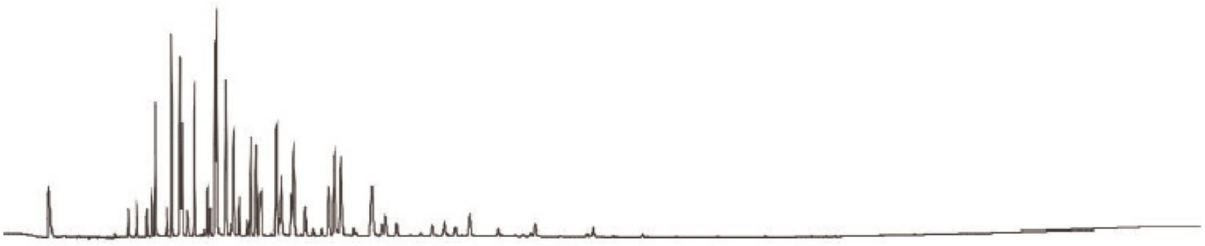

Aroclor 1242

Fundamentals of the Method for Analysis of PCB Congeners and Coplanars in Method 0010 Train Samples

MM5 Train Configuration for PCB Congeners and Coplanars (Methods 0010)

The configuration of the MM5 sampling train will be used to collect stack gas for an assessment of PCBs by Method 1668. PCB analytes are listed in Table 1.

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. In cases where the same MM5 train handles the semivolatile analytes and the dioxins and furans, a toluene probe rinse will 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. 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) will be Soxhlet-extracted using methylene chloride for eighteen (18) hours (Method 3540C). PCB isotope dilution internal standards will be added to the samples at this stage of the sample preparation. The PCB sampling surrogates also will be added to

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the particulate samples at this point after the methylene chloride extraction. A subsequent Soxhlet extraction will be conducted using toluene when a combined train is used, at which time the toluene probe rinse will be added to the sample. The methylene chloride extract will be prepared for analysis of PCB congener and coplanar analysis.

Table A
Summary of PCB Compounds for Analysis

TCL PCB Compounds for GC/MS Analysis by Method 1668		
PCB Homolog (Congener Group)	**IUPAC PCB Number**	**CAS Number**
Total Monochlorobiphenyl	N/A	27323-18-8
Total Dichlorobiphenyl	N/A	25512-42-9
Total Trichlorobiphenyl	N/A	25323-68-6
Total Tetrachlorobiphenyl	N/A	26914-33-0
Total Pentachlorobiphenyl	N/A	25429-29-2
Total Hexachlorobiphenyl	N/A	26601-64-9
Total Heptachlorobiphenyl	N/A	28655-71-2
Total Octachlorobiphenyl	N/A	55722-26-4
Total Nonachlorobiphenyl	N/A	53742-07-7
Decachlorobiphenyl	N/A	2051-24-3
PCB Congener (Coplanar)		
3,3',4,4'-Tetrachlorobiphenyl	PCB 77	32598-13-3
2',3,4,4',5-Pentachlorobiphenyl	PCB 123	65510-44-3
2,3',4,4',5-Pentachlorobiphenyl	PCB 118	31508-00-6
2,3,4,4',5-Pentachlorobiphenyl	PCB 114	74472-37-0
2,3,3',4,4'-Pentachlorobiphenyl	PCB 105	32598-14-4
3,3',4,4',5-Pentachlorobiphenyl	PCB 126	57465-28-8
2,3',4,4',5,5'-Hexachlorobiphenyl	PCB 167	52663-72-6
2,3,3',4,4',5-Hexachlorobiphenyl	PCB 156	38380-08-4
2,3,3',4,4',5'-Hexachlorobiphenyl	PCB 157	69782-90-7
3,3',4,4',5,5'-Hexachlorobiphenyl	PCB 169	32774-16-6
2,2',3,4,4',5,5'-Heptachlorobiphenyl	PCB 180	35065-29-3
2,2',3,3',4,4',5-Heptachlorobiphenyl	PCB 170	35065-30-6
2,3,3',4,4',5,5'-Heptachlorobiphenyl	PCB 189	39635-31-9

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

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and analyzed as a separate sample. These samples will also be extracted sequentially using methylene chloride, followed by toluene when a combined train is used. Extractions will be conducted using Soxhlet extraction apparatus (Method 3540C), and the extracts will be prepared for analysis in the same way as the front half fractions.

