Fundamentals of the Method for Analysis of Dioxins/Furans in Method 0010/0023A Train Samples

Method 0023A Train Configuration for Dioxins and Furans

The train configuration of the Method 0023A sampling train is used to collect stack gas for the assessment of the dioxin and furan compound concentrations found in the stack gas using SW-846 Method 0023A. The Method 0023A compounds are listed below.

PCDDs/PCDFs for GC/MS Analysis by Method 0023A	
PCDD/PCDF	**CAS Number**
Dioxins	
2,3,7,8-TCDD	1746-01-6
Total TCDD	41903-57-5
1,2,3,7,8-PeCDD	40321-76-4
Total PeCDD	36088-22-9
1,2,3,4,7,8-HxCDD	39227-28-6
1,2,3,6,7,8-HxCDD	57653-85-7
1,2,3,7,8,9-HxCDD	19408-74-3
Total HxCDD	34465-46-8
Total HpCDD	37871-00-4
1,2,3,4,6,7,8-HpCDD	35822-46-9
OCDD	3268-87-9
Furans	
2,3,7,8-TCDF	51207-31-9
Total TCDF	55722-27-5
1,2,3,7,8-PeCDF	57117-41-6
2,3,4,7,8-PeCDF	57117-31-4
Total PeCDF	30402-15-4
1,2,3,4,7,8-HxCDF	70648-26-9
1,2,3,6,7,8-HxCDF	57117-44-9
2,3,4,6,7,8-HxCDF	60851-34-5
1,2,3,7,8,9-HxCDF	72918-21-9
Total HxCDF	55684-94-1
1,2,3,4,6,7,8-HpCDF	67562-39-4
1,2,3,4,7,8,9-HpCDF	55673-89-7
Total HpCDF	38998-75-3
OCDF	39001-02-0

During each CPT run, the MM5 train is 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 is disassembled, and all train samples are 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, methylene chloride, and toluene, in that order. In cases where the same MM5 train handles the semivolatile analytes (SVOCs) 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 blowdown for extract volume reduction is significantly more difficult than the more volatile acetone and methylene chloride solvents, and provides an undesirable solvent front in the SVOC scan when incorrectly combined.

The particulate filter and front-half rinses will be Soxhlet-extracted using toluene for eighteen (18) hours. Dioxin and furan isotope dilution internal standards are added to the samples at this stage of the sample preparation. The dioxin and furan sampling surrogates are also added to the particulate filter samples at this point.

The XAD-2 resin tube samples and the solvent rinses of the back-half filter holder are handled in the same way as the front half samples, except that they are prepared separately and analyzed as a separate sample. These samples will also be extracted using toluene. Extractions are conducted using Soxhlet extraction apparatus and the extracts are reduced for analysis in the same way as the front half fractions. Dioxin and furan sampling surrogates will not be added to these samples during preparation since they have been applied to the XAD-2 resin prior to field sampling.

A spiking program will be applied to the Method 0023A 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 dioxin and furan 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 the various isomers of dioxins and furans.

- Dioxin and Furan 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. Final analyte concentrations in the samples are corrected for recovery losses of these spikes.

- Dioxin and Furan 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 an actual verification of recovery from the extract.

- Matrix Spike Compounds (back half and spiked resin blanks only)—These compounds are spiked onto separately prepared aliquots of the Method 0023A train 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.

