined lid.

- Silica Gel - The silica gel impinger will be reweighed to the nearest 0.5 gram and the weight gain is calculated as moisture gain in the train.

All of the MM-5 sample components will be assigned unique sample tracking numbers and labeled with date and test run number. The samples will be recovered by the Sample Coordinator and the Stack Sampling Engineer and the sample collection documentation will be recorded. The Sample Coordinator will record the appropriate data in the field logbook and pack the samples on ice in a storage cooler.

Quality Assurance:

A complete MM-5 blank train will be prepared once during the test burn series, set up near the base of the stack in a manner similar to the actual MM-5 sampling train and applying an equivalent number of associated leak checks. It is required

that the blank train be set up during one of the actual PAH sampling runs. The train will remain sealed with the filter holder and probe heated to their standard operating temperature at that location for a time period equivalent to one test run. The blank train samples will be recovered using the same procedures described above for the actual train samples.

An XAD-2 resin field blank will be opened at the location of train assembly one time during the test. The XAD-2 should be open for the duration of actual train assembly. An XAD-2 trip blank should accompany each Method 0010 shipment of samples to the laboratory.

Two spiked resin blanks of the XAD-2 resin are to be prepared at the time of resin tube preparation and analyzed with the field samples.

Liquid samples will have the liquid levels clearly marked on the sample bottles to display the final sample contents level.

Sample Preservation:

The holding times for semivolatile samples is 14 days to extraction from the time of collection, and 30 days from extraction to analysis. All samples should be preserved on ice at approximately 4ºC.

Method References:

Method 3542 – "Extraction of Semivolatile Analytes Collected Using Method 0010 ("Modified Method 5 Sampling Train"). Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 0010 – "Modified Method 5 Sampling Train". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

CARB Method 429 - "Determination of Polycyclic Aromatic Hydrocarbon Emissions from Stationary Sources." State of California, Air Resources Board. September 12, 1989.

Method 5, Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.

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Analytical Procedure for Polynuclear Aromatic Hydrocarbons (PAHs) in MM-5 Train Samples

Sample Name:

MM-5 Train for the collection of PAHs and Project Specific Semivolatile Compounds

The actual sample names given to fractions derived from the MM-5 Train are:

- Front Half Composite - Particulate filter and the front half of the filter holder, probe and nozzle solvent rinses (Figure 1)
- Back Half Composite - XAD-2 resin tube and the back half of the filter holder and coil condenser solvent rinses (Figure 2)
- Condensate Composite - Condensate and impinger composite, and glassware solvent rinses (Figure 3).

Sample Holding Time:

Extract within 14 days of sample collection, and analyze within 40 days from extraction date.

Analysis Procedures:

Front Half Composite
Place Solvent Probe and Nozzle Rinse, Particulate Filter and Fronthalf of the Filter Holder Rinses into a Soxhlet extractor. Add the project specific semivolatile surrogate compounds and isotopically labeled PAH internal standards onto the filter portion prior to extraction. Extract for 18-hours using methylene chloride.

Concentrate the extract to 1 ml for PAH analysis.

Add PAH recovery and analyze for PAHs by GC/MS in the selective ion monitoring (SIM) mode for PAHs (Modified CARB 429/SW-846 Method 8290).

Back Half Composite
Place XAD-2 Resin Tube and the Backhalf of the Filter Holder and Coil Condenser Solvent Rinses into a Soxhlet extractor. Add the project specific semivolatile surrogate compounds and isotopically labeled PAH internal standards onto the filter portion prior to extraction. Extract for 18-hours using methylene chloride.

Concentrate the extract to 1 ml for PAH analysis.

Add PAH recovery standards and analyze by GC/MS in the selective ion monitoring (SIM) mode for PAHs (Modified CARB 429/SW-846 Method 8290).

Condensate Composite
Place a one liter portion of the sample in a separatory funnel and add PAH isotope dilution internal standards onto the sample. Perform a liquid-liquid extraction using Method 3510. Concentrate the extract to 1 mL.

Add the the PAH recovery standards to the appropriate extract portions and analyze for PAHs by GC/MS in the SIM mode (Modified CARB 429/SW-846 Method 8290).

Method References:

Method 3510 - "Separatory Funnel Liquid-Liquid Extraction". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 3540 - "Soxhlet Extraction". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 3542 - "Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train)". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

CARB Method 429 - "Determination of Polycyclic Aromatic Hydrocarbon Emissions from Stationary Sources." State of California, Air Resources Board. September 12, 1989.

Method 8290 - "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Figure 1. MM5 Train Sample Handling and Extract Splitting Scheme for the Particulate Filter and Front Half of the Filter Holder and Probe Solvent Rinses (PAHs)

