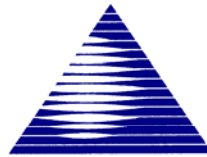

CARBON REACTIVATION FURNACE PERFORMANCE DEMONSTRATION TEST PLAN

PREPARED FOR:

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ACRONYMS AND ABBREVIATIONS

acfm	Actual cubic feet per minute
APC	Air pollution control
ASTM	American Society for Testing and Materials
AWFCO	Automatic waste feed cutoff
Btu	British thermal unit
CAA	Clean Air Act
CARB	California Air Resources Board
CEM or CEMS	Continuous emission monitor or Continuous emission monitoring system
CFR	Code of Federal Regulations
cm	Centimeters
COPCs	Compounds of potential concern
cu. ft.	Cubic foot
CVAAS	Cold vapor atomic absorption spectroscopy
DC	Direct current
DRE	Destruction and removal efficiency
dscf	Dry standard cubic foot
dscfm	Dry standard cubic feet per minute
EPA	United States Environmental Protection Agency
ft	Feet
GC/FID	Gas chromatography/flame ionization detector
GC/MS	Gas chromatography/mass spectrometry
gpm	U.S. Gallons per minute
gr	Grain (equals 1/7000 pound)
HAP	Hazardous air pollutant
HPLC	High performance liquid chromatography
hr	Hour
HRGC/HRMS	High resolution gas chromatography/high resolution mass spectrometry
HWC MACT	Hazardous Waste Combustor Maximum Achievable Control Technology regulations
ICP	Inductively coupled plasma spectroscopy
in	Inch
in w.c.	Inches of water column (pressure measurement)
L	Liter
lb	Pound
mg	Milligram
ml	Milliliter
MTEC	Maximum theoretical emission concentration
NDIR	Non-dispersive infrared
ng	Nanogram
NVOC	Nonvolatile organic compound
P&ID	Piping and instrumentation diagram
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PCDD/PCDF	Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzo furans
PDT	Performance Demonstration Test
PDTP	Performance Demonstration Test Plan

PFD	Process flow diagram
PIC	Product of incomplete combustion
PLC	Programmable logic controller
POHC	Principal organic hazardous constituent
ppmv	Parts per million by volume
ppmvd	Parts per million by volume, dry basis
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RF	Reactivation Furnace
scfm	Standard cubic feet per minute
sq. ft.	Square feet
SVOC	Semivolatile organic compound
TCDD	Tetrachloro dibenzo-p-dioxin
TEQ	Toxicity equivalent (related to 2,3,7,8-TCDD)
TIC	Tentatively identified compound
TOE	Total organic emissions
TSCA	Toxic Substances Control Act
ug	Microgram
VOA	Volatile organic analysis
VOC	Volatile organic compound
VOST	Volatile organic sampling train
WESP	Wet electrostatic precipitator
WCAI	Westates Carbon – Arizona, Inc.

1.0 INTRODUCTION

1.1 FACILITY AND BACKGROUND INFORMATION

Westates Carbon - Arizona, Inc. (WCAI) operates a carbon reactivation facility located in the Colorado River Indian Tribes (CRIT) Industrial Park near Parker, Arizona. The facility treats spent activated carbon that has been used by industry, state and federal government agencies, and municipalities for the removal of organic compounds from liquid and vapor phase process waste streams. Once the carbon has been used and is spent, it must be either disposed of or reactivated at a facility such as WCAI. A Carbon Reactivation Furnace (RF) is used by WCAI to reactivate the spent carbon. Some of the carbon received at the Parker facility is designated as a hazardous waste under the Resource Conservation and Recovery Act (RCRA) regulations. Much of the carbon received at the facility is not a RCRA hazardous waste, as it is either not a characteristic or listed waste. The RF is not a hazardous waste incinerator. "Hazardous waste incinerator" is defined in 40 CFR Part 63, Subpart EEE as a "device defined as an incinerator in § 260.10 of this chapter and that burns hazardous waste at any time." (40 CFR 63.1201). "Incinerator" is defined in 40 CFR 260.10 as "any enclosed device that: (1) Uses controlled flame combustion and neither meets the criteria for classification as a boiler, sludge dryer or carbon regeneration unit, nor is listed as an industrial furnace; or (2) Meets the definition of infrared incinerator or plasma arc incinerator. (emphasis supplied)" The RF does not qualify as an incinerator and instead is designated by Subpart X of the RCRA regulations as a Miscellaneous Unit. According to 40 CFR 264.601 of the Subpart X regulations, permit terms and provisions for a Miscellaneous Unit must include appropriate requirements of 40 CFR Subparts I through O and Subparts AA through CC, 40 CFR 270, 40 CFR 63 Subpart EEE, and 40 CFR 146.

Based on 40 CFR 264.601, WCAI will test the RF to demonstrate performance in accordance with the emission standards of 40 CFR 63 Subpart EEE. These emission standards are more stringent than the RCRA hazardous waste incinerator emission standards of 40 CFR 264 Subpart O. The regulations at 40 CFR 63 Subpart EEE are often referred to as the Hazardous Waste Combustor Maximum Achievable Control Technology (HWC MACT) standards. This terminology will be used in this document.

1.2 TEST PLAN PURPOSE AND ORGANIZATION

The purpose of this Performance Demonstration Test Plan (PDTP) is to identify and document the necessary process details; sampling, analytical, and QA/QC procedures; and anticipated permit operating conditions necessary for demonstration of compliance with the applicable regulatory requirements, and for demonstration of continuing compliance with those standards.

To best address the requirements for demonstrating that the RF can operate within the parameters and limits established by the applicable regulatory requirements the PDTP has been organized into the following major sections:

- 1.0 INTRODUCTION
- 2.0 FEED STREAM DESCRIPTION
- 3.0 ENGINEERING DESCRIPTION
- 4.0 TEST DESIGN AND PROTOCOL
- 5.0 SAMPLING, ANALYSIS, AND MONITORING PROCEDURES
- 6.0 TEST SCHEDULE
- 7.0 OPERATING PERMIT OBJECTIVES
- 8.0 TEST REPORT
- 9.0 DATA IN LIEU OF TESTING

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1.3 PROCESS OVERVIEW

The carbon reactivation process consists of a multiple hearth reactivation furnace, a natural gas fired afterburner used to destroy organic contaminants released from the carbon, a wet quench, venturi scrubber, packed bed scrubber, and wet electrostatic precipitator.