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Method 0023A

Dioxins and Furans in Stack Gas

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]
Dioxins and Furans	Particulate Filter and Front Half Solvent Rinses	Chill with ice 4 °C ±2°C	30 days to extraction, 45 days from extraction to analysis
	XAD-2 Resin and Back Half Solvent Rinses	Chill with ice 4 °C ±2°C	30 days to extraction, 45 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 0023A for Semivolatile Organics in Stack Gas Emissions

Method 0023A 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[a]
Dioxins and Furans	MM5 Train (Particulate Filter and The Front-Half Filter Holder & Probe Solvent Rinses)	3	Soxhlet extraction, GC/MS (SW-8290, SW-0023A)	Isotope dilution internal standard spike	Every filter rinse and solvent combined sample	4	Blank train	1	4
				Carbon-13-labeled sampling surrogate spike	Every filter rinse and solvent combined sample	4			
	MM5 Train (XAD-2 and Back-Half Of The Filter Holder & Coil Condenser Solvent Rinses)	3	Soxhlet extraction, GC/MS (SW-8290, SW-0023A)	Isotope dilution internal standard spike	Every XAD-2 resin tube including blanks	7	Blank train	1	7
				Internal standard recovery spike	Every front-half sample including blanks and rinses	7			
				Spiked resin blank	Two XAD-2 resin tubes	2			
				Carbon-13-labeled sampling surrogate spike	Every XAD-2 resin tube including blanks	7	Trip blank	1	

[a] Total laboratory analyses includes all field samples collected and all laboratory and field QC samples that are analyzed. This number may not be calculated easily by adding the totals from the columns above; however, the total number presented represents the required total analyses for the sample and quality assurance analytical program.

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

Parameter/Method	QC Check	Frequency	Target Criteria	Corrective Action
Dioxin and Furans by High-Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) (Method 8290)	Mass scale calibration (tuning) using PFK	Prior to initial calibration, before each 12 hour shift	• Measured mass of PFK within 5 ppm of exact mass (m/z 3809760) • Resolving power at reduced accelerating voltage > 10,000 at m/z 3809760	Make necessary adjustments until conditions are met
	Retention time window verification and GC column performance (resolution check)	Prior to initial calibration, before each 12 hour shift	Resolution of 2,3,7,8-TCDD from nearest non-2,3,7,8-TCDD isomer • %Valley ≤ 25%	Correct according to the method
	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 ± 20% for unlabeled standards • %RSD ± 30% for labeled standards Other criteria • S/N ratios ≥ 10 • Isotopic ratios within control limits	(1) Repeat linearity check (2) If still unacceptable make necessary adjustments (3) Repeat linearity check
	Continuing Calibration	Beginning and end of each 12-hour shift	%Difference (%D) of RRF from ICAL average RRF • %D ≤ 20% for unlabeled standards • %D ≤ 30% for unlabeled standards Other criteria • Mass scale calibration within specifications • S/N ratios ≥ 10 • Isotopic ratios within control limits	(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
Dioxin and Furans by High-Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) (Method 8290) (continued)	Laboratory Control Sample	Once per sample batch (maximum 20 samples) Analyze after calibration standard and before the first sample	Within established control limits	(1) Flag data (2) Discuss in final report

Parameter/Method	QC Check	Frequency	Target Criteria	Corrective Action
	Internal standard spikes	Every sample (including method blanks and all QC samples)	%Recovery of internal standards • 40 to 130% for TCDD/DF through HxCDD/DF • 20 to 130% for HpCDD/DF and OCDD	Flag data
	Glass fiber filter surrogate and XAD-2 sampling surrogate spike recovery	Each filter spiked before preparation, each XAD-2 sample tube spiked before sampling	%Recovery of surrogates • 70 to 130% recovery	Flag data

SUMMARY OF FIELD QUALITY CONTROL SAMPLE REQUIREMENTS
COMPREHENSIVE PERFORMANCE TEST - 3 RUNS

Sample	QC Sample Type	Frequency	QC Sample Total
Method 0010/0023A Train Dioxins and Furans	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	1 set of train samples per test
	Spiked Resin Blanks	Two per CPT	2 XAD-2 resin tubes

METHOD 0010/0023A SPIKE COMPOUNDS AND EXAMPLE QUANTITY SPIKED

PCDD/PCDF Sampling Surrogates **(**applied to XAD-2 before field sampling)