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. Sequential, base-neutral, acid-extractable extractions will be conducted on the condensate composite. The BNA extracts will be combined, reduced to an extract volume of one (1) milliliter and presented for the analyses of PCB congeners and coplanars by EPA Method 1668. 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 PCB target compounds, the overall method efficiency can be assessed. Four (4) 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

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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 PCBs.

- PCB 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.

- PCB 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 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.

Table 2 lists the specific isomers that will be used to spike the MM5 train and the quantities that will be applied.

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Table 2
MM5 Train Configuration (Method 0010) Spike Compounds and Quantity Spiked

Spike Type	Quantity Spiked
PCB Sampling Surrogate Compounds (applied to XAD-2 before field sampling)	
$^{13}C_{12}$-2,4',5-Trichlorobiphenyl	15 ηg
$^{13}C_{12}$-2,2',3,5',6-Pentachlorobiphenyl	15 ηg
$^{13}C_{12}$-2,2',4,4',5,5'-Hexachlorobiphenyl	30 ηg
PCBs Isotope Dilution Internal Standard Compounds (applied at commencement of sample prep)	
$^{13}C_{12}$-4-Monochlorobiphenyl	30 ηg
$^{13}C_{12}$-4,4'-Dichlorobiphenyl	30 ηg
$^{13}C_{12}$-2,4,4'-Trichlorobiphenyl	30 ηg
$^{13}C_{12}$-3,3',4,4'-Tetrachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,3,3',4,4'-Pentachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,3,4,4',5-Pentachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,3',4,4',5-Pentachlorobiphenyl	30 ηg
$^{13}C_{12}$-2',3,4,4',5-Pentachlorobiphenyl	30 ηg
$^{13}C_{12}$-3,3',4,4',5-Pentachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,3,3',4,4',5-Hexachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,3,3',4,4',5'-Hexachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,3',4,4',5,5'-Hexachlorobiphenyl	30 ηg
$^{13}C_{12}$-3,3',4,4',5,5'-Hexachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,2',3,3',4,4',5-Heptachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,2',3,4,4',5,5'-Heptachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,3,3',4,4',5,5'-Heptachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-Octachlorobiphenyl	30 ηg
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	30 ηg
$^{13}C_{12}$-Decachlorobiphenyl	30 ηg

Notes:
ηg Nanogram

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Method 0010 for PCBs in Stack Gas Emissions

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Method 0010 PCB Train 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]
PCB Congeners and Coplanars	MM5 Train (Particulate Filter and Front-Half Filter Holder and Probe Solvent Rinses)	3	Soxhlet extraction, GC/MS (SW-3540C, SW-3542, SW-8290, EPA Method 1668)	PCB isotope dilution internal standard spike	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, EPA Method 1668)	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 sample including blanks	7	Blank train	1	
				PCB isotope dilution internal standard spike	Every XAD-2 resin tube sample 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, EPA Method 1668)	PCB isotope dilution internal standard spike	Every impinger composite including blanks	6	Blank train	1	6
				MS/MSD	One set per test condition	2			

The PCB Sampling Surrogate Spike Compounds are:

Polychlorinated Biphenyl (PCB) Sampling Surrogate Compounds	*Target Percent Recovery Range*
$^{13}C_{12}$-2,4',5-Trichlorobiphenyl	40-160%
$^{13}C_{12}$-2,2'3,5'6-Pentachlorobiphenyl	40-160%
$^{13}C_{12}$- 2,2'4,4'5,5'-Hexachlorobiphenyl	40-160%

