APPENDIX E-15a

CPT Sampling and Analysis Procedures

1. PROPOSITO

1.1 Establecer un protocolo para el muestreo de desperdicios industriales líquidos almacenados en tanques para almacenaje de desperdicios peligrosos en las facilidades de TAPI Puerto Rico Inc. Este protocolo asegura que las muestras tomadas son representativas del, material muestreado en cumplimiento con la reglamentación aplicable de la Ley de Conservación y Recuperación de Recursos y el permiso Parte B, PRD090613357, para el Almacenaje, Tratamiento y Disposición de Desperdicios Peligrosos.

1.2 Cualquier procedimiento descrito en este Procedimiento Normalizado de Trabajo puede sufrir desviaciones para utilizar métodos alternos aprobados par la EPA, guías y procedimientos que cumplan los objetivos de calidad de data del programa de muestreo.

2. ALCANCE Y APLICACION

2.1 Aplica a todo el personal de operaciones ambientales.

3. RESPONSABILIDADES

3.1 Es la responsabilidad del Operador Ambiental el llevar a cabo éste procedimiento.

3.2 Es la responsabilidad del Gerente del área de operaciones ambientales o designado asegurar que su personal lleven a cabo éste procedimiento.

4. DEFINICIONES

4.1 CFR: Código Federal de Reglamentaciones

4.2 USEPA (EPA): Agencia de Protección Ambiental de Estados Unidos

4.3 RCRA : Ley de Conservación y Recuperación de Recursos

5. REFERENCIAS

5.1 40 CFR 261

PROCEDIMIENTO

6.1 Procedimiento de Garantía de Calidad

6.1.1 Los envases de muestreos serán suplidos por el laboratorio analítico previo a la actividad de muestreo.

6.1.2 Registre la fecha, 1a hora, el nombre de la persona tomando 1a muestra, numero de identificación de la muestra, matriz de la muestra, parámetros analíticos, etc. en el libro de registros de campo.

6.2 Muestreo de Desperdicios Líquidos

6.2.1 Todo personal que realice un muestreo de desperdicios líquidos debe asegurarse que la muestra sea representativa. (Los desperdicios ha ser muestreados serán agitados en el tanque durante el muestreo. La agitación promueve una toma de muestra representativa).

6.2.2 Antes de tomar la muestra, drene la línea de muestreo. Recoja el volumen drenado y retórnelo al tanque que se esta muestreando o a otro tanque de desperdicios peligrosos. El material drenado será colectado en un envase can tapa utilizando un embudo y se mantendrá cerrado en todo momento excepto cuando se este añadiendo o removiendo el desperdicio.

6.2.3 No utilizar artefactos con componentes plásticos cuando se toman muestras de sustancias conteniendo materiales orgánicos.

6.2.4 No utilice artefactos con componentes de cristal cuando se toman muestras de sustancias conteniendo materiales altamente corrosivos.

6.2.5 Tomará la muestra a ser analizada e identificará la botella con una etiqueta que contenga la siguiente información:

a. Fecha / Hora
b. Nombre (ej.: Desperdicios acuosos, Desperdicio Orgánico, etc.)
c. Origen (ej.: V-450, etc.)
d. Nombre personal colectando la muestra
e. Número de muestra

6.2.6 Llene cada envase de muestra con el volumen requerido de desperdicio líquido. Las muestras ha ser analizadas para orgánicos volátiles serán tomadas de tal forma de que no haya espacio o burbuja de aire en el envase de muestra ("vial").

6.2.7 Entregar la muestra al representante del laboratorio ambiental externo y completar la cadena de custodia.

6.2.8 Verificar que los análisis de la muestra contiene los siguientes parámetros:

a. Valor de Calentamiento
b. Total de Cloro Orgánico
c. Total Ceniza
d. Viscosidad

e. pH
f. Gravedad Específica
g. Arsénico
h. Cadmio
i. Cromio
j. Mercurio
k. Berilio
l. Plomo

7. HISTORIAL DE CAMBIOS

Fecha	*Revisión*	*Descripción*
10/14/98	00	Nuevo
01/26/01	01	Se eliminaron los pasos 6.2.3 y 6.2.8. Se modificaron los pasos 6.2.7, 6.3.1 y 6.3.2. Se le añadió Número de libreta de referencia a los Anejos III y IV.
01/31/03	02	Se revisó debido a su fecha de expiración.
	03	Se modificó el formato y se cambió logo y nombre de la compañía. No hubo cambios en el contenido.
04/07/11	04	Se modificó el formato. Se cambió el contenido en su totalidad.

Sample name: Stack Gas Method 26A (PM, HX, X_2)

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 5 sampling train components assembled as shown in Figure 1 of this table; amber glass sample jars with Teflon-lined lids; pre-weighed Teflon mat particulate filters [or quartz fiber filters if the stack gas temperature exceeds 410°F (210°C)] in sealed petri dishes; balance; glass graduated cylinder; ambient air conditioning tube containing Ascarite II.

Frequency: Continuous sampling for a minimum of 60 minutes during each test run.

Procedure Summary: This procedure is used to collect samples of stack gas particulate matter, hydrogen halides, and halogens. The stack gases are sampled under isokinetic conditions to collect particulate matter on a filter, hydrogen halides and halogens in absorbing solutions, and to determine the stack gas flow rate. The amount of particulate matter collected is determined by weighing and the halide content of the absorbing solutions is determined by ion chromatography.

The Method 26A sampling train (Figure 1) includes a glass probe nozzle, a heated sample probe with borosilicate or quartz glass probe liner, a heated filter holder with Teflon filter support, a condensate knockout impinger (optional), two acid impingers, an empty impinger (optional), two alkaline impingers, and a silica gel impinger. All impingers are placed in an ice bath. An air tight gas pump, flow rate meter, and dry gas meter complete the train.

All train components, reagents, and cleaning solutions are prepared according to the procedures specified in the methods referenced below. The pretest preparations include soaking the train glassware in hot (≥50°C) soapy water, rinsing it three times each with hot water, distilled/deionized water, and acetone, and allowing it to dry in air. When glassware is dry, all openings are covered with Teflon film until sample train assembly.

The sampling train is assembled in a clean area and train components are handled in a manner that minimizes potential for contamination from air or by direct contact. A carefully inspected Teflon mat (or quartz fiber) filter is placed in the filter holder. When sampling a stack gas that has high moisture content, a condensate knockout impinger containing 50 ml of 0.1N sulfuric acid may be included as the first impinger in the train. The next two impingers each contain 100 ml of 0.1N sulfuric acid. An optional empty impinger may be placed after the sulfuric acid impingers to collect any liquid carryover. The next two impingers each contain 100 ml of 0.1N sodium hydroxide solution. The last impinger is filled with 200 to 300 grams of indicating silica gel.

Stack sampling point locations are determined in accordance with Method 1. An initial traverse is made with a pitot tube at each sample point following Method 2 to establish stack gas velocity profile, temperature, and flow rate, and to check for cyclonic flow (cyclonic flow is checked only on the first day of testing). An Orsat analyzer or continuous emissions monitoring system (CEMS) is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to either Method 3, 3A, or 3C procedures. The stack gas moisture content is determined according to Method 4.

Pretest and post-test leak checks, isokinetic sampling rate, filter change outs (if needed), and data recording are performed according to Method 5 procedures.

The stack gas sampling is conducted following the general procedure given in Method 5 with the exception that the temperature of the sample probe and filter assembly is maintained at a minimum of 248°F (instead of 248°F ± 25°F). As required by Method 5, the isokinetic sampling rate is maintained within ±10% of 100%.

After sampling, the probe is removed from the stack and the post-test leak check is performed. The filter surface is inspected for visible moisture which if present, is recovered by the procedure specified in Section 8.1.6 of Method 26A. While visible moisture is not expected to be present under normal sampling conditions, this procedure involves attaching the ambient air conditioning tube to the nozzle tip and operating the train for the time(s) needed to evaporate the moisture and to capture the gases in the impinger solutions.

The sampling train is allowed to cool and when the probe temperature is safe to handle, it is disconnected from the train and the inlet to the filter is cleaned and capped. The probe and the filter/impinger assembly are transported to the sample recovery area. The sample recovery and sampling train cleanup procedures are summarized below.

- Particulate Filter -- The particulate matter filter is recovered from its holder and placed in its original petri dish (Container 1). Any PM and/or filter fibers that adhere to the filter holder or gasket are also recovered and placed in the petri dish, which is then sealed with Teflon tape, placed in a plastic bag.
- Front-half Rinse -- The internal surfaces of the nozzle, probe liner, and front half of the filter holder are brushed and rinsed three times with acetone. All rinses are placed in a sample bottle (Container 2) and the final liquid level is marked on the bottle.
- Acid Impinger Liquid -- The liquid contents of the condensate knockout impinger (if present), the two sulfuric acid impingers, and the carryover impinger (if present), are measured to the nearest milliliter or weighed to the nearest 0.5 g and placed into a sample bottle (Container 3). Any condensate found in the back half of the particulate filter holder or the connecting glassware (or flexible tubing) is also measured or weighed and transferred to Container 3. The back half of the filter holder, acid impingers and all connecting glassware are rinsed with deionized water, and the rinses are added to Container 3. The final liquid level is marked on the sample container. Any color or film observed is noted on the sample recovery sheet.
- Alkaline Impinger Liquid -- The liquid contents of the sodium hydroxide impingers are measured to the nearest milliliter or weighed to the nearest 0.5 g and placed into a sample bottle (Container 4). The sodium hydroxide impingers and all connecting glassware are rinsed with deionized water, and the rinses are added to Container 4. A sodium thiosulfate solution is added to this container in the amount of 0.7 mg per ppm of halogen anticipated in the stack gas times the stack gas sample volume in dscf, (i.e., 0.7 mg/ppm-dscf). The container is sealed, shaken to mix and the final liquid level marked on the sample container. Any color or film observed is noted on the sample recovery sheet.

- Silica Gel -- The silica gel contents of the last impinger are weighed to the nearest 0.5 g. The color and condition of the silica gel is noted on the sample recovery sheet.

A schematic diagram of the above sample recovery and cleanup procedures is provided in Figure 2 of this table. Following sample recovery, the glassware may be reused at the same sampling location.

Samples of the absorbing reagent solutions equivalent to the amounts used in the sampling train are collected once during the test for reagent blanks. Deionized water from the wash bottle is added to these samples to bring the volumes up to the corresponding recovered sample volumes. The same ratio of sodium thiosulfate solution used for Container 4 is added to the sodium hydroxide solution blank. Blank samples of the deionized water and acetone in the wash bottles are also collected.