1.4 TEST OBJECTIVES AND APPROACH

The WCAI Performance Demonstration Test Plan has been prepared to provide comprehensive performance testing of the RF unit to demonstrate compliance with the applicable HWC MACT emission standards and to gather data for use in a site-specific risk assessment. The objectives of the PDTP are to demonstrate regulatory compliance with standards such as Destruction and Removal Efficiency (DRE) and particulate matter emission concentration, while operating at “worst case” conditions processing normal feed materials, which have been augmented with metals, chloride, etc., to establish operating conditions that will be included in the permit. More specifically, the objectives of the Performance Demonstration Test (PDT) are as follows:

1. Demonstrate Compliance with Applicable USEPA Regulatory Performance Standards (Based on HWC MACT Standards for Existing Hazardous Waste Incinerators):
 - Demonstrate a DRE of greater than or equal to 99.99% for the selected principal organic hazardous constituents (POHCs) chlorobenzene and tetrachloroethene.
 - Demonstrate stack gas carbon monoxide concentration less than or equal to 100 ppmv, dry basis, corrected to 7% oxygen.
 - Demonstrate stack gas hydrocarbon concentration of less than or equal to 10 ppmv, as propane, dry basis, corrected to 7% oxygen.
 - Demonstrate a stack gas particulate concentration less than or equal to 34 mg/dscm (0.015gr/dscf) corrected to 7% oxygen.
 - Demonstrate that the stack gas concentration of hydrogen chloride (HCl) and chlorine (Cl₂) are no greater than 77 ppmv, dry basis, corrected to 7% oxygen, expressed as HCl equivalents.
 - Demonstrate that the stack gas mercury concentration is less than or equal to 130 µg/dscm, corrected to 7% oxygen.
 - Demonstrate that the stack gas concentration of semivolatile metals (cadmium and lead, combined) is less than or equal to 240 µg/dscm, corrected to 7% oxygen.

- Demonstrate that the stack gas concentration of low volatility metals (arsenic, beryllium, and chromium, combined) is less than or equal to 97 $\mu\text{g/dscm}$, corrected to 7% oxygen.
 - Demonstrate that the stack gas concentration of dioxins and furans does not exceed 0.40 ng/dscm, corrected to 7% oxygen, expressed as toxic equivalents of 2,3,7,8-TCDD (TEQ). This is the applicable standard since the gas temperature entering the first particulate matter control device is less than 400°F.
2. Establish Permit Operating Limits
- Demonstrate maximum feed rate for spent activated carbon.
 - Demonstrate minimum afterburner gas temperature
 - Demonstrate maximum combustion gas velocity (or a suitable surrogate indicator)
 - Demonstrate maximum total chlorine/chloride feed rate
 - Establish a Maximum Theoretical Emission Concentration (MTEC) limit for mercury
 - Demonstrate system removal efficiency (SRE) for semivolatile and low volatility metals so feed rate limits can be developed by extrapolation from test results.
 - Establish appropriate operating limits for the air pollution control system components.
3. Gather Information for Use in a Site-Specific Risk Assessment
- Measure emissions of metals, including hexavalent chromium
 - Measure emissions of specific volatile and semivolatile products of incomplete combustion (PICs)
 - Measure emissions of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDF)
 - Measure emissions of polychlorinated biphenyls (PCBs)
 - Measure emissions of specific organochlorine pesticides
 - Measure emissions of total volatile, semivolatile, and nonvolatile organics
 - Determine the stack gas particle size distribution.

1.5 TEST PROTOCOL SUMMARY

In order to accomplish the PDT objectives, (i.e., demonstrating that the unit will meet all applicable environmental performance standards) a single test condition representing “worst case” operations of minimum temperature, maximum combustion gas velocity (minimum residence time), and maximum waste feed rate will be performed. The test will consist of at least three replicate sampling runs.

A summary description of the testing conditions, analytical parameters, and sampling methods follows:

1.5.1 Test Condition 1 (“Worst-Case” Operations)

Sampling and monitoring protocols that will be utilized while carrying out the performance test are summarized as follows:

- Spent Activated Carbon Feed - total chlorine/chloride, elemental (C, H, N, O, S, moisture), volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Makeup Water - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Caustic feed to APC - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Cu, Co, Pb, Hg, Ag, Tl, Se, Ni, V, Zn)
- Scrubber Blowdown - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Wastewater Discharge to POTW - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Stack gas particulate, HCl, and Cl₂ using EPA Method 26A
- Stack gas target volatile organics using VOST, SW-846 Method 0030
- Stack gas target semivolatile organics and organochlorine pesticides using SW-846 Method 0010
- Stack gas PAHs and PCBs using a separate SW-846 Method 0010 sampling train
- Stack gas PCDD/PCDF using SW-846 Method 0023A
- Stack gas total volatile organics using SW-846 Method 0040
- Stack gas total semivolatile and nonvolatile organics using SW-846 Method 0010
- Stack gas metals (Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn) using EPA Method 29
- Stack gas hexavalent chromium using SW-846 Method 0061
- Stack gas particle size distribution using a cascade impactor

- Stack gas CO and O₂ by permanently installed CEM according to the protocols in the Appendix to 40 CFR 63, Subpart EEE; Performance Specification 4B of 40 CFR 60, Appendix B.
- Stack gas total hydrocarbons (as propane) by temporary CEM according to EPA Method 25A and the protocols in the Appendix to 40 CFR 63, Subpart EEE.

1.6 DEVELOPMENT OF PERMIT LIMITS

WCAI is required to establish operating limits (applicable whenever RCRA hazardous spent activated carbon is in the reactivation furnace) in its permit to ensure that the RF system complies with the applicable USEPA environmental performance standards at all times that RCRA hazardous spent activated carbon is being treated. Under the HWC MACT, the regulations establish a comprehensive list of regulated parameters at 40 CFR 63.1209 (j) through (p) which are used to ensure continuing regulatory compliance. Other RCRA permitting guidance documents also suggest certain permit limits and means for establishing those limits. Finally, the RF has been operating since 1996 with certain limits imposed by plant operations personnel.

Considering the configuration of the RF system and the characteristics of the spent activated carbon to be fed, WCAI anticipates establishing process operational limits on the following parameters:

- Minimum afterburner gas temperature
- Maximum spent activated carbon feed rate
- Maximum total chlorine and chloride feed rate
- Maximum feed rate of mercury (based on MTEC)
- Maximum feed rate of semivolatile metals (total combined lead and cadmium)
- Maximum feed rate of low volatility metals (total combined arsenic, beryllium, and chromium)
- Minimum venturi scrubber pressure differential
- Minimum quench/venturi scrubber total liquid flow rate
- Minimum packed bed scrubber pH
- Minimum packed bed scrubber pressure differential
- Minimum packed bed scrubber liquid flow rate
- Minimum scrubber blowdown flow rate
- Minimum WESP secondary voltage

- Maximum stack gas flow rate (indicator of combustion gas velocity).

These operating limits will be established as described in Section 7.0 of this document. The approach taken for establishing operating limits generally follows the specifications of 40 CFR 63 Subpart EEE and guidance prepared for RCRA incinerator permits. However, since those regulations and guidance do not strictly apply to this unit, but are used as guidelines for the development of appropriate limits, WCAI is also proposing to establish some limits based on current operating practices and automation system capabilities, which have proven to be effective for this particular facility.

1.7 TEST PLAN DEVELOPMENT AND CONDUCT OF THE PERFORMANCE DEMONSTRATION TEST

In August 2001, USEPA requested that WCAI prepare a performance demonstration test plan and a risk assessment workplan as part of the process for completing the RCRA facility permit. In its August letter, USEPA identified a variety of general requirements for the plans. In response to USEPA's request, WCAI selected Focus Environmental, Inc. to provide permitting assistance, overall project management, and preparation of the Performance Demonstration Test Plan. Focus Environmental, Inc. is an independent engineering consulting firm headquartered in Knoxville, Tennessee, and has no affiliation with WCAI or its parent company, USFilter, other than its contract to conduct the permitting activities for the Parker facility.