The PCB Cleanup Standard Compounds are:

Polychlorinated Biphenyl (PCB) Cleanup Standard Compounds	*Target Percent Recovery Range*
$^{13}C_{12}$-3,4,4',5-Tetrachlorobiphenyl	40-160%
$^{13}C_{12}$-2,3,3',5,5'-Pentachlorobiphenyl	40-160%

The PCB Isotope Dilution Internal Standard Spike Compounds and Laboratory Target Recovery Ranges are as follows:

Internal Standard Compound	**IUPAC PCB Number**	**Laboratory Target Recovery Limits (%)**
$^{13}C_{12}$-4-Monochlorobiphenyl	PCB 3	20-180%
$^{13}C_{12}$-4,4'-Dichlorobiphenyl	PCB 15	20-180%
$^{13}C_{12}$-2,4,4'-Trichlorobiphenyl	PCB 28	20-180%
$^{13}C_{12}$-3,3',4,4'-Tetrachlorobiphenyl	PCB 77	24-169%
$^{13}C_{12}$-2,3,3',4,4'-Pentachlorobiphenyl	PCB 105	21-178%
$^{13}C_{12}$-2,3,4,4',5-Pentachlorobiphenyl	PCB 114	21-178%
$^{13}C_{12}$-2,3',4,4',5-Pentachlorobiphenyl	PCB 118	21-178%
$^{13}C_{12}$-2',3,4,4',5-Pentachlorobiphenyl	PCB 123	21-178%
$^{13}C_{12}$-3,3',4,4',5-Pentachlorobiphenyl	PCB 126	21-178%
$^{13}C_{12}$-2,3,3',4,4',5-Hexachlorobiphenyl	PCB 156	26-152%
$^{13}C_{12}$-2,3,3',4,4',5'-Hexachlorobiphenyl	PCB 157	26-152%
$^{13}C_{12}$-2,3',4,4',5,5'-Hexachlorobiphenyl	PCB 167	26-152%
$^{13}C_{12}$-3,3',4,4',5,5'-Hexachlorobiphenyl	PCB 169	26-152%
$^{13}C_{12}$-2,2',3,3',4,4',5-Heptachlorobiphenyl	PCB 170	23-143%
$^{13}C_{12}$-2,2',3,4,4',5,5'-Heptachlorobiphenyl	PCB 180	23-143%
$^{13}C_{12}$-2,3,3',4,4',5,5'-Heptachlorobiphenyl	PCB 189	23-143%
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-Octachlorobiphenyl	PCB 194	20-180%
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	PCB 206	20-180%
$^{13}C_{12}$-Decachlorobiphenyl	PCB 209	20-180%

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Method 0010 (MM-5 Train) PCBs in Stack Gas Emissions

Method 0010 PCB 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)	PCB Congeners and Coplanars	Petri Dishes, 250 mL Boston-Round Amber Glass	Method 3542[a] Method 0010[b] Method 1668[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)	PCB Congeners and Coplanars	XAD-2 Resin Tubes, 250 mL Boston Round Amber Glass	Method 3542[a] Method 0010[b] Method 1668[c]	Collect 3 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 Field Blank, 1 Trip Blank, 1 Blank Train Back-Half Composite	6
MM5 Train Impinger Composite (Impinger composite and glassware solvent rinses)	PCB Congeners and Coplanars	1 Liter Amber Boston Round	Method 3542[a] Method 0010[b] Method 1668[c]	Collect 3 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 Blank Train Impinger Composite	4

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[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] Method 1668: "Toxic Polychlorinated Biphenyls by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry", Environmental Protection Agency, Washington, DC. Office of Water.