All of the sample containers are assigned numbers and labeled with the date and test-run number. The samples are turned over to the Sample Coordinator who records the appropriate data in the field logbook and packs the samples in insulated cold chests. Samples are stored in the sample holding area separate from the container supply area.

References: “Method 26A - Determination of Hydrogen Halide and Halogen Emissions from Stationary Sources – Isokinetic Method"

“Method 1 - Sample and Velocity Traverses for Stationary Sources”

“Method 2 – Determination of stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)”

“Method 3 – Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight”

“Method 4 – Determination of Moisture Content in Stack Gases”

“Method 5 – Determination of Particulate Emissions from Stationary Sources”

Source: Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.

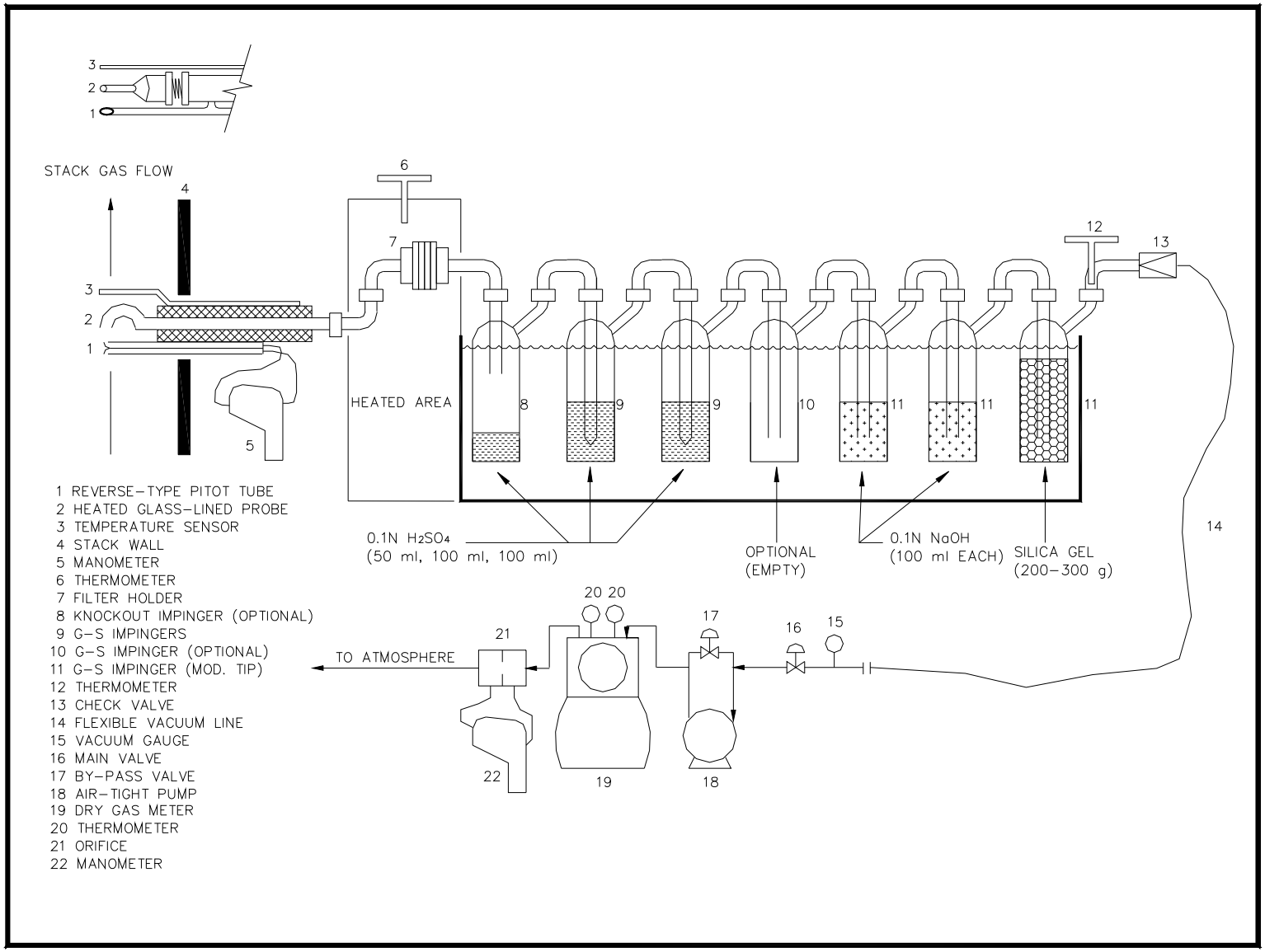

Figure 1. Method 26A Sample Train for Particulate Matter, Hydrogen Halide, and Halogen

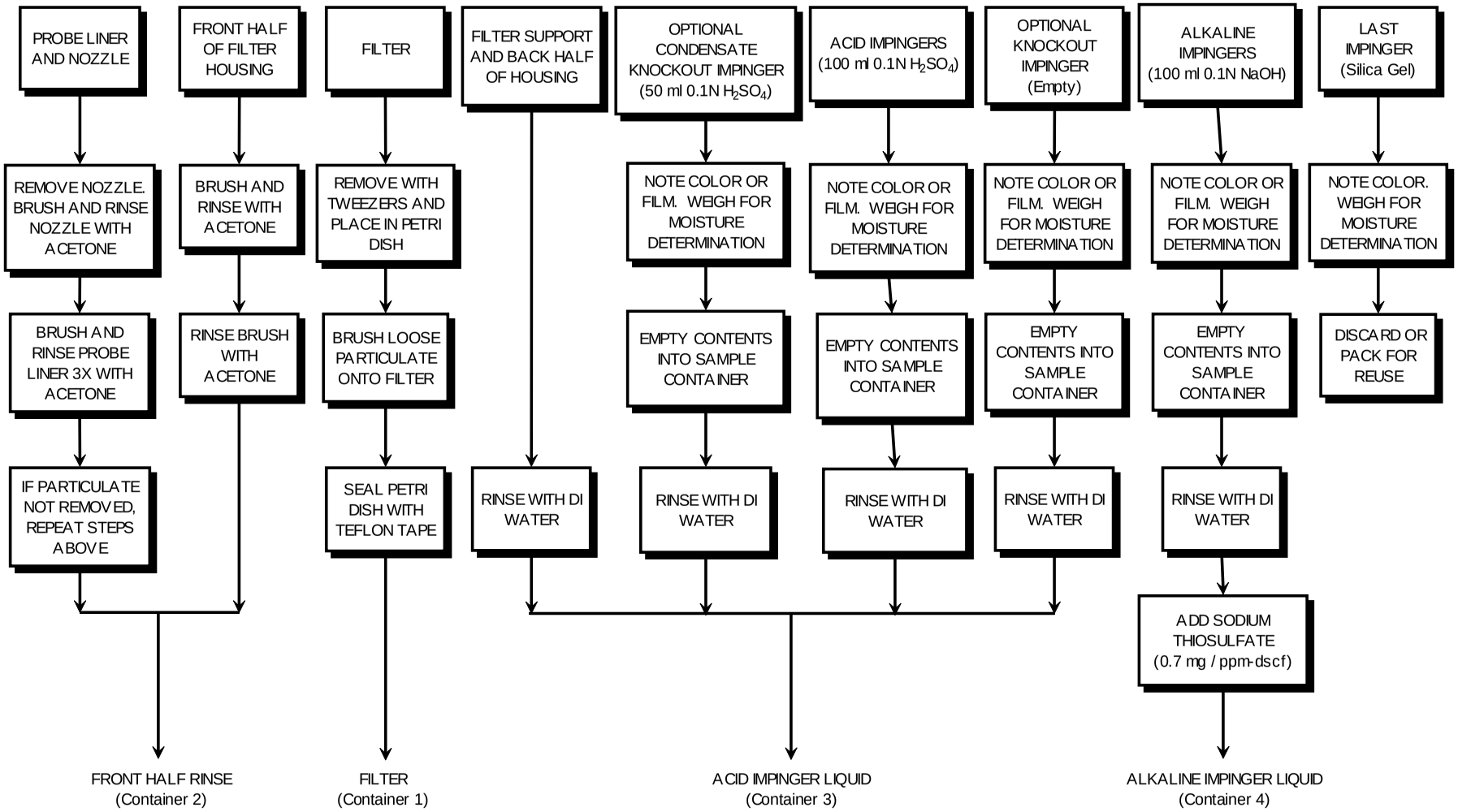

Figure 2. Method 26A Particulate Matter, Hydrogen Halide, and Halogen Sample Recovery

Sample name: Stack Gas Method 29 (MM, No Hg, No PM)

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 29 multiple metals sampling train as shown in Figure 1 of this table; amber glass sample jars with Teflon-lined lids; low-metals content particulate filters sealed in petri dishes; balance; glass graduated cylinder.

Frequency: Continuous for 120 minutes during each test run.

Procedure Summary: Gases are sampled isokinetically to collect the metals Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, V, and Zn on a particulate filter and in an absorbing solution. [Note: Mercury (Hg) and particulate matter (PM) are not included in this analyte list.] The filter and absorbing solutions are recovered for subsequent metals analysis by inductively coupled plasma emission spectroscopy (ICP), atomic absorption spectroscopy (AAS), or graphite furnace AAS (GFAAS).

The Method 29 multiple metals train includes a heated sample probe with borosilicate or quartz nozzle and liner, a heated particulate filter holder with Teflon filter support, a condensate knockout impinger (optional), two acidified hydrogen peroxide impingers, and a silica gel impinger. All impingers are placed in an ice bath. An air tight gas pump, dry gas meter, and manometer complete the train.

All train components, reagents, and cleaning solutions are prepared according to the procedures specified in the methods referenced below. The pretest preparations include rinsing the train glassware with hot tap water, washing with hot soapy water, rinsing three times each with tap water and distilled/deionized water (ASTM D1193-99, Type II). The glassware is then soaked in 10% (v/v) nitric acid solution for a minimum of four hours, rinsed three times with distilled/deionized water, a final rinse with acetone, and allowed to air dry. All glassware openings are covered with Teflon film until sample train assembly.