WCAI will select qualified and experienced performance testing contractors and laboratories to conduct all aspects of the Performance Demonstration Test, including overall test management, stack gas sampling, laboratory analysis, data review, calculation of results, and test reporting. These firms will be independent contractors having no affiliation with WCAI, or its parent company, USFilter, other than their contract to conduct the testing services for the Parker Facility.

2.0 FEED STREAM DESCRIPTION

2.1 FEED STREAM CHARACTERISTICS

The feed streams for the WCAI RF are described in the following sections.

2.1.1 Spent Activated Carbon

An aqueous slurry of spent activated carbon is the only material treated in the RF. The facility treats spent activated carbon that has typically been used for treating industrial and municipal wastewater, groundwater, surface water, process materials, or for air pollution control. Constituents in the streams being treated are adsorbed onto the surface and into the internal pores of the activated carbon. The activated carbon is said to be "spent" when it has adsorbed a certain amount of chemicals. The amount of chemicals adsorbed will vary from site to site, but generally the organic loading is no greater than 0.3 pounds of chemicals per pound of dry activated carbon. The organic loading on an average basis is much lower than the 0.3 pounds per pound of activated carbon maximum loading. Average loading data for the period from 1997 through 2001 indicated a range of 0.0038 to 0.0071 pounds of organic per pound of dry carbon, with an overall weighted average of 0.0055 pounds of organic per pound of dry carbon.

The number of different regulated constituents adsorbed on the activated carbon from a given source depends on the composition of the stream being treated. The list of organic constituents that may be adsorbed on spent carbon is very extensive, and includes, but is not limited to, volatile organic compounds, polynuclear aromatic hydrocarbons, phthalates, amines, and pesticides. Activated carbon is not customarily used to remove metals from a waste stream, although, low concentrations may be expected in the spent carbon. Actual facility data for the spent activated carbon is included in Tables 2-1 and 2-2. The spent activated carbon will be received, stored and handled as per the Waste Analysis Plan located in the facility's RCRA Part B permit application. The facility will not accept spent carbon containing TSCA-regulated levels of PCBs, infectious wastes, regulated levels of radioactive wastes (as regulated by the Nuclear Regulatory Commission) or spent carbon exhibiting the characteristics of corrosivity or reactivity. Additionally, WCAI will not accept spent activated carbon that is classified as a dioxin-listed hazardous wastes (i.e., those carrying EPA Waste Codes F020, F021, F022, F023, F026, and F027).

2.1.2 Fuels

The only auxiliary fuel used for the RF is natural gas. Typical characteristics of natural gas are included in Table 2-3.

2.1.3 Other Feed Streams

The only other feed stream to the RF is steam. The steam is used in the RF to assist in the reactivation process of the spent carbon. The steam is not expected to contain any RCRA Appendix VIII Hazardous Constituents or Hazardous Air Pollutants (HAPs).

2.2 FEED STREAM MANAGEMENT

The feed stream management of the spent carbon is described in the following sections.

2.2.1 Storage

Spent carbon is received by truck in containers (i.e., drums, vessels, supersacks, roll-off bins, etc.) or in tank trucks. Following inspection and acceptance at the facility, containerized spent carbon is unloaded in the unloading and receiving area where it is inspected and sampled. If the load is accepted for treatment, the containerized spent carbon is either transferred into one of the four spent carbon storage tanks via a feed hopper or moved to the Container Storage Area.

Spent carbon received in large containers, such as roll-offs and slurry trailers, is typically transferred directly to the spent carbon storage tanks through a feed hopper. Spent carbon received in smaller containers, such as drums, is typically moved to the container storage area in the containers in which it was received and subsequently transferred to the spent carbon storage tanks. The containerized spent carbon is transferred to the storage tanks via a hopper because it cannot be pumped directly from the container to the storage tank. Water is added as the carbon passes through the hopper to facilitate removal of the spent carbon from the hopper via an eductor. The carbon is transferred to the storage tanks as a water-carbon slurry.

The tank trucks carrying the bulk loads are retained in the unloading and receiving area and the spent carbon is inspected and sampled. If the shipment is accepted for treatment, the spent carbon is transferred in slurry form to one of the four process storage tanks, directly or through a feed hopper. Water used in the transfer process is supplied from the recycle water system

which consists of two recycle water storage tanks and associated valves and piping. The recycle water is periodically monitored and pH-adjusted, when required, for corrosion control. From the process storage tanks, the carbon is transferred in slurry form to the Carbon Reactivation Furnace (RF).

2.2.2 Blending

Blending of the spent carbon is required to control the concentration of chlorine and chlorides present in the feed material to maintain compliance with WCAI's wastewater discharge limit for TDS.

3.0 ENGINEERING DESCRIPTION

A block flow diagram of the carbon reactivation process is shown in Figure 3-1. Spent carbon slurry is fed from the Furnace Feed Hopper into a dewatering screw where the carbon is dewatered prior to introduction into the Carbon Reactivation Furnace (RF). Water from the dewatering screw is returned to one of the two recycle water storage tanks. The RF is a multiple hearth furnace consisting of five hearths. The spent carbon is introduced into the top hearth and flows downward through the remaining four hearths. Reactivated carbon exits the bottom hearth through a cooling screw. The RF is equipped with a primary combustion air fan and two shaft cooling fans. Steam from a small boiler is introduced into the RF to complete the reactivation process. Natural gas burners are provided to ensure adequate heat input to the reactivation unit for all of the spent carbons that are reactivated at the facility. The hot gases generated in the RF flow upward through the hearths and exit from the topmost hearth and are routed to an afterburner to ensure the thermal oxidation of any organic matter that is not oxidized in the reactivation unit. The afterburner is equipped with two burners that utilize natural gas as the fuel source. From the afterburner, the gases are quenched by direct water contact and routed through a variable throat venturi scrubber for particulate matter control. From the venturi scrubber, the gases are routed to a packed bed scrubber for acid gas control. From the packed bed scrubber, the gases flow through a wet electrostatic precipitator, used for fine particulate matter and metals control. From the wet electrostatic precipitator (WESP), the gases are routed through a stack to the atmosphere. The motive force for moving the gases through the air pollution control system is supplied by an induced draft fan located between the WESP and stack.

A pH-controlled scrubbing medium (water and caustic solution) is supplied to the air pollution control system from the scrubber water system. The pH is continuously monitored to ensure efficient acid gas removal in both the quench/venturi scrubber and the packed bed scrubber. Caustic is added based on the pH of the scrubber water.

The air pollution control equipment uses a closed loop recycle water system. Periodically, a portion of the scrubber water in the system is discharged (blowdown) in order to prevent the excessive build-up of total dissolved solids in the scrubber water system.

Scrubber blowdown from the RF air pollution control equipment is either discharged directly to the Publicly Owned Treatment Works (POTW) or is treated in a RCRA-exempt wastewater treatment unit, and then discharged to the POTW. The discharge to the POTW is continuously monitored for pH, total dissolved solids, flow and temperature to ensure compliance with the discharge limitations found in the facility's industrial wastewater discharge permit.