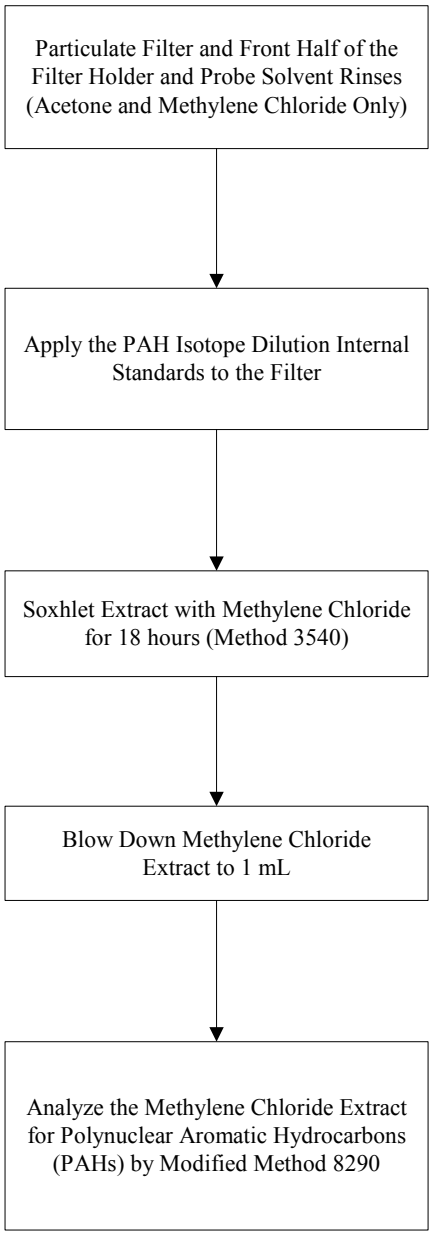

Figure 2. MM5 Train Sample Handling and Extract Splitting Scheme for the XAD-2 Resin Tube and the Back Half of the Filter Holder and Coil Condenser Solvent Rinses (PAHs)

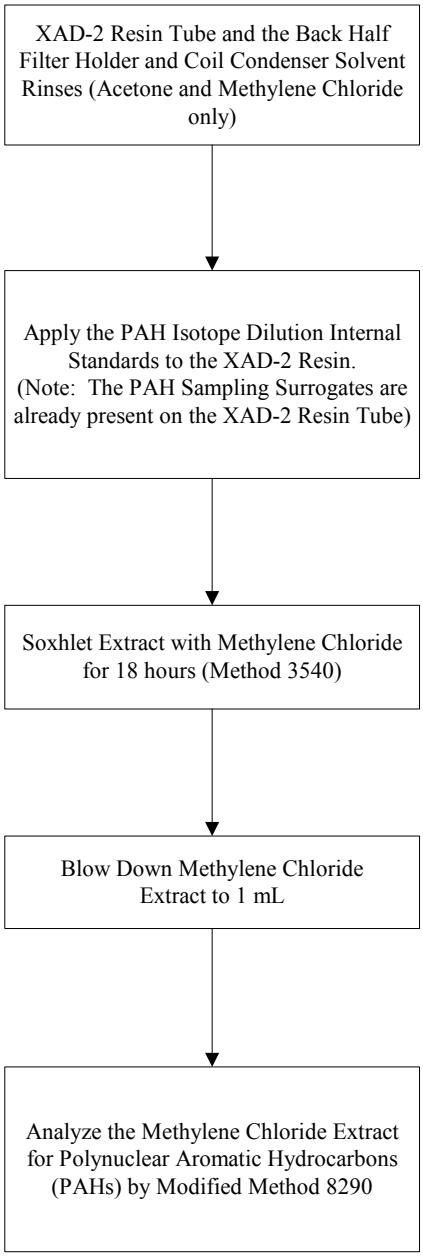

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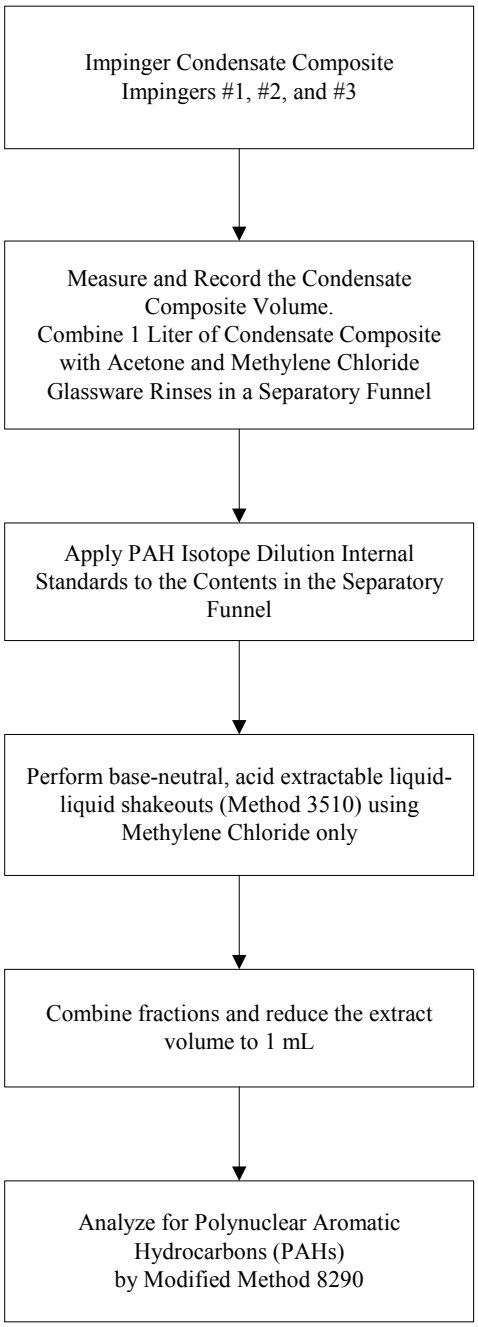
Figure 3. MM5 Train Sample Handling Scheme for the Impingers #1, #2, and #3 Condensate Composite and Glassware Solvent Rinses (PAHs)



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