PCDD/PCDF Sampling Surrogate Compounds	*Target Percent Recovery Range*	*Quantity Spiked*
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	70-130%	2 ηg
$^{13}C_{12}$-2,3,4,7,8-PeCDF	70-130%	2 ηg
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	70-130%	2 ηg
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	70-130%	2 ηg
$^{37}Cl_4$-2,3,7,8-TCDD	70-130%	2 ηg

PCDD/PCDF Isotope Dilution Internal Standards (applied at commencement of sample prep)

PCDD/PCDF Isotope Dilution Internal Standard Compounds	*Target Percent Recovery Range*	*Quantity Spiked*
$^{13}C_{12}$-2,3,7,8-Tetrachlorodibenzodioxin	40-135%	1 ηg
$^{13}C_{12}$-2,3,7,8-Tetrachlorodibenzofuran	40-135%	1 ηg
$^{13}C_{12}$-1,2,3,7,8-Pentachlorodibenzodioxin	40-135%	1 ηg
$^{13}C_{12}$-1,2,3,7,8-Pentachlorodibenzofuran	40-135%	1 ηg
$^{13}C_{12}$-1,2,3,6,7,8-Hexachlorodibenzodioxin	40-135%	1 ηg
$^{13}C_{12}$-1,2,3,6,7,8-Hexachlorodibenzofuran	40-135%	1 ηg
$^{13}C_{12}$-1,2,3,4,6,7,8-Heptachlorodibenzodioxin	40-135%	1 ηg
$^{13}C_{12}$-1,2,3,4,6,7,8-Heptachlorodibenzofuran	40-135%	1 ηg
$^{13}C_{12}$-Octachlorodibenzodioxin	40-135%	2 ηg

PCDD/PCDF Recovery Standard Compounds (applied to extracts prior to instrument analysis)

Dioxin and Furan Recovery Standard Compounds (applied to extracts prior to instrument analysis)	***Quantity Spiked***
$^{13}C_{12}$-1,2,3,4-Tetrachlorodibenzodioxin	2 ηg
$^{13}C_{12}$-1,2,3,7,8,9-Hexachlorodibenzodioxin	2 ηg

Method 0010/Method 0023A

Method for Determining Dioxins and Furans in Stack Gas

Sampling and Field Procedure for Dioxins/Furans in Stack Gases

Sample Name:	Modified Method 5 (Method 0010/Method 0023A) ▪ Dioxins/Furans
Sampler:	Stack Sampling Engineer
Process Sample Location:	Stack Sampling Platform
Sampling & Health & Safety Equipment:	Sampling and safety equipment is as follows: ▪ Method 0010/Method 0023A Sampling Train ▪ Organic-free DI water ▪ Aluminum foil ▪ 250 mL amber Boston Round - acetone probe rinse, the methylene chloride probe rinse, and toluene 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, methylene chloride, and toluene ▪ Squirt bottles for acetone, methylene chloride and toluene ▪ 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 $\leq$ 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, the five (5) Method 0023A sampling surrogates will be spiked onto the XAD-2. 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 referenced below. Pre-cleaned XAD-2 resin is commercially available (Supelco®) 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 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 from the Stack Sampling Engineer 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 eight (8) 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.

- Toluene Probe Rinse - As a separate rinse following the acetone and methylene chloride, the nozzle, probe, the front-half of the filter holder will be brushed and rinsed three times with toluene. The rinses will be placed into a separate 250 mL amber labeled Boston Round sample collection bottle with a Teflon®-lined lid only when other analyte types are being measured such as SVOCs or PAHs. Do not combine this toluene rinse with the acetone/methylene chloride solvent rinses under combined train conditions. These samples are to be handled separately in the laboratory preparation when combined trains are conducted.

- 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.

- Toluene Back Half of the Filter Holder and Coil Condenser glassware rinses - The back half of the filter holder, coil condenser, and connecting glassware will then be rinsed three times with toluene. The rinses will be placed in a separate 250 mL amber Boston Round sample bottle with Teflon®-lined lid only when other analyte types are being measured such as SVOCs or PAHs. Do not combine this toluene rinse with the acetone/methylene chloride solvent rinses under combined train conditions. These samples are to be handled separately in the laboratory preparation when combined trains are conducted.