3.1 THERMAL TREATMENT SYSTEM

The thermal treatment system is a multiple hearth furnace, consisting of five hearths followed by an afterburner. Spent carbon is introduced into the top hearth of the reactivation unit and flows downward through the remaining four hearths. The top two hearths are unfired hearths. Hot combustion gases generated in the bottom three hearths are used to complete the dewatering of the spent carbon. The bottom three hearths are fired hearths where the reactivation process occurs. Rabble arms, with teeth, each connected to a rotating center shaft, are located above each hearth. The rabble teeth plow the carbon material across the hearth surface and towards drop holes. The carbon falls through the drop holes to the next lower hearth, and eventually to the outlet of the reactivation unit. Reactivated carbon exits the bottom hearth through a cooling screw. The RF is equipped with a primary combustion air fan, and two center shaft cooling fans. Steam from a natural gas fired boiler is introduced into the RF to complete the reactivation process. Natural gas burners are provided to ensure adequate heat input to the reactivation unit for all carbons that are reactivated at the facility.

3.1.1 Type, Manufacturer's Name and Model Number

The RF is a multiple hearth furnace consisting of five hearths and an afterburner manufactured by Hankin Environmental Systems, Inc. The Hankin multiple hearth furnace is a 12'10 ³/₄" O.D. x 5 Hearths designed to nominally reactivate 2,760 lb/hr of spent carbon feed. Drawings and specifications for the multiple hearth furnace and afterburner are presented in Attachment E.

3.1.2 System Capacity

The RF unit is currently authorized to reactivate 2,760 pounds per hour of spent carbon feed.

3.1.3 Thermal Treatment and Combustion Chamber(s)

Following dewatering the spent granular carbon is fed to the top section of the multiple-hearth furnace. In the pre-drying and drying zones (the top hearths) the water retained in the pores

and on the surface of the carbon is evaporated by the counter-current flow of hot combustion gases. The temperature of the carbon is raised to approximately 210°F. Upon application of heat, water will evaporate freely when the particle temperature goes over 200°F. The adsorbed water is freed at temperatures of approximately 212°F to 230°F.

Upon the application of heat to the particles at temperatures over 600°F, the high molecular weight organic impurities will crack to produce gaseous hydrocarbons, hydrogen and water vapor which escape the pores of the granular carbon while some fixed carbon is retained in the pores of the granules. In these pre-heating and decomposition zones (middle hearths) the temperature of the carbon is increased to about 750°F in a virtually oxygen-free atmosphere. Under these conditions the adsorbed organic impurities in the pores of the carbon are pyrolyzed and all volatile materials are driven off.

The carbonaceous residue, left behind in the pore structure of the carbon by the pyrolyzation process, is oxidized in the final heat up and gasification zones (lower hearths) in a way which avoids damage to the original pore structure of the carbon. This is the most important commercial phase of the reactivation process, with the critical parameters being carbon temperature, retention time, and steam and carbon dioxide concentration in the furnace atmosphere. Carbon bed temperatures may range from 1450°F-1600°F. Combustion of natural gas provides the required heat. Carbon dioxide and some water vapor are part of the products of combustion. Steam is injected as necessary.

The afterburner is a self-supporting vertical cylindrical chamber approximately 33 feet high with an inside refractory diameter of approximately 5 feet. The design incorporates a mixing zone, choke ring and a minimum residence time at temperature of greater than one second. The afterburner shell is constructed of steel plate and is internally lined with firebrick and castable insulation. The afterburner is equipped with two low NO_x burners, which utilize heated combustion air. The afterburner chamber is fitted with a total of six air injection nozzles which are placed to provide combustion air and turbulence to promote the oxidation of organic materials in the flue gas. The afterburner is designed to thermally oxidize greater than 99.99 percent of all organic matter entering the afterburner in the furnace off gas. A cross-section of the afterburner and the specification for the afterburner can be found in Attachment E. Actual material usages will be those listed in the specification or their functional equivalent.

3.1.4 Residence Time Determination

The residence time for the solid carbon in the Carbon Reactivation Furnace is 42 minutes at a shaft speed of 1 rpm.

3.2 BURNER AND FEED SYSTEMS

Six natural gas burners are installed in the RF, two per hearth on hearths 3, 4, and 5. Two natural gas burners are installed in the afterburner.

3.2.1 Burner Description

The six burners installed in the RF are North American Manufacturing Company burners (NA 6422-6) or their functional equivalent. The two burners installed in the afterburner are North American Manufacturing Company burners (NA 6514-8-B) or their functional equivalent. Literature describing these burners can be found in Attachment E. Materials of construction of these burners are listed in the literature.

3.2.2 Spent Activated Carbon Feed System

The spent activated carbon feed system to the RF consists of a feed hopper, a dewatering screw, and a weigh belt conveyor. The spent carbon/recycle water slurry is discharged from the feed hopper to the dewatering screw via a control valve. The dewatered spent carbon is discharged from the dewatering screw on to the weigh belt conveyor, which is used to measure the feed rate to the RF.

3.2.3 Auxiliary Fuel System

The six burners in the RF and the two burners in the afterburner are fired with natural gas, supplied by the local utility company via pipeline.

3.2.4 Combustion Air

Combustion air is supplied to the six RF burners and two afterburner burners by a combustion air blower. The blower is designed to supply approximately 351,600 ACFH of preheated combustion air. Fan specifications are located in Attachment E.

3.3 REACTIVATED CARBON HANDLING SYSTEM

The reactivated carbon exiting from the RF is a product. The reactivated carbon is discharged from the RF into a screw cooler and from the screw cooler through an enclosed conveyor system into one of three reactivated carbon product storage tanks. From the reactivated carbon storage tanks, the reactivated carbon product is transported through an enclosed conveyor to a product packaging facility. At the product packaging facility, the reactivated carbon is removed from the storage tanks and placed in appropriate containers for shipment to customers.

Scrubber blowdown from the RF air pollution control equipment is treated in a RCRA-exempt wastewater treatment unit, or discharged directly to the POTW. The discharge to the POTW is continuously monitored for pH, total dissolved solids, flow and temperature to ensure compliance with the discharge limitations found in the facility's industrial wastewater discharge permit.

3.4 AIR POLLUTION CONTROL (APC) SYSTEM

The APC system for the RF includes a quench/venturi scrubber, a packed bed scrubber and a wet electrostatic precipitator. Exhaust gases from the thermal treatment system are continuously routed through the APC equipment, and cannot by-pass the APC equipment under any circumstances. The individual components of the APC equipment are described in the following sections.

3.4.1 Quench/Venturi Scrubber

The Quench/Venturi Scrubber is a dual-purpose device used to rapidly quench the hot combustion gases exiting the afterburner and to remove particulate matter. The quench section uses water sprays to cool the afterburner exit gas to the point of adiabatic saturation (approximately 170 to 190°F). The venturi scrubber has an adjustable throat, and is a low energy, vertical down flow type. The throat area is adjusted by a pneumatic cylinder actuator and an electro/pneumatic positioner. The remotely adjustable throat is automatically controlled to maintain a constant pressure differential. The venturi scrubber is located directly below the quench section and is connected by a flooded elbow to the packed bed scrubber. The elbow incorporates a water-filled gas impact section directly beneath the throat to prevent erosion of the shell. The water supply for quench and venturi irrigation is recirculated scrubber water at a total flow of approximately 7.5 gpm/1000 ACFM.