The sampling train is assembled in a clean area at the test site and the train components are handled in a manner that minimizes contamination from air or by direct contact. A clean and inspected filter is placed in the filter holder. The first impinger is initially empty and serves as a condensate knockout impinger. (This impinger may be eliminated if the moisture to be collected is less than 100 ml.) The next two impingers each contain 100 ml of 5% HNO_3/10% H_2O_2 solution. The last impinger contains 200 to 300 g of indicating silica gel.

Stack sampling point locations are determined in accordance with Method 1. An initial traverse is made with a pitot tube at each sample point following Method 2 to establish stack gas velocity profile, temperature, and flow rate, and to check for cyclonic flow (cyclonic flow is checked only on the first day of testing). An Orsat analyzer is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to Method 3 procedures. The stack gas moisture content is determined according to Method 4.

The sample train is operated according to Method 5 procedures for pretest and post-test leak-checks, isokinetic sampling rate, and data recording. The train leak rate must be the less than 0.02 cfm, or less than 4% of the average sampling rate. The sampling rate is

adjusted during the test to maintain isokinetic conditions at the probe nozzle within ± 10% of 100%.

After sampling, the probe is removed from the stack and a post-test leak check (Method 5) is conducted. The probe nozzle is wiped to remove PM and covered loosely. After cooling, the probe is wiped to remove external PM, disconnected from the train, and both ends are capped. The probe and filter box-impinger assemblies are transported to the sample recovery area. The sample recovery and sample train cleanup are summarized below.

- Particulate Filter – A non-metallic tool is used to remove the particulate filter from its holder and place it in the original petri dish (Container 1). A nylon bristle brush is used to remove any PM or filter fibers from the filter gasket or the holder onto the filter, and the petri dish is sealed with tape and placed in a plastic bag.
- Front Half Rinse -- The internal surfaces of the nozzle, probe, and front half of the filter holder are cleaned by rinsing, brushing, and final rinsing with exactly 100 ml of 0.1N nitric acid. All rinses are placed into an sample bottle (Container 3). [Note: There is no Container 2 (Acetone Rinse) because the collection of PM samples is not included in this procedure.]
- Acidified Peroxide Impingers -- The liquid contents of the condensate knockout impinger (if used) and the two HNO_3/H_2O_2 impingers are measured to the nearest milliliter or weighed to the nearest 0.5 g and placed into a sample bottle (Container 4). The back half of the particulate filter holder is inspected for condensate, which if observed, is measured and/or weighed as described above and transferred to Container 4. The back half of the filter holder, the filter support, the optional condensate knockout impinger (if used), and the two HNO_3/H_2O_2 impingers, and all connecting glassware are rinsed with exactly 100 ml of 0.1N nitric acid, and the rinses are added to Container 4. [Note: There are no Containers 5A, 5B or 5C because the collection of mercury samples is not included in this procedure.]
- Silica Gel -- The silica gel contents of the last impinger are weighed to the nearest 0.5 g. Note the color and condition of the silica gel on the sample recovery sheet.

A schematic diagram of the above sample train recovery and cleanup procedures is provided in Figure 2 of the table. Following sample recovery, the train may be reused at the same sampling location.

The following reagent blank samples are collected once during the test program: one unused particulate filter; 300 ml of the 0.1N nitric acid solution; 100 ml of the water used in sample recovery; and 200 ml of the nitric acid/hydrogen peroxide reagent solution.

Once during the test program, two complete blank trains are assembled in a clean area, brought up to operating temperatures, leak tested, and allowed to sit idle (no sample gas flow) for the duration of one sampling run. The train samples are recovered as described above and subsequently used in matrix spike and matrix spike duplicate analyses to evaluate conformance with analytical data quality objectives.

All of the sample containers are assigned numbers and labeled with the date and test-run number. The samples are turned over to the Sample Coordinator who records the appropriate data in the field logbook and pack the samples in insulated cold chests. Samples are stored in the sample holding area separate from the container supply area.

References: “Method 29 - Determination of Metals Emissions form Stationary Sources."

“Method 1 - Sample and Velocity Traverses for Stationary Sources”

“Method 2 – Determination of stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)”

“Method 3 – Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight”

“Method 4 – Determination of Moisture Content in Stack Gases”

“Method 5 – Determination of Particulate Emissions from Stationary Sources”

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

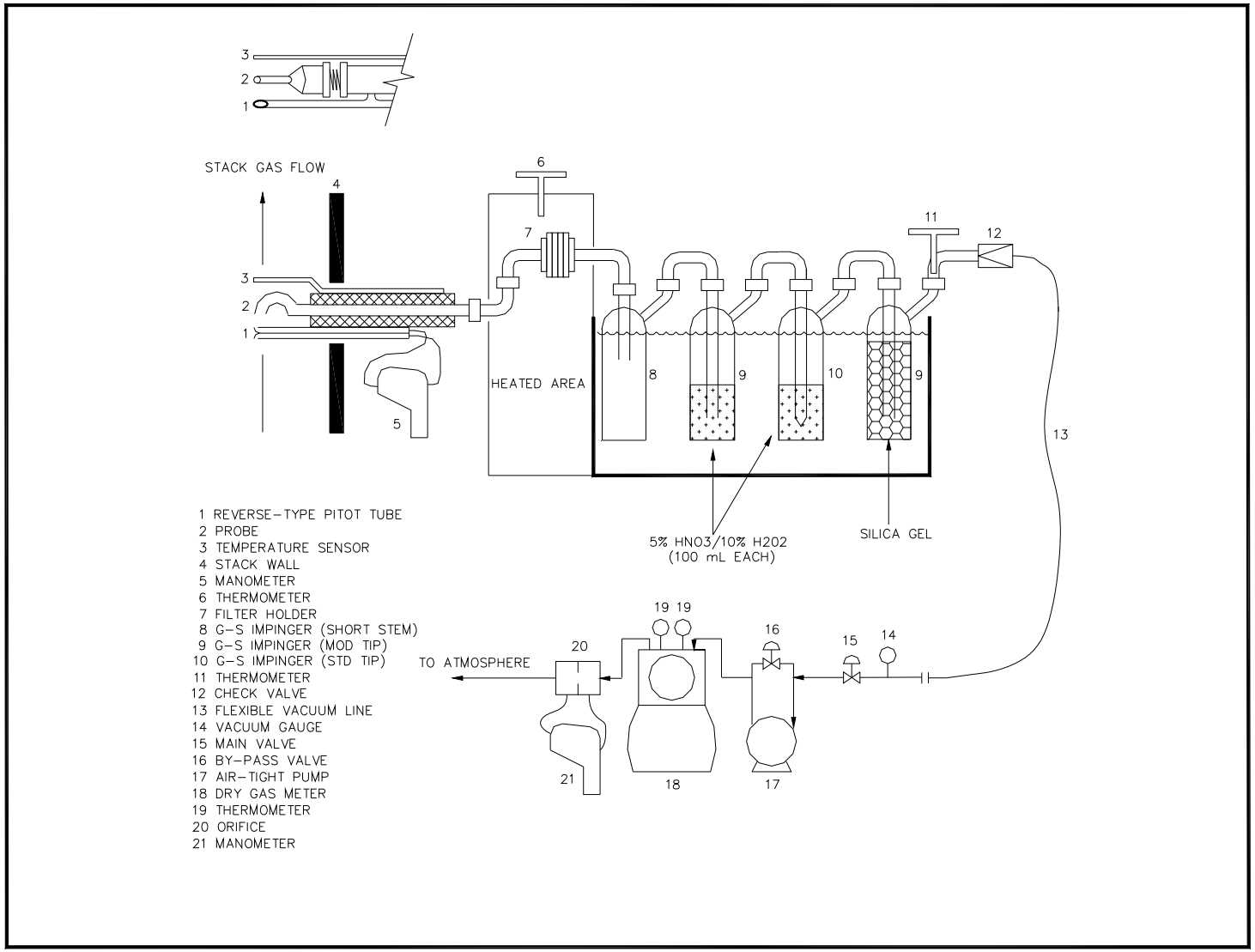

Figure 1. Method 29 Sample Train for Multiple Metals (No Hg) (No Particulate)