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Method 0010/Method 0023A

Method for Determining PCBs in Stack Gas

Sampling and Field Procedure for PCB Congeners and Coplanars in the Stack Gases in Stack Gases (MM-5 Train)

Sample Name:	Modified Method 5 (MM-5 Train) ▪ PCB Congeners and Coplanars
Sampler:	Stack Sampling Engineer
Process Sample Location:	Stack Sampling Platform
Sampling & Health & Safety Equipment:	Sampling and safety equipment is as follows: ▪ Method 0010 Sampling Train ▪ Organic-free DI water ▪ Aluminum foil ▪ 250 mL amber Boston Round - acetone probe rinse and the methylene chloride probe rinse ▪ 250 mL amber Boston Round - toluene probe rinse (when combined train is used) ▪ Glass Petri Dish - particulate filter ▪ XAD-2 Resin Tube ▪ 250 mL Amber Boston Round - back half rinse of the filter holder behind the particulate filter, coil condenser and connecting glassware with acetone and methylene chloride ▪ 250 mL Amber Boston Round - toluene rinse of the back half components (when combined train is used) ▪ 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, 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.

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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 three (3) PCB 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

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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 when a combined train is used, the nozzle, probe, the front-half of the filter holder will be brushed and rinsed three times with toluene. The rinses will be placed in a separate 250 mL amber labeled Boston Round sample collection bottle with a Teflon®-lined lid. Do not combine this toluene rinse with the acetone/methylene chloride solvent rinses. These samples are to be handled separately in the laboratory preparation.

- 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 Backhalf 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 when a combined train is used. The rinses will be placed in a separate 250 mL amber Boston Round sample bottle with Teflon®-lined lid. Do not combine this toluene rinse with the acetone/methylene chloride solvent rinses. These samples are to be handled separately in the laboratory preparation.

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- 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 separate 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 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 PCB 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 times for semivolatile samples is 14 days to extraction from the time of collection, while dioxins and furans is 30 days from the time of collection. All samples should be preserved on ice at approximately 4ºC.

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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.

Method 1668 - "Toxic Polychlorinated Biphenyls by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry", USEPA, October 4, 1995 Draft.

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

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Analysis of PCB Congeners and Coplanars in MM-5 Train Samples

Sample Name: MM-5 Train for the collection of PCB Congeners and Coplanars

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 front half of the filter holder rinses into a Soxhlet extractor. Add isotopically labeled PCB internal standards onto the filter portion prior to extraction. Extract for 18 hours using methylene chloride.

Concentrate extract to 10 ml. Extract the Front Half Composite a second time using toluene, for 18 hours. Concentrate the extract down to 10 mLs.

Add the PCB recovery standards and analyze by Method1668 for PCB congeners and coplanars.

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 PCB internal standards onto the filter portion prior to extraction. Extract for 18 hours using methylene chloride.

Concentrate extract to 10 ml. Analyze the extract for PCB congeners and coplanars.

Extract the Back Half Composite a second time using toluene, for 18 hours. Concentrate the extract down to 10 mLs.

Add the PCB recovery standards and analyze by Method 1668 for PCB congeners and coplanars.

Condensate Composite
Place a one liter portion of the sample in a separatory funnel and add the PCB isotope dilution internal standards onto the sample. Perform a liquid-liquid extraction using Method 3510. Concentrate extract to 10 mLs.
Add the PCB recovery standards to the extract and analyze by Method 1668 for PCB congeners and coplanars.

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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 1668 - "Toxic Polychlorinated Biphenyls by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry", Environmental Protection Agency, Washington, DC. Office of Water.

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Figure 1. MM5 Train Sample Handling and Extract Splitting Scheme for the Particulate Filter and Front Half of the Filter Holder, Nozzle, and Probe Solvent Rinses (Semivolatile POHCs, PCB Congeners and Coplanars, and Dioxins/Furans)