The design data and equipment descriptions for the quench/venturi scrubber as well as a description of the physical dimensions of the venturi scrubber section can be found in Attachment E. Actual material usages will be those listed therein, or their functional equivalent.

3.4.2 Packed Bed Scrubber

The packed bed scrubber consists of a vertical up flow and cylindrical disengaging section followed by a packed bed section and mist eliminator. The bottom portion of the scrubber is used to separate entrained water droplets from the gas prior to entering the packed section of the scrubber.

The packed bed scrubber is designed to remove a minimum of 99 percent of the incoming hydrogen chloride.

The design data and equipment description for the packed bed scrubber as well as a description of the physical dimensions of the packed bed scrubber can be found in Attachment E. Actual material usages will be those listed therein or their functional equivalent.

3.4.3 Wet Electrostatic Precipitator

The wet electrostatic precipitator (WESP) is a vertical hexagonal tube design with self-irrigating tubes. The WESP consists of inlet gas distribution to promote even distribution of the process gas flow entering the WESP, inlet and outlet plenums and a collecting electrode tube bundle. The WESP is equipped with outboard high voltage insulator compartments which include a purge air system, high voltage distribution-support grids, high intensity rigid tube type charging/precipitating discharge electrodes, high voltage power supply (transformer/rectifier and controller) system, ground sticks, safety key interlocks, warning labels, and electronic control logic equipment and valving.

The WESP, in conjunction with the venturi scrubber, is designed to achieve a maximum outlet particulate matter grain loading of 0.015 grains/dscf adjusted to 7 percent oxygen.

The design data and equipment description for the WESP as well as a description of the physical dimensions of the WESP can be found in Attachment E. Actual material usages are those listed therein or their functional equivalent.

3.4.4 ID Fan

A variable speed induced draft fan is provided to exhaust combustion gases from the furnace and afterburner and through the air pollution control system. Design specifications for the fan can be found in Attachment E. Actual materials of construction will be those listed in the specification or their functional equivalent.

3.4.5 Stack

The treated gas stream is exhausted to the atmosphere via a 110 foot high stack with an inside diameter of two feet and a gas outlet that is 19.75 inches in diameter. The stack is equipped with ports for continuous emissions monitoring, stack gas flow rate monitoring, and exhaust gas sampling. A stack drawing is provided in Attachment E. Actual materials of construction will be those listed in the specification or their functional equivalent. Additional sampling ports may be installed for the extensive sampling to be conducted during this Performance Demonstration Test. A stack sampling port location drawing is included in Attachment E.

3.5 PROCESS MONITORING, CONTROL, AND OPERATION

The facility is equipped with a programmable logic control (PLC) system which monitors and/or controls process variables to ensure proper facility operation. The RF system is equipped with instrumentation to monitor and control process flows, temperatures, and pressures, and to transmit signals to the main control system. The automation system has the capabilities of controlling valves, motors, pumps, and fans as well as alarming and initiating waste feed cutoff interlocks if process conditions deviate from established limits.

Figure 3-2 shows the location of pertinent instrumentation related to permit compliance. Complete Piping and Instrumentation Diagrams (P&IDs) are included in Attachment E. It is important to note that these drawings include many components of the facility that are exempt from permitting under various provisions of RCRA. These components are provided for informational purposes and ease of review only, and they are not intended to become regulated components of the facility. Information concerning the major process instruments associated with regulatory compliance is presented in Table 3-1. Instrument tag numbers correspond to the designations shown on the P&IDs. Calibration schedules are based on manufacturer's recommendations and WCAI operating experience.

Process monitoring and emissions monitoring performed for regulatory compliance is conducted on a continuous basis in accordance with USEPA definitions of continuous monitors.

A "Continuous Monitor" is a device (or series of devices) which continuously samples the regulated parameter without interruption, evaluates the detector response at least once every 15 seconds, and computes and records the average value at least every 60 seconds, except during periods of calibration or as otherwise allowed by the applicable regulations or guidelines. For many parameters, rolling averages are calculated. A "Rolling Average" is defined as the arithmetic mean of a defined number of the most recent one-minute average values calculated by the continuous monitor. For example, an hourly rolling average would incorporate the 60 most recent one-minute average values. As each new one-minute average value is computed, the least recent of the 60 values is discarded and a new hourly rolling average is calculated and recorded. 12-hour rolling averages use 720 one-minute average values rather than 60.

Two subsets of continuous monitoring systems are employed on the RF: process continuous monitoring systems (CMS) and continuous emissions monitoring systems (CEMS). The following is a discussion of each type of continuous monitoring system.

3.5.1 Process Continuous Monitoring System (CMS)

Figure 3-2 shows the general location and function of the temperature, pressure, and flow indicating and control devices for the carbon reactivation system. The specifications for these devices are shown in Table 3-1.

The following is a discussion of each type of process monitoring and control to be performed in the RF system for regulatory compliance purposes.

Spent Activated Carbon Feed Rate

The flow rate of the spent activated carbon is monitored and controlled using a weigh belt conveyor and carbon slurry feed valve. When the feed valve is open, carbon slurry drops into the dewatering screw and is then discharged onto the weight belt conveyor, which feeds the carbon to the RF. The feed rate control system consists of a weigh cell, weight transmitting element, weight indicating controller, variable timed open/closed carbon slurry feed valve, and continuous weight feed rate recorder. The desired spent activated carbon feed rate is achieved

by the control system adjusting the time that the carbon slurry feed valve is open and closed. Automatic waste feed cutoff interlocks stop the weigh belt conveyor which stops the feed of carbon to the RF.

Regulated Constituent Feed Rates

The total feed rate of mercury, semivolatile metals (the combination of cadmium plus lead), and low volatility metals (the combination of arsenic, beryllium, plus chromium) will be continuously monitored and recorded in accordance with the HWC MACT regulations. This will be accomplished by the process computer which continuously monitors the flow rate of spent activated carbon, and multiplies that flow rate by the constituent concentration, which is input to the computer whenever the feedstream characterization is updated. If a regulated constituent is believed to have the potential to be present in the spent activated carbon, but is not detected by the relevant analysis, then the detection limit for that constituent will be used in the calculation. If a constituent is not expected to have the potential to be present in the spent activated carbon, then the concentration of that constituent will be set as zero.

Afterburner Temperature

The RF afterburner combustion temperature is continuously measured by thermocouples located in the afterburner chamber. The automatic temperature controller accepts the signal from the thermocouple and manipulates the auxiliary fuel feed rate. The automatic waste feed cutoff interlock is activated during low temperature conditions.

Venturi Pressure Differential

Venturi scrubber pressure differential is measured and controlled as an indicator of the energy supplied for particulate matter removal. A minimum pressure differential is necessary for proper control efficiency. The pressure differential is continuously measured by a pressure differential indicator with pressure taps located at the inlet and outlet of the venturi. The pressure differential is controlled by changing the position of the venturi throat control valve elements.

Quench/Venturi Scrubber Liquid Flow Rate

The recycle flow rate is continuously monitored using magnetic flow meters in the recycle water lines. A minimum recycle water flow rate is maintained in order to provide sufficient cooling and

scrubbing water for particle removal. A low total recycle flow rate will initiate an automatic waste feed cutoff.