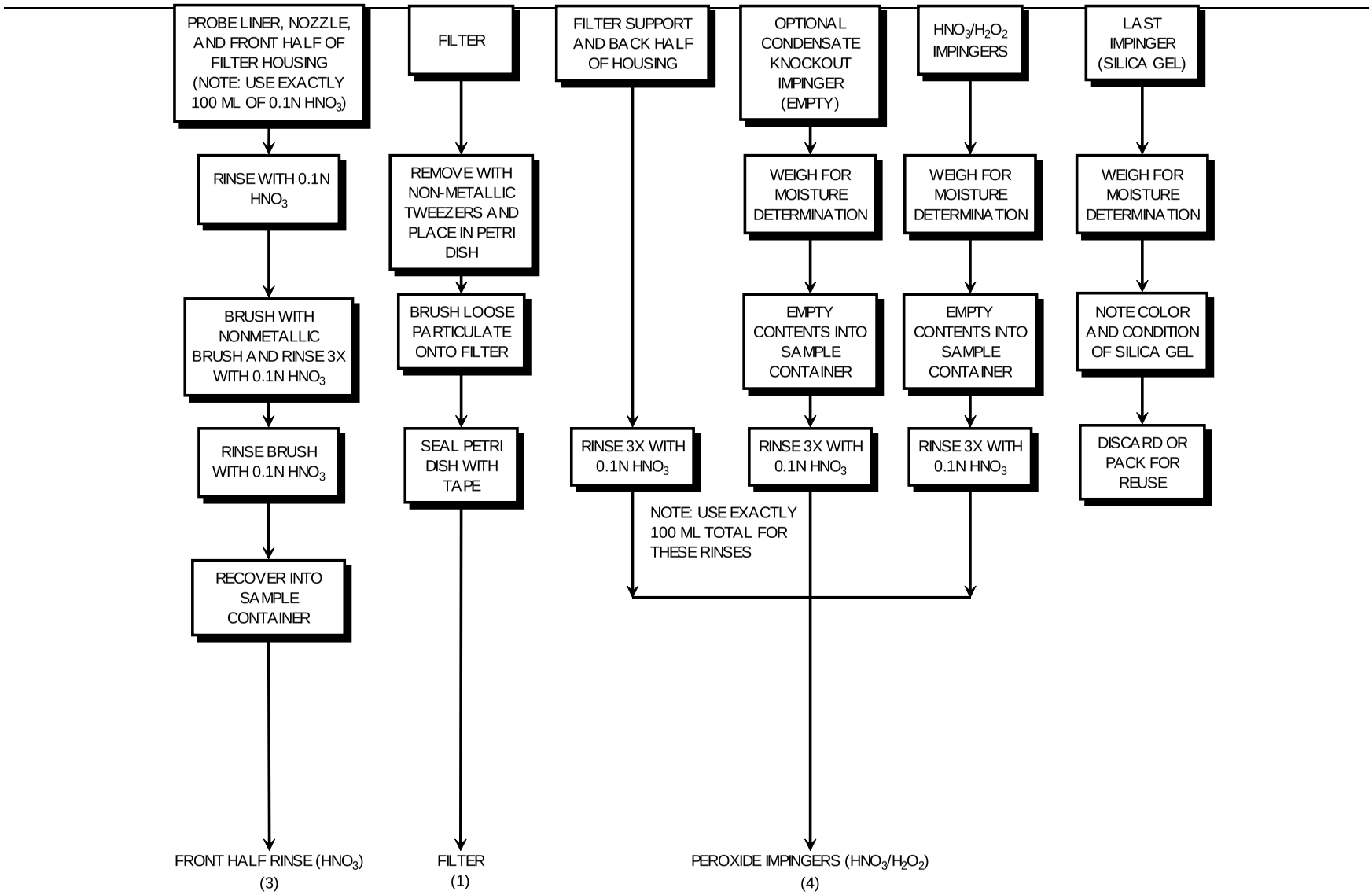

Figure 2. Method 29 Multiple Metals (No Hg) (No Particulate) Sample Recovery

Sample name: Stack Gas Method 23 (DF)

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 23 sampling train as shown in Figure 1 of this table; clean and inspected glass fiber particulate filters sealed in petri dishes; prepared XAD-2 resin traps; amber glass sample bottles with Teflon-lined lids; cleaned aluminum foil; scale balance; glass graduated cylinder.

Frequency: Continuous to collect a minimum of 106 dry standard cubic feet of sample during each sampling run.

Procedure Summary: Stack gases are sampled isokinetically to collect PCDD/PCDFs on a particulate filter and on adsorbent resin. The train component samples are recovered in the field and shipped to an analytical laboratory where the mass amounts of PCDD/PCDFs present are subsequently determined by extraction, concentration, and analysis by high resolution gas chromatograph/high resolution mass spectrometry (HRGC/HRMS).

The Method 23 train includes a glass probe nozzle, heated sample probe with a borosilicate or quartz probe liner, a heated filter holder with Teflon filter support, a water cooled condenser, a water cooled adsorbent resin trap, a condensate knockout impinger, two water impingers, an empty impinger, and a silica gel impinger. A water pump is provided to circulate ice bath water through the condenser and around the trap. All of the impingers are placed in an ice bath. An air tight gas pump, dry gas flow meter, and flow rate meter complete the train.

All train components, reagents, and cleaning solutions are prepared according to the procedures specified in the methods referenced below. The pretest preparations include soaking the train glassware in hot (50°C) soapy (Alconox or equivalent) water, rinsing it three times each with hot water, distilled/deionized water, and drying in an oven for 2 hours at 400 °C. After cooling, the glassware is rinsed 3 times each with pesticide grade methylene chloride and pesticide grade toluene. When glassware is dry, all openings are covered with cleaned aluminum foil or clean glass plugs until sample train assembly.

The particulate filters and the XAD-2 adsorbent resin are prepared according to Method 23 procedures summarized as follows:

- The filters are cleaned by soxhlet extraction using toluene and dried in a stream of clean nitrogen. The filters are stored in a glass petri dish sealed with Teflon tape.
- XAD-2 resin is cleaned by HPLC water rinses followed by soxhlet extractions with HPLC water, methanol, and methylene chloride then dried using a flow of inert gas. An extract from a portion of the prepared XAD-2 resin is analyzed to confirm that it is free of significant background contamination. As an alternate to in-house preparation, resin that is certified clean by laboratory analysis may be purchased. The resin is stored at less than 120 °F at all times and re-cleaned if not used within 4 weeks.

- The adsorbent trap is loaded with approximately 40 g of the XAD-2 resin and the open end of the trap is packed with clean glass wool. The adsorbent traps are spiked with isotope labeled PCDD/PCDF surrogate standards. The ends of the adsorbent trap are capped and the traps are wrapped in aluminum foil, sealed in a plastic bag, and stored in an insulated cold chest. The resin traps are used within two weeks of spiking.

The sampling train is assembled in a clean area at the test site and the train components are handled in a manner that minimizes the potential for contamination from air or by direct contact. The train glassware may be rinsed before using with acetone and methylene chloride. A particulate filter is placed in the filter holder. Before each sampling run, the Sample Coordinator supplies an XAD-2 adsorbent trap to the sampling team, who measures and records the weight of the trap to the nearest 0.5 gram and installs the trap in the train. Impingers 1 and 4 are initially empty, impingers 2 and 3 each contain 100 ml of distilled deionized water, and impinger 5 contains 200 to 300 grams of indicating silica gel. All impingers are weighed separately to nearest 0.5 gram and installed in the sample train. No sealant grease is used in assembling the train.

Stack sampling point locations are determined in accordance with Method 1. An initial traverse is made with a pitot tube at each sample point following Method 2 to establish stack gas velocity profile, temperature, and flow rate, and to check for cyclonic flow (cyclonic flow is checked only on the first day of testing). An Orsat analyzer is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to Method 3 procedures. The stack gas moisture content is determined according to Method 4. Pretest and post-test leak checks, isokinetic sampling rate, filter change outs (if needed), and data recording are performed according to Method 5 procedures.

The stack gas sampling is conducted following the procedures given in Method 23. The sample probe exit and filter assembly temperatures are maintained at 248°F ± 25°F, and the gas sample entering the adsorbent module is maintained less than 68 °F. The sampling rate is adjusted during the test to maintain isokinetic conditions at the probe nozzle within ± 10% of 100%.

After sampling, the probe is removed from the stack and a post-test leak check is conducted. The probe nozzle is wiped to remove PM and covered with aluminum foil. After cooling, the probe is wiped to remove external PM, disconnected from the train, and both ends are capped. The probe and filter box-impinger assemblies are transported to the sample recovery area. The sample recovery and sample train cleanup procedures are summarized below.

- Particulate Filter - The filter is removed from its holder and placed into its original petri dish (Container 1) which is sealed with Teflon tape and sealed in a plastic bag.
- XAD-2 Adsorbent Resin Trap - The XAD-2 adsorbent resin trap is removed from the train, and both ends are capped. The XAD-2 adsorbent resin trap is then weighed to the nearest 0.5 gram and the weight recorded. The trap is then labeled, covered with aluminum foil, sealed in a plastic bag and stored in an insulated cold chest.
- Front Half and Back Half Rinses - The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware are brushed and rinsed three times each with acetone and methylene chloride. The rinses are placed into a glass sample bottle (Container 2). The back half of the filter holder, the filter support, transfer

line (if used) and condenser (if separate from trap) are rinsed three times with acetone and then soaked with three separate portions of methylene chloride for 5 minutes each. The rinses and soaks are added to the front half rinses in Container 2.

- Toluene Rinse - The front half and back half methylene chloride rinse and soak procedures described above are repeated using toluene as the solvent. The rinse and soak liquids are collected in a separate glass sample bottle (Container 3). (Note: The knockout impinger, impingers 2, 3, and 4 and connecting glassware are not included in this procedure.)
- Impinger water - The contents of the knockout impinger and impingers 2, 3, and 4 are measured to the nearest milliliter or weighed to the nearest 0.5 g and observed for the presence or absence of film or color, which is recorded on the sample recovery sheet. The impinger water is then discarded.
- Silica Gel - The silica gel contents of impinger 5 are weighed to the nearest 0.5 g.

A schematic diagram of the above sample train recovery and cleanup procedures is provided in Figure 2 of this table. Following sample recovery, the train may be used at the sample sampling location.

Field blank samples are collected from a blank sampling train once during the field test program. A complete sample train including particulate filter and resin trap is assembled from glassware that has not been used to collect any field samples during the test program. The train is taken to the sampling area, brought up to operating temperature, leak tested, allowed to sit idle (no sample gas flow) for the duration of one sampling run, and the leak test is repeated. The field blank samples are recovered from the train as described above for the actual test samples.

Reagent blank samples are collected are collected from each of the acetone, methylene chloride, and toluene wash bottles used in recovering the train component samples. An unused particulate filter and an unused XAD-2 resin trap are also collected as trip blanks. (Note: The reagent blanks, filter, and trap samples are archived for analysis in the event that analyses of the blank train samples indicate a potential contamination problem.)

All of the sample containers are assigned numbers and labeled with the date and test-run number. The samples are turned over to the Sample Coordinator who records the appropriate data in the field logbook and packs the samples in insulated cold chests. Samples are stored in the sample holding area separate from the container supply area.

References:

"Method 23 - Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources"

"Method 1 - Sample and Velocity Traverses for Stationary Sources"

"Method 2 – Determination of stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)"

"Method 3 – Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight"

"Method 4 – Determination of Moisture Content in Stack Gases"

"Method 5 – Determination of Particulate Emissions from Stationary Sources"

Source for Methods 1,2 3, 4, 5 and 23: Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.