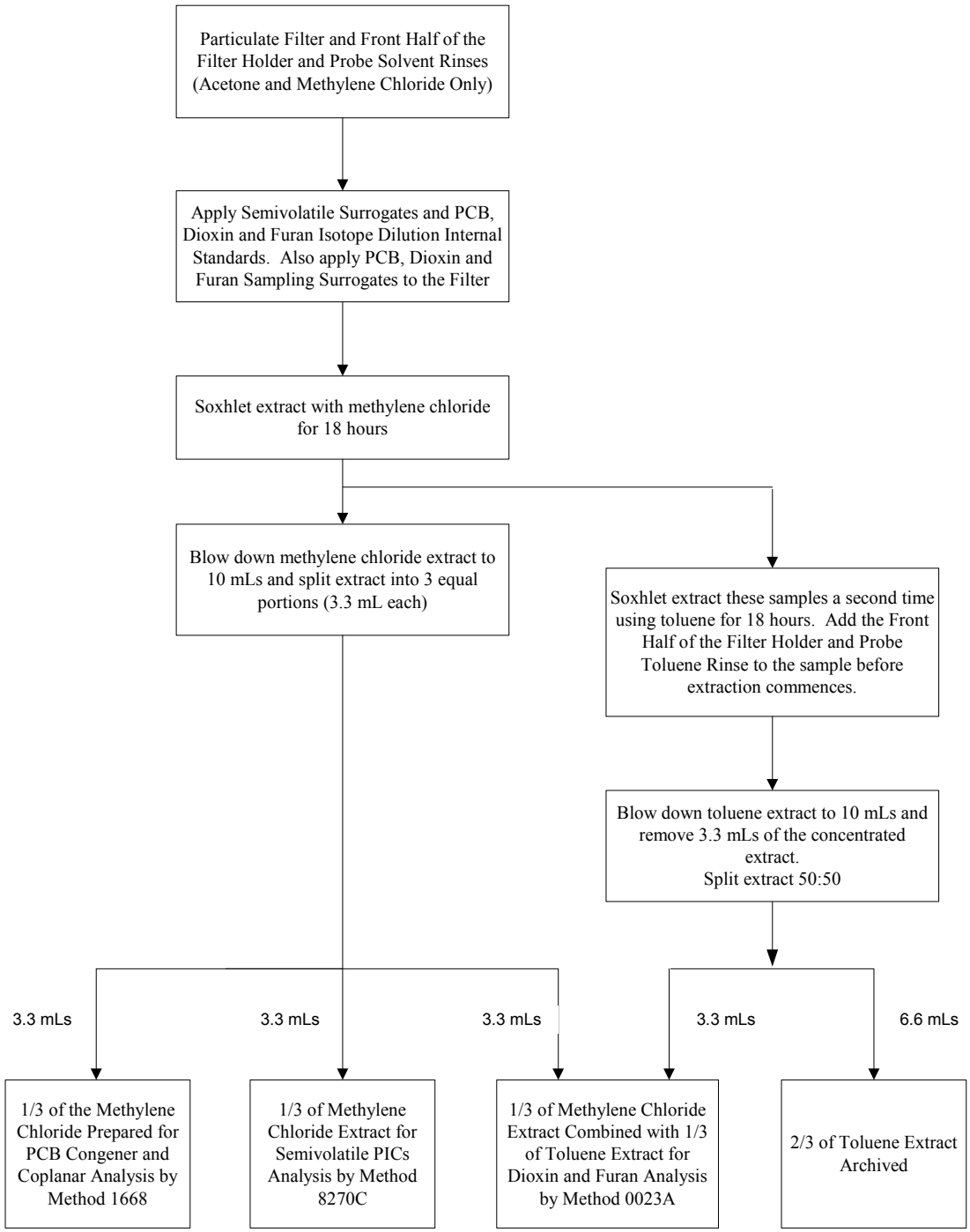

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Figure 2. MM5 Train Sample Handling and Extract Splitting Scheme for the XAD-2 Resin Tube and the Back Half of the Filter Holder and Coil Condenser Solvent Rinses (Semivolatile POHCs, PCB Congeners and Coplanars, and Dioxins/Furans)