Packed Bed Scrubber pH and Flow Rate

The packed bed scrubber recycle pH and the flow rate of recycled liquid to the packed bed scrubber influence the effectiveness of acid gas removal. The pH is measured continuously by an in-line pH probe installed in the recycle liquid piping. The recycle flow rate is continuously monitored using a magnetic flow meter in the recycle water line. Either low pH or low packed bed scrubber recycle flow rate will initiate an automatic waste feed cutoff.

Packed Bed Scrubber Pressure Differential

The differential pressure across the packed bed is measured as an indicator of proper liquid and gas distribution in the tower. The pressure differential is continuously measured by a differential pressure element with taps located at the inlet and outlet of the packed bed scrubber.

WESP Secondary Voltage

Although the HWC MACT regulations, do not require monitoring of any WESP performance indicators, WCAI will monitor the secondary voltage as an indicator of proper collection of fine particles and metals. A minimum secondary voltage of 14 kVDC has been established based upon operating experience.

Scrubber Blowdown Flowrate

In order to conserve water, WCAI recycles most of the liquid from the air pollution control system. In order to prevent the buildup of dissolved solids, WCAI bleeds water from the system. As water is bled, fresh makeup water is added. The APC system blowdown flow rate is continuously monitored using a magnetic flowmeter, and a low flow rate will trigger an automatic waste feed cutoff.

Stack Gas Flow Rate

The flow rate of stack gases is used as the indicator of combustion gas velocity prescribed by the applicable regulations. A flow sensor located in the stack provides the direct flow measurement. High stack gas flow rate will initiate an automatic waste feed cutoff.

3.5.2 Continuous Emissions Monitoring System (CEMS)

The exhaust gases are continuously monitored for carbon monoxide and oxygen content as an indicator of proper operation of the combustion process. To ensure these monitors are functioning properly, they are calibrated according to the protocols specified in the Appendix to 40 CFR 63 Subpart EEE, and Performance Specification 4B of 40 CFR 60 Appendix B.

The oxygen analyzer is an Ametek FCA-Control paramagnetic analyzer. The carbon monoxide analyzer is a Thermo Environmental Model 48, non-dispersive infrared monitor having a dual range of 0-100 ppm and 0-1000 ppm.

Performance specifications for the CEMS are shown in Table 3-2. Additional specifications, as well as a drawing of the sampling system can be found in Attachment E.

3.5.3 Safety and Automatic Waste Feed Cutoffs

The control system includes an automatic waste feed cutoff (AWFCO) system that stops the feed of spent activated carbon when operating conditions are at or near limits necessary to comply with specific permit conditions. In addition, the spent activated carbon feed is automatically stopped if the range of the measurement instrument is exceeded or if there is a malfunction of the continuous monitoring system. A listing of the AWFCO parameters is provided in Table 3-3. When any of these parameters deviates from the established limit, an electronic signal from the control system will stop the carbon weigh belt feeder. Anticipated limits for these and other permit conditions are discussed in Section 7.0 of this plan.

On a monthly basis, during RF operations, the AWFCO system will be tested, without feed interruption, by running a software routine to check PLC logic functions and alarm logging associated with the AWFCO subsystem. Each of the regulatory AWFCOs will be tested by using a control system console to input a software value which corresponds to an exceedance of the permit limit. Verification will then be made that the control system, in response to the test input, sends out a signal to trigger AWFCOs. The alarm logs will be checked to verify that the appropriate alarm was recorded. It should be noted that during the brief period of time when the AWFCO parameters are being tested, regulatory AWFCOs will be precluded. A maximum time limit of one minute per test for each parameter will be imposed so as to minimize AWFCO downtime. Non-regulatory AWFCOs will not be affected by the test.

3.6 PROCEDURES TO RAPIDLY STOP WASTE FEEDS AND CONTROL EMISSIONS

3.6.1 Rapidly Stopping Spent Activated Carbon Feeds

The RF is controlled by a process control computer. WCAI has included alarms and waste feed cutoff interlock setpoints which will automatically stop the feed of spent activated carbon before any permit limits are exceeded. In the event any of these preprogrammed operating setpoints are reached, the computer will take automatic action to stop the carbon weigh belt conveyor to immediately stop spent activated carbon feed to the system. The same action to cease spent activated carbon feed can be activated from the control room by operating personnel. These actions do not necessarily constitute a shutdown of the RF; only a stoppage of spent activated carbon feed. The RF will normally operate on auxiliary fuel after spent activated carbon feed is ceased to maintain operating temperature.

3.6.2 Shutting Down the System

RF system shutdowns may occur for two reasons:

1. A loss or malfunction of systems or controllers critical to maintaining performance standards and operating requirements.
2. A scheduled shutdown for normal maintenance or other operational purposes.

In the event of a system failure, the RF system is equipped with spent activated carbon feed and fuel shutoff mechanisms which fail to the "safe" (closed or off) position. Critical automation equipment or instrumentation failures will result in automatic stoppage of spent activated carbon feed and partial or complete system shutdown, depending on the severity of the failure or malfunction. Operations personnel have the ability to initiate an emergency system shutdown manually from the control room, although a controlled shutdown is preferred. Complete shutdown of the RF system can be undertaken as required in an orderly fashion to allow for a proper rate of cooling. WCAI maintains standard operating procedures including those for normal shutdown of the RF system. Normal and emergency system shutdown procedures are summarized in Attachment E.

3.6.3 Controlling Emissions During Equipment Malfunctions

The RF system is totally sealed to prevent fugitive emissions under all operating or malfunction conditions. Equipment shells and interconnecting ductwork are free from openings or gaps. Emissions from the spent activated carbon feed point are prevented through the use of a rotary

air lock on the multiple hearth furnace feed port. Emissions from the rotating parts in the multiple hearth are prevented by a sand seal. Reactivated carbon product handling is totally enclosed. Daily inspections are conducted in accordance with the inspection procedures of the RCRA Permit Application. Process gases are always directed through the emissions control equipment, and there are no provisions to bypass the air pollution control system. In addition, the emissions control equipment is among the last equipment to be taken off-line under any circumstance. In the event of an equipment malfunction affecting RF system performance, spent activated carbon feed is automatically discontinued. Stopping the spent activated carbon feed immediately eliminates the flow of untreated material into the RF system, however since the spent activated carbon takes 42 minutes to travel through the reactivation furnace hearths, a slight potential for emissions remains during this time. To the greatest extent possible, the afterburner and emissions control equipment will continue to operate while the malfunction is corrected. Spent activated carbon feed may be resumed once operating conditions have been returned within the permit limits. If the malfunction can not be corrected in a reasonable time frame or requires the unit to be taken off line, the reactivation furnace, afterburner, and APC systems will be shut down in an orderly fashion according to standard operating procedures. Spent carbon feed will not resume until the malfunction has been corrected and the entire RF system has been returned to operating conditions within the permitted limits.

3.6.4 Emergency Safety Vent Operations

The WCAI RF design does not require or utilize an emergency safety vent. Process gases are always directed through the emissions control equipment, and there are no provisions to bypass the air pollution control system.