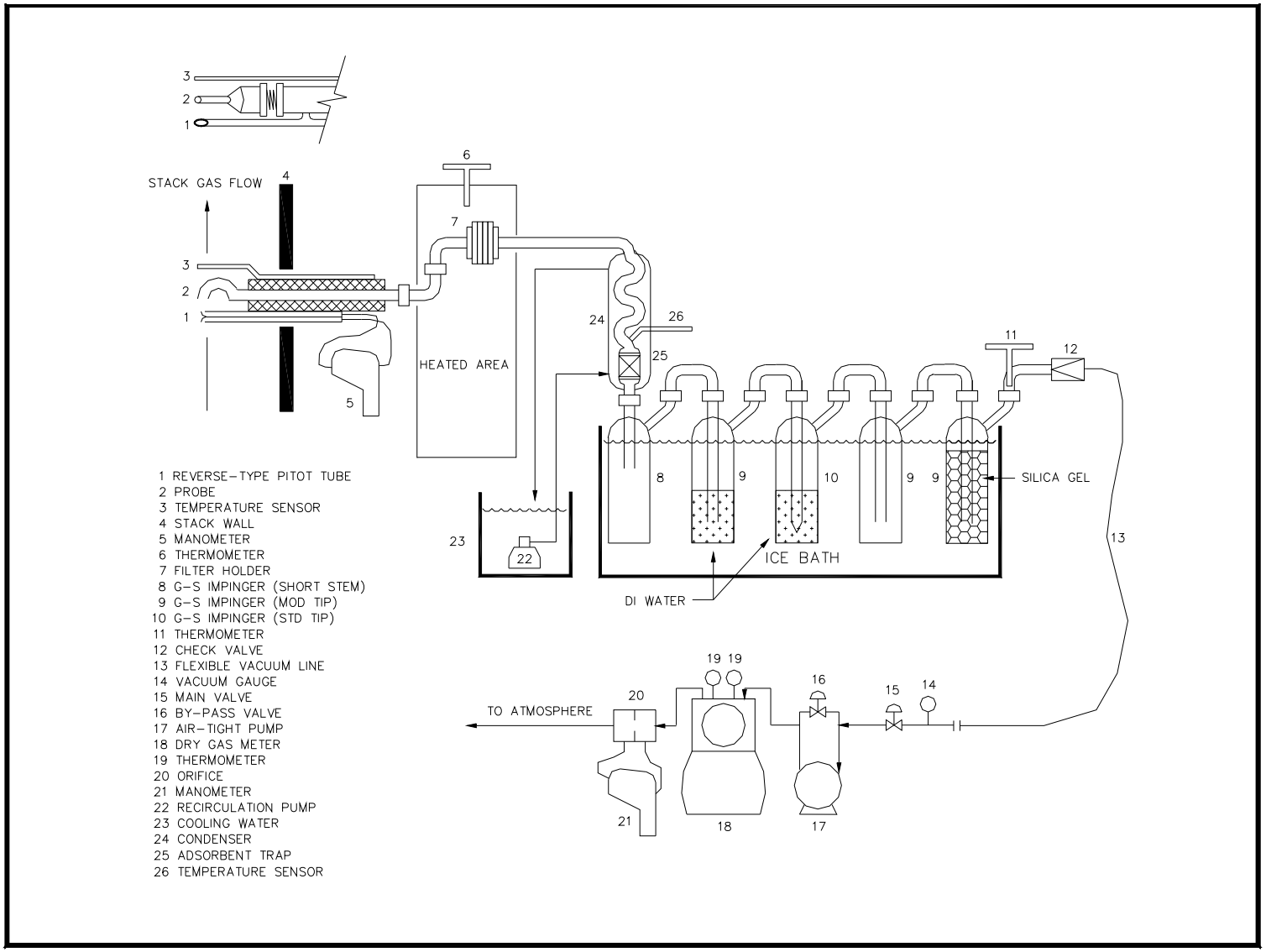

Figure 1. Method 23 Sample Train for PCDD/PCDF

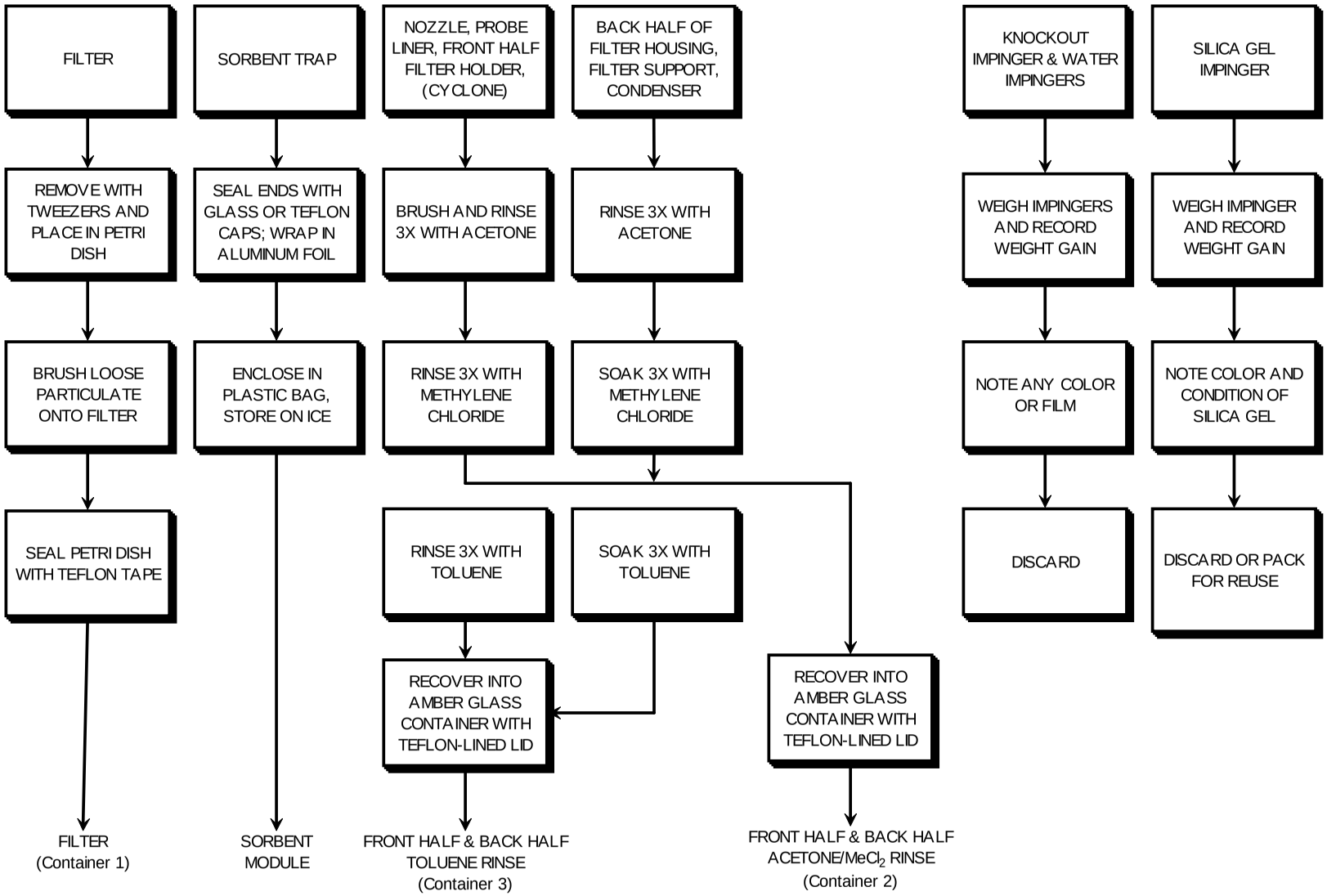

Figure 2. Method 23 PCDD/PCDF Sample Recovery

Matrices: Liquid waste

Procedure Summary: Liquid samples will be analyzed according to procedures in ASTM D 482. An aliquot of a well-mixed sample will be transferred to a prepared crucible, weighed, and ignited under controlled conditions.

The combustion residue will be ashed to constant weight in a muffle furnace at 775°C ± 25°C. Ash procedure will be terminated when consecutive weighings differ by not more than 0.5 mg. Ash content will be calculated according to method procedures.

References: ASTM D-482, American Society for Testing and Materials, Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series.

Matrices: Liquid waste

Procedure Summary: Liquid samples will be mixed by shaking, and a measured volume of liquid will be weighed to the nearest 0.1 g. A graduated cylinder calibrated with ASTM Type II water will be used.

References: ASTM D-1963, American Society for Testing and Materials, Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series.

Matrices:	Liquid waste
Procedure Summary:	Samples will be thoroughly mixed and a weighed aliquot will be combusted in an adiabatic bomb calorimeter per ASTM methods D 240. Quartz liners and lids will be used as appropriate in the bomb to minimize chloride attack. Special handling procedures will be used to minimize volatile losses from the samples.
References:	ASTM D-240, American Society for Testing and Materials, Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series.

Matrices: Organic liquid waste
Aqueous liquid waste

Procedure Summary: Liquid kinematic viscosity is determined by measuring the time required for a fixed volume of the liquid to flow under gravity through a calibrated glass capillary viscometer under a reproducible driving head and under closely controlled temperature conditions.

The kinematic viscosity is the product of the measured flow time and the calibration constant for the viscometer. The dynamic viscosity can be determined by multiplying the kinematic viscosity by the density of the liquid.

References: ASTM D 445, American Society for Testing and Materials, Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series.

Matrices: Liquid Process Samples

Procedures: Thoroughly mixed sample aliquots will be measured and transferred to a pre-weighed dish and evaporated to dryness on a steam bath of in a drying oven. The evaporated sample is then dried for at least one hour at 103 – 105°C, cooled in a desiccators and weighed. The cycle of drying, cooling, desiccating, and weighing is repeated until the loss of weight is less than 4% of the previous weight, or 0.5 mg, whichever is less per EPA method 160.3.

References: EPA 160.3, Methods for the Chemical Analysis of Water and Waste (MCAWW), EPA/600/4-79/020

Matrices: Liquid Waste Feeds

Procedures: Thoroughly mixed sample aliquots will be injected into an electrolytic cell containing the Karl Fischer solvent and will be titrated with an anhydrous solvent containing iodine per ASTM methods D-4928.

References: ASTM D-4928, American Society for Testing and Materials, Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series.

Matrices:	Organic liquid waste Aqueous liquid waste
Procedure Summary:	Organic samples are combusted in an oxygen bomb using the procedures given in SW-846 Method 5050 or 9076, and the chloride concentration is determined by microcoulometric titration using SW-846 Method 9076 or by ion chromatography using SW-846 Method 9056. Noncombustible samples are mixed with white oil and then combusted as organic samples.
References:	Methods 5050, 9056, and 9076, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986 and Updates.

Procedure Summary:

Calibration of Microwave Equipment Note: No calibration is required of units that use temperature feed back controls capable of replicating the performance specifications of the method. A three-point calibration method may be used with units that have utilized linear circuits. All other units must use the multiple point calibration method.

Place one kg of reagent water (23 $\pm$ 2°C measured to $\pm$ 0.05^{o}C) into a flourocarbon (PFA or TFM) beaker. Cover the beaker and place in the microwave. Microwave for 2 minutes at the desired partial setting with the units exhaust fan on maximum. Remove the beaker and stir vigorously with a magnetic stirring bar. Record the maximum temperature within the first 30 seconds to $\pm$ 0.05^{o}C.