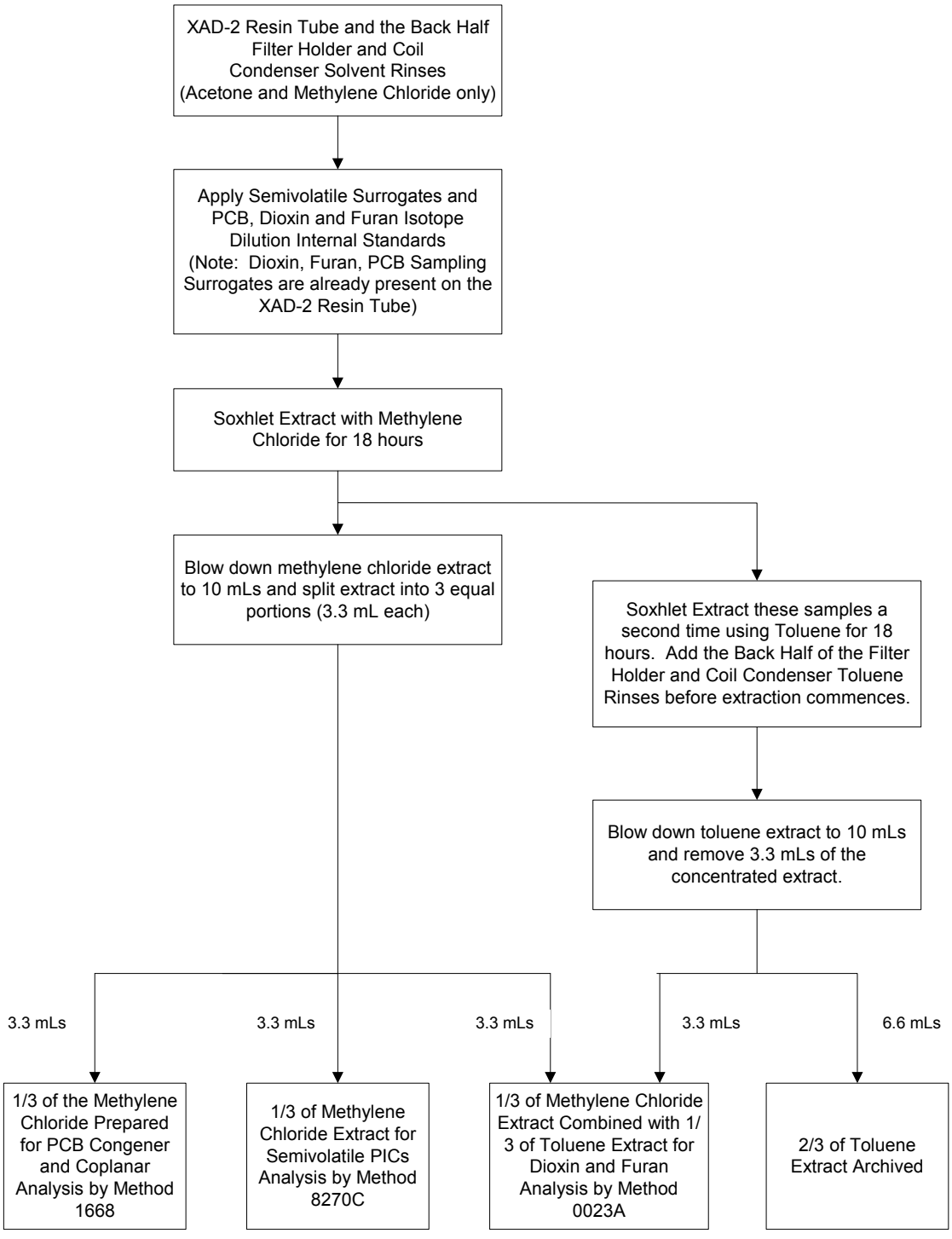

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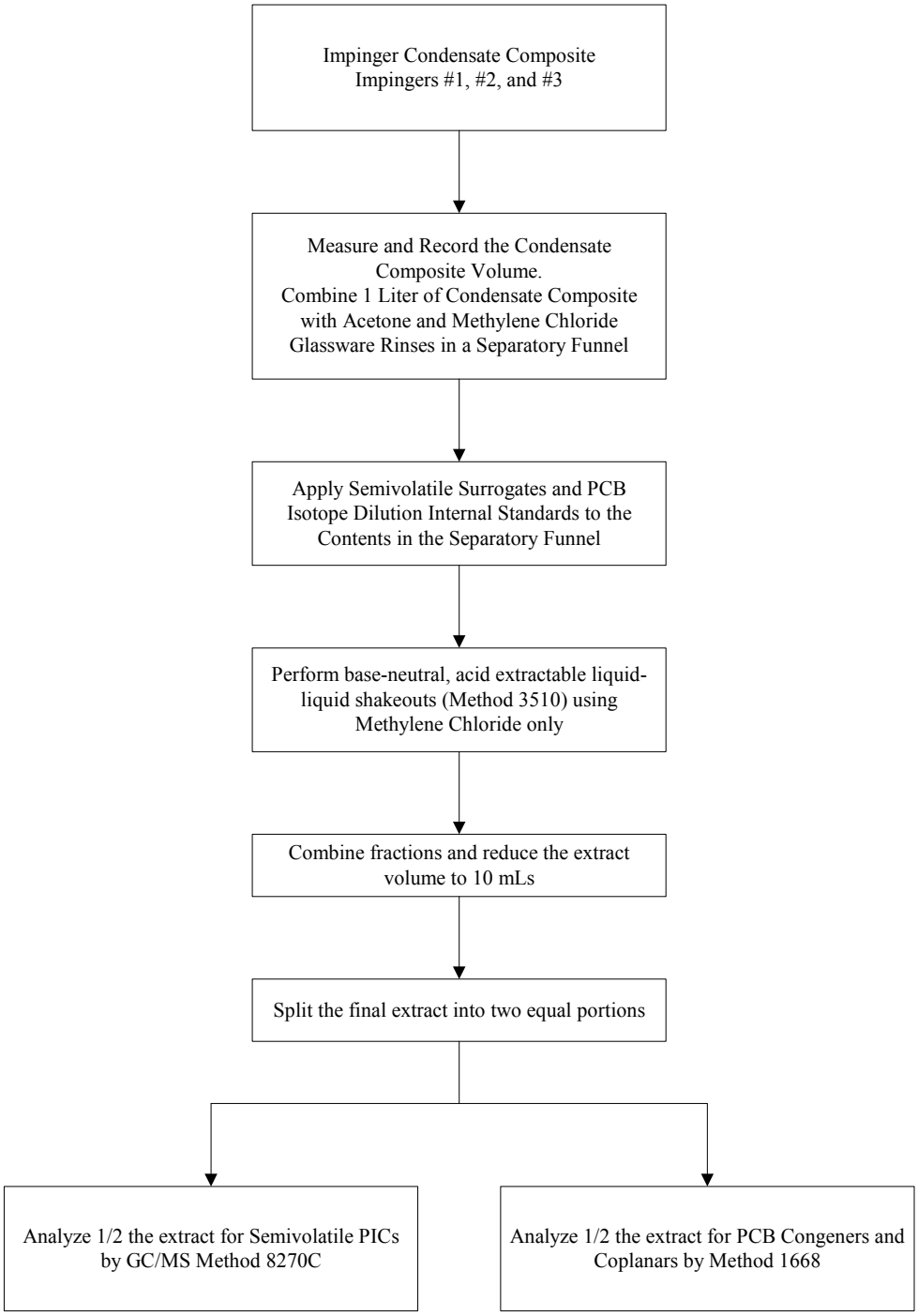
Figure 3. MM5 Train Sample Handling Scheme for the Impingers #1, #2, and #3 Condensate Composite and Glassware Solvent Rinses (Semivolatile PICs and PCB Congeners and Coplanars)



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