4.0 TEST DESIGN AND PROTOCOL

4.1 REGULATORY REQUIREMENTS

A Carbon Reactivation Furnace is used by WCAI to reactivate spent activated carbon. Some of the carbon received at the Parker Facility is designated as a hazardous waste under the Resource Conservation and Recovery Act (RCRA) regulations. Much of the carbon received at the facility is not a RCRA hazardous waste, as it is either not a characteristic or listed waste. The RF is not a hazardous waste incinerator. "Hazardous waste incinerator" is defined in 40 CFR 63, Subpart EEE, as a "device defined as an incinerator in §260.10 of this chapter and that burns hazardous waste at any time." (40 CFR 63.1201). "Incinerator" is defined in 40 CFR 260.10 as "any enclosed device that: (1) Uses controlled flame combustion and neither meets the criteria for classification as a boiler, sludge dryer or carbon regeneration unit, nor is listed as an industrial furnace; or (2) Meets the definition of infrared incinerator or plasma arc incinerator. (emphasis supplied)" The RF does not qualify as an incinerator and instead is designated by Subpart X of the RCRA regulations as a Miscellaneous Unit. According to 40 CFR 264.601 of the Subpart X regulations, permit terms and provisions for a Miscellaneous Unit must include appropriate requirements of 40 CFR Subparts I through O and Subparts AA through CC, 40 CFR 270, 40 CFR 63 Subpart EEE, and 40 CFR 146.

Based on 40 CFR 264.601, WCAI will test the RF to demonstrate performance in accordance with the emission standards of 40 CFR 63 Subpart EEE applicable to existing incinerators. These emission standards are more stringent than the RCRA hazardous waste incinerator emission standards of 40 CFR 264 Subpart O. WCAI is proposing to establish operating limits to ensure continuing compliance with its permit using an approach which generally follows the specifications of 40 CFR 63 Subpart EEE and guidance prepared for RCRA incinerator permits. However, since those regulations and guidance do not strictly apply to this unit, but may be used as guidelines for the development of appropriate limits, WCAI is also proposing to establish some limits based on current operating practices and automation system capabilities, which have proven to be effective for this particular facility.

4.1.1 40 CFR 60 Subpart EEE CAA Requirements

As stated above, WCAI will test the RF to demonstrate performance in accordance with the emission standards of 40 CFR 63 Subpart EEE applicable to existing incinerators. According to

40 CFR 63.1201, an existing source under Subpart EEE is any affected source, the construction or reconstruction of which commenced on or before April 19, 1996. Part 63, Subpart A, defines "commenced", with respect to construction or reconstruction, as either (a) undertaking a continuous program of construction or reconstruction, or (b) entering into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or reconstruction.

WCAI signed a Trade Contract (No. 21-4527-AF) with Hankin Environmental Systems, dated October 17, 1995, to construct RF-2 (the currently operating RF unit). The contract was signed by Stephen McDonough (Hankin) and Mark Hepp (WCAI). A Purchase Order was written on December 27, 1995 to construct the concrete pad for RF-2, with actual pad construction beginning on December 29, 1995. The multiple hearth was erected in January 1996. WCAI has dated pictures showing the construction process. Startup occurred July 11, 1996. Consequently, WCAI had clearly entered into a contractual obligation to undertake the construction of RF-2 well before April 19, 1996, and under a continuous program of construction, the unit was completed within a reasonable period of time.

Since this RF system would qualify as an existing unit if it were subject to Subpart EEE, the appropriate emission standards for this unit are the standards for existing incinerators under the Hazardous Waste Combustor Maximum Achievable Control Technology (HWC MACT) regulations of 40 CFR 63, Subpart EEE. Specific requirements are summarized as follows:

- Demonstrate a DRE of greater than or equal to 99.99% for the selected POHCs chlorobenzene and tetrachloroethene.
- Demonstrate stack gas carbon monoxide concentration less than or equal to 100 ppmv, dry basis, corrected to 7% oxygen.
- Demonstrate stack gas hydrocarbon concentration of less than or equal to 10 ppmv, as propane, dry basis, corrected to 7% oxygen.
- Demonstrate a stack gas particulate concentration less than or equal to 34 mg/dscm (0.015 gr/dscf), corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of hydrogen chloride (HCl) and chlorine (Cl₂) are no greater than 77 ppmv, dry basis, corrected to 7% oxygen, expressed as HCl equivalents..
- Demonstrate that the stack gas mercury concentration is less than or equal to 130 µg/dscm, corrected to 7% oxygen.

- Demonstrate that the stack gas concentration of semivolatile metals (cadmium and lead, combined) is less than or equal to 240 µg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of low volatility metals (arsenic, beryllium, and chromium, combined) is less than or equal to 97 µg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of dioxins and furans does not exceed 0.40 ng/dscm, corrected to 7% oxygen, expressed as toxic equivalents of 2,3,7,8-TCDD (TEQ). This standard is applicable since the gas temperature entering the first particulate matter control device is less than 400°F.

4.1.2 Regulatory Policy Requirements

In addition to the specific regulatory requirements, WCAI has been requested to perform both a human health and an ecological risk assessment, in accordance with EPA policy. As such, the performance test must include specific data gathering activities for use in the risk assessments.

For this facility, those risk assessment data gathering activities are:

- Measure emissions of metals, including hexavalent chromium
- Measure emissions of specific volatile and semivolatile products of incomplete combustion (PICs)
- Measure emissions of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDF)
- Measure emissions of polychlorinated biphenyls (PCBs)
- Measure emissions of specific organochlorine pesticides
- Measure emissions of total volatile, semivolatile, and nonvolatile organics
- Determine the stack gas particle size distribution.

4.2 TEST OBJECTIVES AND APPROACH

The WCAI Performance Demonstration Test Plan has been prepared to provide comprehensive performance testing of the RF unit to demonstrate compliance with the applicable HWC MACT emission standards and to gather data for use in a site-specific risk assessment. The objectives of the PDTP are to demonstrate regulatory compliance with standards such as Destruction and Removal Efficiency (DRE) and particulate matter emission concentration (described above), while operating at “worst case” conditions processing normal feed materials, which have been augmented with metals, chloride, etc., to establish operating conditions that will be included in the permit.

4.3 TEST PROTOCOL

In order to accomplish the PDT objectives, (i.e., demonstrating that the unit will meet all applicable environmental performance standards) a single test condition representing “worst case” operations of minimum temperature, maximum combustion gas velocity (minimum residence time), and maximum spent activated carbon feed rate will be performed. The test will consist of at least three replicate sampling runs.

A summary description of the testing conditions, analytical parameters, and sampling methods follows:

4.3.1 Test Condition 1 (“Worst-Case” Operations)

Sampling and monitoring protocols that will be utilized while carrying out the performance test are summarized as follows:

- Spent Activated Carbon Feed - total chlorine/chloride, elemental (C, H, N, O, S, moisture), volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Makeup Water - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Caustic feed to APC - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Cu, Co, Pb, Hg, Ag, Tl, Se, Ni, V, Zn)
- Scrubber Blowdown - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Wastewater Discharge to POTW - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Stack gas particulate, HCl, and Cl₂ using EPA Method 26A
- Stack gas target volatile organics using VOST, SW-846 Method 0030
- Stack gas target semivolatile organics and organochlorine pesticides using SW-846 Method 0010
- Stack gas PAHs and PCBs using a separate SW-846 Method 0010 sampling train
- Stack gas PCDD/PCDF using SW-846 Method 0023A
- Stack gas total volatile organics using SW-846 Method 0040
- Stack gas total semivolatile and nonvolatile organics using SW-846 Method 0010
- Stack gas metals (Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn) using EPA Method 29
- Stack gas hexavalent chromium using SW-846 Method 0061
- Stack gas particle size distribution using a cascade impactor

- Stack gas CO and O₂ by permanently installed CEM according to the protocols in the Appendix to 40 CFR 63, Subpart EEE; Performance Specification 4B of 40 CFR 60, Appendix B.
- Stack gas total hydrocarbons (as propane) by temporary CEM according to EPA Method 25A and the protocols in the Appendix to 40 CFR 63, Subpart EEE.