The absorbed power is determined by the following relationship:

Power absorbed in Watts = (change in temperature) (34.86)

Multi-Point Calibration involves the measurement of absorbed power over a large range of power settings. Typically 100, 99, 98, 97, 95, 90, 70, 60, 50, and 40% for a 600 W unit. The final calibration point should be the partial power setting that will be used in the test. This setting should be checked periodically to evaluate the calibration. If a significant change is detected ($\pm$ 10W), the entire calibration should be reevaluated.

Three-Point Calibration involves the measurement of absorbed power at three different power settings. Measure the power at 100% and 50% and calculate the power setting corresponding to the required power in watts specified from those two points. Measure the absorbed power at the calculated partial setting. If the measured absorbed power does not correspond to the specified power within $\pm$ 10W, use the multiple point calibration. This point should be checked periodically to verify the integrity of the calibration.

Sample Digestion Weigh the fluorocarbon (PFA or TFM) digestion vessel to 0.01 g. Add 45 ml of sample and 5 ml of HNO_3 to the vessel. Weigh each vessel again. Microwave the vessels to 160°C $\pm$ 4°C in 10 minutes and 165 – 170°C in the second 10 minutes based on the manufacture's instructions and the calibration of the individual microwave. Allow the vessels to cool and weigh again. If the weight has decreased by more than 10%, discard the sample. Transfer samples to acid-cleaned bottles and filter, centrifuge, or allow to settle as necessary.

References:

Metals Preparation Method 3015 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

Methods 6010, 6020, and 7000 series, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

EPA Method 29 Code of Federal Regulations, Title 40, Part 60, Appendix A

Procedure Summary:

Calibration of Microwave Equipment Note: No calibration is required of units that use temperature feed back controls capable of replicating the performance specifications of the method. A three-point calibration method may be used with units that have utilized linear circuits. All other units must use the multiple point calibration method.

Place one kg of reagent water (23 $\pm$ 2°C measured to $\pm$ 0.05^{o}C) into a flourocarbon (PFA or TFM) beaker. Cover the beaker and place in the microwave. Microwave for 2 minutes at the desired partial setting with the units exhaust fan on maximum. Remove the beaker and stir vigorously with a magnetic stirring bar. Record the maximum temperature within the first 30 seconds to $\pm$ 0.05^{o}C.

The absorbed power is determined by the following relationship:

Power absorbed in Watts = (change in temperature) (34.86)

Multi-Point Calibration involves the measurement of absorbed power over a large range of power settings. Typically 100, 99, 98, 97, 95, 90, 70, 60, 50, and 40% for a 600 W unit. The final calibration point should be the partial power setting that will be used in the test. This setting should be checked periodically to evaluate the calibration. If a significant change is detected ($\pm$ 10W), the entire calibration should be reevaluated.

Three-Point Calibration involves the measurement of absorbed power at three different power settings. Measure the power at 100% and 50% and calculate the power setting corresponding to the required power in watts specified from those two points. Measure the absorbed power at the calculated partial setting. If the measured absorbed power does not correspond to the specified power within $\pm$ 10W, use the multiple point calibration. This point should be checked periodically to verify the integrity of the calibration.

Sample Digestion Weigh the fluorocarbon (PFA or TFM) digestion vessel to 0.001 g. Weigh a well-mixed sample to the nearest 0.001 g into the fluorocarbon sample vessel. Add 10 $\pm$ 0.1 ml HNO_3 and reweigh the vessel. Microwave the samples for 10 minutes. The temperature of the sample should rise to 175° in less than 5.5 minutes and remain between 170 – 180°C for the balance of the 10 minutes. Allow vessels to cool for at least 5 minutes and reweigh. If the weight of the acid plus the sample has decreased by more than 10%, discard the sample. Transfer samples to acid-cleaned bottles and filter, centrifuge, or allow to settle as necessary.

References:

Metals Preparation Methods 3051 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

Methods 6010, 6020, and 7000 series, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

EPA Method 29 Code of Federal Regulations, Title 40, Part 60, Appendix A

Matrices:

Procedure Summary:

SW-846 Method 6010 Before using this procedure there must be data available documenting initial demonstration of performance including the selection criteria of background correction points, analytical dynamic ranges, applicable equations, and written verification of interelement correction equations or other routines for correcting spectral interference. The analyst should follow the instructions provided by the manufacturer for operating conditions. Specific wavelengths to be used are listed in Table 1 of SW-846 Method 6010. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Prior to sample analysis, the instrument must be setup with proper operating parameters as detailed in the Method and allowed to become thermally stable.

To begin analysis of samples, reset the nebulizer gas flow rate to the determined optimized flow. The instrument should then be profiled and an initial calibration should be performed in accordance with the manufacture's instructions. The calibration should consist of a minimum of a blank and a standard. An analysis of an initial calibration verification sample (ICV), a calibration blank (ICB), and a continuing calibration verification sample (CCV) should immediately follow the daily calibration. An interference check sample should also be run at the beginning of each analytical run. The results of this check sample should be within $\pm$ 20% of the true value. A calibration blank and either an ICV or a CCV must be analyzed after every tenth sample and at the end of the run. The system should be rinsed between each sample. Check standards and calibration verifications must be within 10% of the calibration with relative standard deviation < 5% from replicate (minimum of two) integrations. If the calibration cannot be verified within limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated.

Quality Control:

One laboratory method blank will be analyzed for every batch of samples analyzed. The method blank is a performance control sample that is prepared in the laboratory and processed in a manner identical to the field sample.

To document the effect of the matrix, a minimum of at least one matrix spike and one duplicate or one matrix spike/matrix spike duplicate pair should be analyzed.

A laboratory control sample (LCS or method spike) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight and volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the matrix spike results indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

If the analyte concentration is sufficiently high in a group of samples (minimally, a factor of 10 above the instrument detection limit after dilution), an analysis of a 1:5 dilution should be performed on a typical sample. The diluted and undiluted concentrations should agree within 10% to indicate the absence of interferants.

References:

"Methodology for the Determination of Metal Emissions in Exhaust Gases from Hazardous Waste Incineration and Similar Combustion Processes." EPA Methods Manual for Compliance with the BIF Regulations, 40 CFR 266 Appendix IX.

Metals analytical Methods 6010 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

Matrices:

Procedure Summary:

SW-846 Methods 7470 Prior to sample analysis, a calibration curve should be created by plotting the absorbances of standards versus micrograms of mercury. A minimum of a calibration blank and three standards should be used for this calibration. After the calibration, the curve must be verified by the use of at least a calibration blank and a calibration check standard. The check standard must be within 10% of the true value for the curve to be considered valid. If more than ten samples are analyzed, the curve must be verified by measuring a mid-range standard after every tenth sample. This calibration check value must be within 20% of the true value or the previous ten samples must be reanalyzed.

To determine the absorbance of samples or standards, the circulating pump should be adjusted to 1 liter/minute and allowed to run continuously during analysis. Remove the aeration apparatus from the BOD bottle and Allow the sample or standard to stand quietly without manual agitation. Attach the stopper and frit to the BOD bottle. The absorbance should reach a maximum within 30 seconds. As soon as the recorder pen levels off (approximately 1 minute), open the bypass valve and continue aeration until the absorbance returns to its minimum value. Close the bypass valve, remove the stopper and frit from the BOD bottle, and continue aeration.

Quality Control:

One laboratory method blank will be analyzed for every batch of samples analyzed. The method blank is a performance control sample that is prepared in the laboratory and processed in a manner identical to the field sample.

To document the effect of the matrix, a minimum of at least one matrix spike and one duplicate or one matrix spike/matrix spike duplicate pair should be analyzed.

A laboratory control sample (LCS or method spike) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight and volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the matrix spike results indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

References:

"Methodology for the Determination of Metal Emissions in Exhaust Gases from Hazardous Waste Incineration and Similar Combustion Processes." EPA Methods Manual for Compliance with the BIF Regulations, 40 CFR 266 Appendix IX.

Metals analytical Method 7470 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

Matrices:	Particulate filter Front half acetone rinses
Procedure Summary:	The Method 5 particulate filter will be oven dried at 105°C (220°F) for 2 to 3 hours and the weight will be determined to the nearest 0.1 mg. The Method 5 front half rinse will be evaporated to dryness at ambient temperature and pressure, dessicated for 24 hours, and the residue weight will be determined to the nearest 0.1 mg.
References:	EPA Method 5, Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.

Matrices: Acid Impinger Liquid - sulfuric acid solution for HCl analysis (Container 3)
Alkaline Impinger Liquid - sodium hydroxide solution for Cl_2 analysis (Container 4)

Procedure Summary: The M26A Impinger samples will be analyzed for hydrogen chloride and chlorine using an ion chromatograph. All chloride ions found in the acid impinger solution are assumed to occur due to the absorption of HCl. All chloride ions found in the alkaline impinger solution are assumed to result from the absorption of Cl_2.

References: Method 9057, Protocol for the Analysis of Samples from HCl/Cl_2 Emission Sampling Trains, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates.

Matrices:

Particulate filter (Container 1)
Front-half acetone rinse (Container 2)
Front-half nitric acid rinse (Container 3)
Acidified peroxide impinger solutions plus nitric acid rinses (Container 4)
Acetone reagent blank (Container 7)
0.1N nitric acid reagent blank (Container 8A)
Deionized water blank (Container 8B)
Acidified H_2O_2 impinger solution blank (Container 9)
Particulate filter blanks (Container 12)

Procedure Summary:

Particulate filter (Container 1) – The filter is dried to constant weight and weighed to the nearest 0.1 mg following the procedures specified in Method 5. (Note: This step may be omitted when the determination of particulate matter is not required by the test plan.)

The particulate filter sample is divided into portions containing approximately 0.5 g each and each portion is placed into either an individual fluorocarbon based microwave pressure relief vessel, or a Parr® Bomb. Six mL of HNO_3 and four mL of HF are added to either vessel. For the microwave vessel, each filter sample is digested according to the procedures specified in Method 3051. For the Parr® Bomb, each filter sample is digested at 140°C for six hours following the manufacturer's instructions. The filter digestate is recovered from the vessel(s) and added to the front-half nitric acid rinse digestate as described below for Container 3.

Front-half acetone rinse (Container 2) - The acetone rinse is measured to the nearest 1 mL (or to the nearest 0.5 g.) and evaporated to dryness in a 250 mL beaker. The rinse residue is dried to constant weight and the results are recorded to the nearest 0.1 mg. The rinse residue is then dissolved with 10 mL HNO_3 and combined with the front-half nitric acid rinse in Container 3 before it is prepared for digesting. (Note: The weighing step may be omitted when the determination of particulate matter is not required by the test plan.)