- 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.

- 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 log book 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 dioxin and furan 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 remain open for the duration of actual train assembly. An XAD-2 trip blank should accompany each Method 0010/0023A shipment of samples to the laboratory.

Two spiked resin blanks of the XAD-2 resin are to be prepared at the time or 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 time for dioxins and furans is 30 days to extraction from the time of collection. All samples should be preserved on ice at approximately 4^{o}C.

Method References:

Method 0023A – “Sampling Method for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofuran Emissions from Stationary Sources”. 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.

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

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Analysis of Dioxins/Furans in Method 0023A Train Samples

Sample Name:	MM-5 Train for the collection of Dioxins and Furans The actual sample names given to fractions derived from the Method 0023A Train are: ▪ Front Half Composite - Particulate filter and the front half of the filter holder, probe and nozzle solvent rinses (Figure 2) ▪ Back Half Composite - XAD-2 resin tube and the back half of the filter holder and coil condenser solvent rinses (Figure 3)
Sample Holding Time:	Extract within 14 days of sample collection, and analyze within 40 days from extraction date.
Analysis Procedures:	<u>Front Half Composite</u> Place solvent probe and nozzle rinse, particulate filter and front half of the filter holder rinses into a Soxhlet extractor. Add semivolatile surrogate compounds and isotopically labeled dioxin/furan and PCB internal standards onto the filter portion prior to extraction. Extract for 18 hours using methylene chloride. Concentrate extract to 10 ml. Divide the extract into three equal portions: a 3.3 ml portion for semivolatile PIC analyses, a 3.3 ml portion for dioxin/furan analysis, and a 3.3 ml portion for PCB congeners and coplanars. Add the toluene probe rinse portion of the sample to the Soxhlet extractor. Extract the Front Half Composite a second time using toluene, for 18 hours. Concentrate the extract down to 10 mLs. Remove a 3.3 mL portion of the extract and combine it with the methylene chloride extract portion designated for dioxins and furans. Add semivolatile internal standards and dioxin/furan and PCB recovery standards to the appropriate extract portions, and analyze by Methods 8270, 8290, and 1668 for semivolatile PICs, dioxins/furans, and PCBs.

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 semivolatile surrogate compounds and isotopically labeled dioxin/furan and PCB internal standards onto the filter portion prior to extraction. Extract for 18 hours using methylene chloride.

Concentrate extract to 10 ml. Divide the extract into three equal portions: a 3.3 ml portion for semivolatile PIC analyses, a 3.3 ml portion for dioxin/furan analysis, and a 3.3 ml portion for PCB congeners and coplanars.

Add the toluene probe rinse portion of the sample to the Soxhlet extractor. Extract the Back Half Composite a second time using toluene, for 18 hours. Concentrate the extract down to 10 mLs. Remove a 3.3 mL portion of the extract and combine it with the methylene chloride extract portion designated for dioxins and furans.

Add semivolatile internal standards and dioxin/furan and PCB recovery standards to the appropriate extract portions, and analyze by Methods 8270, 8290, and 1668 for semivolatile PICs, dioxins/furans, and PCBs.

Condensate Composite

Place a one liter portion of the sample in a separatory funnel and add semivolatile surrogate compounds and PCB isotope dilution internal standards onto the sample. Perform a liquid-liquid extraction using Method 3510. Concentrate extract to 10 mL followed by a splitting of the sample 50:50 for semivolatiles and PCBs.

Add semivolatile internal standards and the PCB recovery standards to the appropriate extract and analyze by Methods 8270 and 1668 for semivolatile PICs and PCBs, respectively.

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.

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.

Method 0023A – “Sampling Method for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofuran Emissions from Stationary Sources” 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.

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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 (Dioxins and Furans)