Front-half nitric acid rinse (Container 3) – The rinse sample pH is checked and adjusted to <2 with HNO_3 if necessary. The sample is transferred with water rinse into a beaker with a ribbed watch glass. The sample volume is reduced to approximately 20 mL by heating on a hot plate at a temperature just below boiling. The sample is then digested following the procedures in Method 3010A, Method 3015, or the Parr Bomb operating manual, as appropriate. This digestate is added to the particulate filter digestate and mixed thoroughly. The combined digestate is filtered through a Whatman 541 filter paper and diluted with water to 300 mL (or other appropriate volume depending on the expected concentration). This diluted sample is labeled Analytical Fraction 1 for multiple metals analysis.

Acidified Peroxide Impinger Solutions (Container 4) - The sample volume is measured to the nearest 0.5 mL and labeled Sample Fraction 2. The sample pH is measured and adjusted, if needed, to 2 or less. The sample is transferred with water into a beaker with a ribbed watch glass. The sample volume is reduced to approximately 20 mL by heating on a hot plate at a temperature just below boiling. The sample is then acid digested following procedures in Methods 3010 or 3015, Parr Bomb operating manual, or Method 29. The digestate is labeled Analytical Fraction 2.

The preparation of the test samples is summarized in Figure 1. The container numbers are the same as those used in the reference methods for the determination of multiple metals. Container numbers 5 and 6 are not included because they are associated with the determination of mercury emissions, which in not included in the test plan.

Quality Control:

Particulate filter blank (Container 12) - A blank filter is processed in the same manner as the Container 1 sample. As an option, two additional particulate filter blanks may be processed in

the same manner as provided for in Section 8.1 of Method 0060. The average of the three particulate weights is then used as the front-half blank correction value.
Acetone reagent blank (Container 7) - A 100 mL portion is processed in the same manner as the Container 2 sample. (The acetone sample residue is combined with the nitric acid reagent blank.)

Nitric acid reagent blank (Container 8A) – A 100 mL portion is processed in the same manner as the Container 3 sample. (The acetone residue is added to the nitric acid blank and the mixture is digested.)

The filter digestate and the acetone-nitric acid digestate are combined and filtered to form Analytical Fraction Blank 1 for total metals analyses. The analytical results are used as the front-half blank correction values for the target metals.

Deionized water blank (Container 8B) – Portions of this sample are used in the preparing other blank samples for analysis.

Acidified peroxide impinger blank (Container 9) – A 200 mL portion is combined with a 100 mL portion of nitric acid blank (Container 8A) to form Sample Fraction Blank 2. This sample is digested to produce Analytical Blank Fraction 2 for target metals analyses. The analytical results are used as back-half blank correction values for the target metals.

The preparation of the reagent blank samples is summarized in Figure 2 of this table. The container numbers are the same as those used in the reference methods. Container numbers 10 and 11 are not included because they are associated with the determination of mercury emissions, which is not included in the test plan.

References:

‘Method 5 – Determination of Particulate Emissions From Stationary Sources”, Appendix A – Test Methods, New source Performance Standards. 40 CFR 60.

“Method 29 – Determination of Metals Emissions from Stationary Sources”, Appendix A-Test Methods, New Source Performance Standards, 40 CFR 60.

“Method 0060 – Determination of Metals in Stack Emissions”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

“Method 3010A – Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

“Method 3015 – Microwave Assisted Acid Digestion of Aqueous Samples and Extracts”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

“Method 3020A – Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

“Method 3050B – Acid Digestion of Sediments, Sludges, Soils, and Oils”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

“Method 3051Microwave Assisted Acid digestion of Sediments, Sludges, Soils, and Oils”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

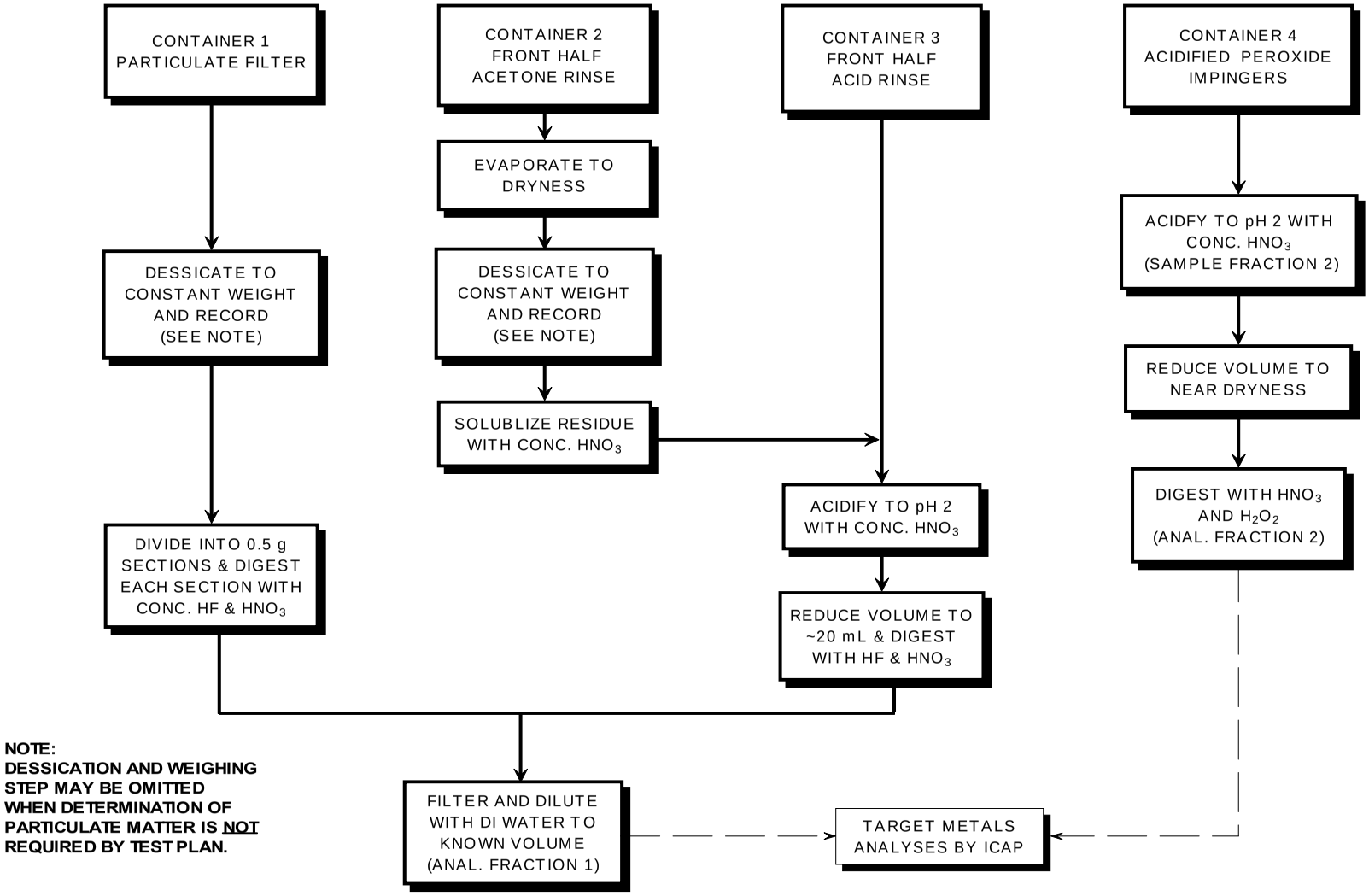

Figure 1. Preparation of Methods 29 and 0060 Stack Gas Samples for Multiple Metals Analyses (Excluding Hg)

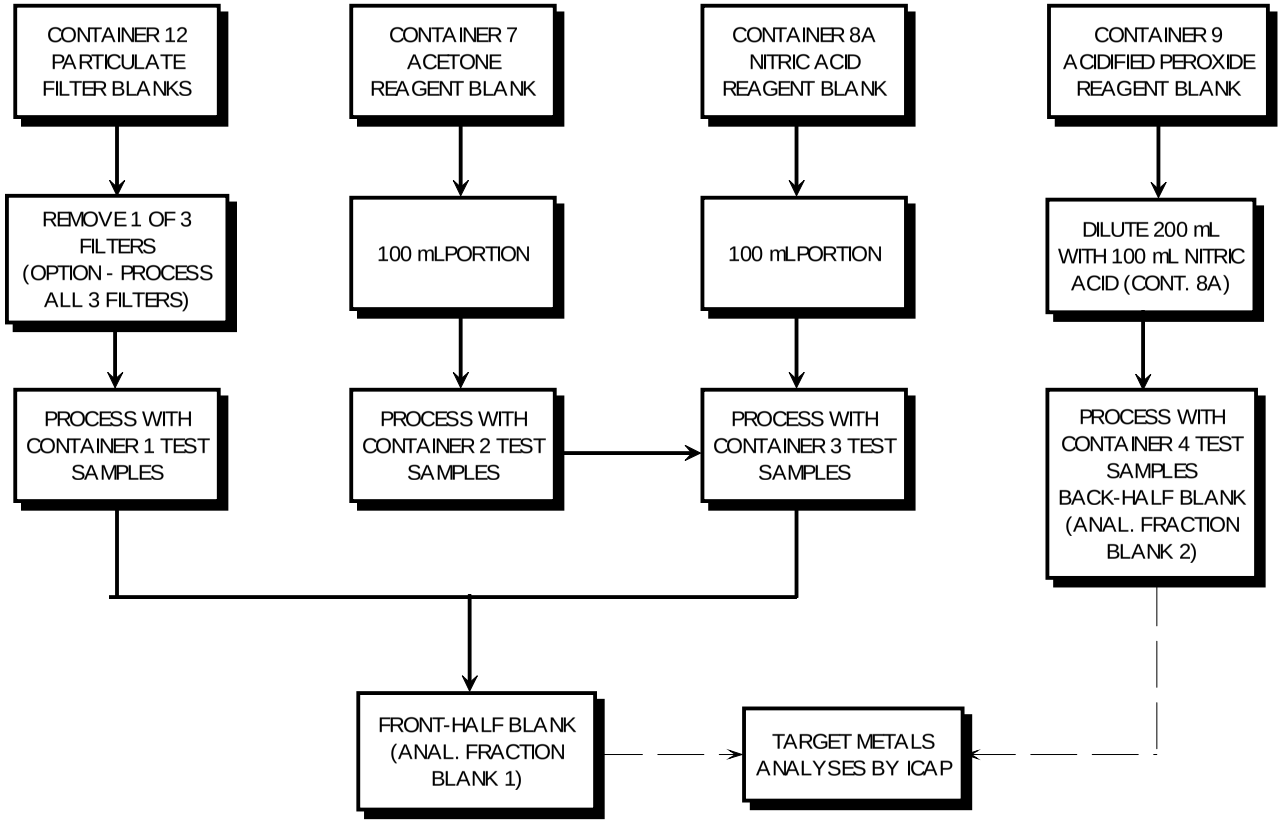

Figure 2. Preparation of Quality Control Samples for Multiple Metals Analyses (Excluding Mercury)

Matrices:
Fraction 1A
Fraction 2A
Fraction Blank 1A
Fraction Blank 2A

Procedure Summary:

Fraction 1A, Fraction 2A, Fraction Blank 1A, and Fraction Blank 2A are analyzed using SW-846 Method 6010 – Inductively Coupled Plasma-Atomic Emission Spectrometry.

SW-846 Method 6010 Before using this procedure there must be data available documenting initial demonstration of performance including the selection criteria of background correction points, analytical dynamic ranges, applicable equations, and written verification of interelement correction equations or other routines for correcting spectral interference. The analyst should follow the instructions provided by the manufacturer for operating conditions. Specific wavelengths to be used are listed in Table 1 of SW-846 Method 6010. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Prior to sample analysis, the instrument must be setup with proper operating parameters as detailed in the Method and allowed to become thermally stable.

To begin analysis of samples, reset the nebulizer gas flow rate to the determined optimized flow. The instrument should then be profiled and an initial calibration should be performed in accordance with the manufacture's instructions. The calibration should consist of a minimum of a blank and a standard. An analysis of an initial calibration verification sample (ICV), a calibration blank (ICB), and a continuing calibration verification sample (CCV) should immediately follow the daily calibration. An interference check sample should also be run at the beginning of each analytical run. The results of this check sample should be within $\pm$ 20% of the true value. A calibration blank and either an ICV or a CCV must be analyzed after every tenth sample and at the end of the run. The system should be rinsed between each sample. Check standards and calibration verifications must be within 10% of the calibration with relative standard deviation < 5% from replicate (minimum of two) integrations. If the calibration cannot be verified within limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated.

Quality Control:

SW-846 Method 6010

One laboratory method blank will be analyzed for every batch of samples analyzed. The method blank is a performance control sample that is prepared in the laboratory and processed in a manner identical to the field sample.

To document the effect of the matrix, a minimum of at least one matrix spike and one duplicate or one matrix spike/matrix spike duplicate pair should be analyzed.

A laboratory control sample (LCS or method spike) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight and volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the matrix spike results indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

If the analyte concentration is sufficiently high in a group of samples (minimally, a factor of 10 above the instrument detection limit after dilution), an analysis of a 1:5 dilution should be performed on a typical sample. The diluted and undiluted concentrations should agree within 10% to indicate the absence of interferants.

References: "Methodology for the Determination of Metal Emissions in Exhaust Gases from Hazardous Waste Incineration and Similar Combustion Processes." EPA Methods Manual for Compliance with the BIF Regulations, 40 CFR 266 Appendix IX.

Metals analytical Methods 6010 and 7470, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

Matrices:

Particulate Filter (Container 1)
Combined front and back half acetone and methylene chloride rinse (Container 2)
Combined front and back half toluene rinse (Container 3)
Adsorbent resin module -XAD-2 resin and glass wool
Acetone, methylene chloride, and toluene reagent blanks
Adsorbent resin module trip blank
Particulate filter trip blank

Procedure Summary:

The particulate filter (Container 1) is transferred directly to the thimble of a Soxhlet extraction apparatus. The XAD-2 resin is transferred from the adsorbent module to the thimble using toluene to flush the resin and to rinse the glass module and the rinse catch beaker. The glass wool plug from the adsorbent module is added to the thimble. The sample preparation and analytical steps are summarized in Figure 1.

The front and back half acetone and methylene chloride rinse (Container 2) is transferred to a Kuderna-Danish concentrator apparatus and concentrated to 1-2 mL followed by nitrogen blow down at a temperature of less than 37°C. Container 2 is rinsed three times with small portions of methylene chloride and these rinses are added to the K-D concentrate, which is then further concentrated to near dryness. The K-D concentrate is then added to the Soxhlet thimble (which already contains the filter, XAD resin, and glass wool plug from the resin module). Forty μL of PCDD/PCDF internal standards are added to the thimble and the components are extracted in toluene for 16 hours. The Soxhlet extract is allowed to cool, transferred with three 10 mL rinses to a rotary evaporator, and concentrated to approximately 10 mL. Approximately 5 mL of the extract is archived for future use if needed. The remaining 5 mL is evaporated to near dryness in a N_2 evaporator and the residue is then dissolved in 5 mL of hexane. This sample is then processed through chromatographic cleanup steps of Method 8290 to prepare the sample for analysis.

The front and back half toluene rinse sample (Container 3) is transferred to a rotary evaporator where 40 μL of PCDD/PCDF internal standards are added, and the sample is concentrated to 1 to 5 mL. Container 3 is rinsed three times with toluene and the rinse is added to the residue in the rotary evaporator. The rinse/residue mixture is further concentrated to near dryness (approx. 10 mL??). Approximately one-half of the extract is archived for future use if needed. The remaining one-half is processed through chromatographic cleanup steps of Method 8290 to prepare the sample for analysis.

Each prepared sample is spiked with 20 μL of the recovery standard solution and immediately analyzed by HRGC/HRMS according Method 8290 procedures. Results are reported for the total mass of each tetra- through octa-chlorinated PCDD and PCDF congener group, and for each individual 2,3,7,8- chlorine substituted isomer.

Quality Control:

One laboratory method blank will be analyzed for every batch of samples analyzed. The method blank is a performance control sample that is prepared in the laboratory and processed in a manner identical to the field sample. The XAD-2 resin used for the method blank will be obtained from the same batch used for preparation of the field modules.

References: “Method 23 - Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Municipal Waste Combustors”, Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.

“Method 8290 - Polychlorinated Dibenzodioxin (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromotography/High-Resolution Mass Spectrometry (HRGC/HRMS)”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.

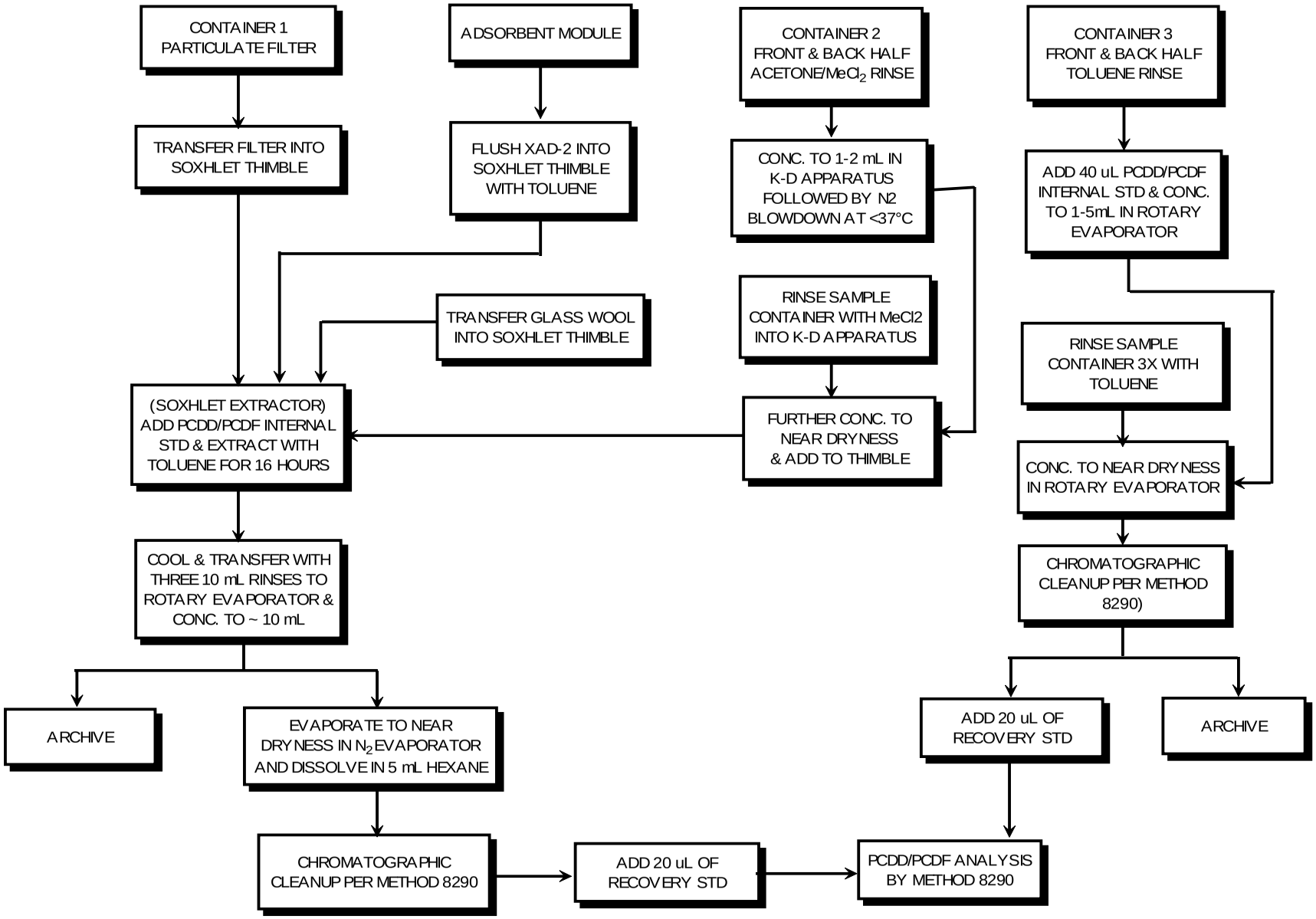

Figure 1. Flow Diagram for Preparation of M23 Stack Gas Samples for PCDD/PCDF Analysis