4.4 FEED MATERIAL CHARACTERISTICS

4.4.1 Description and Composition of Spent Activated Carbon

Spent activated carbon is the only material treated in the RF. The current spent activated carbon profile data is presented in Tables 2-1 and 2-2. Actual spent activated carbon will be treated during the performance test. The spent activated carbon will be augmented, as discussed below, by the addition of POHCs (principle organic hazardous constituents), other organic surrogates, chlorine/chloride, and metals, as needed to achieve the test objectives. The expected composition of the spent activated carbon, as fed to the RF during the PDT, is summarized in Table 4-1.

4.4.2 Expected Constituent Levels in Natural Gas, Process Air, & Other Feed Streams

In addition to the spent activated carbon, WCAI feeds natural gas to the burners in the multiple hearth furnace and afterburner, ambient air for combustion, and steam to assist in the reactivation process. Neither the ambient air, nor the steam are expected to contain regulated constituents. Natural gas may contain low concentrations of metals, as shown in Table 2-3. These metals concentrations are so low that their contribution to emissions is negligible, and will not be considered further.

4.4.3 POHC Selection Rationale

The RF system exclusively treats spent activated carbon. A wide variety of organic contaminants may be present on the carbon, thus WCAI needs flexibility to treat carbon containing any RCRA Appendix VIII Hazardous Constituent or CAA HAP except for carbons classified as dioxin wastes (EPA Waste Codes F020, F021, F022, F023, F026, or F027) or containing TSCA-regulated levels of PCBs. The compounds to be used as POHCs during the PDT were selected for their ability to demonstrate the effectiveness of the unit in destroying compounds that are equal or more thermally stable, and are thus equal or more challenging to treat, than those currently found on the spent activated carbon. This provides assurance that the unit will be effective for all of the spent carbon contaminants.

EPA has developed a thermal stability ranking system based on laboratory studies conducted under low oxygen conditions in a non-flame environment. The EPA's thermal stability ranking system has divided organic compounds into seven thermal stability classes, with Class 1 compounds being the most stable, and Class 7 compounds being the least thermally stable. The EPA thermal stability ranking system is structured on the principle that if a combustion system is successful in destroying compounds in a particular class, it is appropriate to assume that other compounds within the same and lower classes will be destroyed at efficiencies equal to or greater than the efficiencies demonstrated.

A review of WCAI's spent activated carbon characterization information shows that compounds from several thermal stability classes, including Class 1, may be present on the spent activated carbon. WCAI has chosen to demonstrate the DRE of chlorobenzene (a Class 1 compound) and tetrachloroethene (a Class 2 compound) as the Principal Organic Hazardous Constituents during the Performance Demonstration Test. Chlorobenzene was chosen since it is a compound sometimes present on spent activated carbon received at the Parker Facility, thus its use is representative of normal operations. It is also relatively available and less hazardous to handle than other Class 1 compounds for spiking into the furnace during the PDT. Chlorobenzene is an aromatic compound and will also provide a source of organic chlorine to challenge the system during the performance test. WCAI believes that its choice of chlorobenzene as a POHC represents the most significant challenge possible to the thermal destruction capabilities of the RF unit.

Tetrachloroethene is a per-chlorinated aliphatic compound that is also sometimes present on spent activated carbon received at the Parker Facility. It represents a significant source of organic chlorine to the system, and was chosen as a POHC so the test would include both an aromatic and an aliphatic compound.

Since the selected POHC compounds rank among the most difficult to destroy on the thermal stability ranking system and represent a variety of aromatic and aliphatic compounds, successful DRE demonstration should allow WCAI to treat spent activated carbon represented by the waste codes in the facility's most current RCRA Part A permit application. WCAI will

spike chlorobenzene and tetrachloroethene, as needed for both DRE demonstration and as sources of organic chlorine.

Samples of the spent activated carbon, collected before spiking, will be analyzed for POHC content. The spent activated carbon feed POHC analyses and carbon feed rates will be used to determine the feed rate of native POHC, if any. WCAI will utilize the services of a spiking contractor to provide additional POHC spiking. The spiked POHC will be prepared by the contractor for spiking directly into the furnace. The contractor's certification of composition of the spiking material and the spiking logs will be used to determine the POHC spike rate. Samples of the spiking material will be collected and archived. For the DRE calculation, the POHC feed rates will include the native POHC in the spent activated carbon feed and the spiked POHC.

4.4.4 Feed Material Specifications for the Test

While a wide variety of organic compounds can be on the spent activated carbon, and the specific constituents and concentrations vary over time according to the generator, the actual material fed to the RF is quite homogeneous. As stated earlier, organic compounds can account for up to 0.3 pounds per pound of dry carbon from a given generator. However, when added to other more lightly loaded carbons, and processed for feeding, the actual feed material is typically low in organics. Based on actual feed data from 1997 to 2002, the feed stream is predominantly carbon granules (~56 to 57 wt%, wet basis) which are wet from the slurring and subsequent de-watering process (~43 wt% water). Average loading data indicated a range of 0.0038 to 0.0071 pounds of organic per pound of dry carbon, with an overall weighted average of 0.0055 pounds of organic per pound of dry carbon. On a wet (as fed) basis, the organic loading accounts for only about 0.2 to 0.4 wt% of the total feed. The variability in the feed is thus restricted to only this 0.2 to 0.4% of the total material.

For purposes of this test, the feed materials must support WCAI's need to demonstrate DRE for selected POHCs, demonstrate system removal efficiency (SRE) for representative metals, demonstrate maximum total chlorine/chloride feed rate, and gather emissions data for the risk assessment which are reasonably representative of the long-term operation of the RF system. The most desirable situation would be if carbon could be received and stockpiled from a number of sources such that the test feeds would contain sufficient quantities of POHCs, metals,

chlorine, and other organics for use during the test. Unfortunately WCAI has limited capacity for stockpiling materials, and cannot control when various generators send spent activated carbon for treatment. In light of this situation, WCAI will feed actual spent activated carbon available on-site, but will supplement it with POHCs, metals, chlorine, and other organics to meet the test objectives. Feed composition targets for the Performance Demonstration Test are shown in Table 4-1. Constituent feed rates and target process operating conditions for all three runs of the Performance Demonstration Test are shown in Table 4-2.

The POHCs, chlorobenzene and tetrachloroethene, will be spiked into the reactivation furnace in sufficient quantities for DRE demonstration and to add chlorine to the feed.

Lead will be spiked as a representative metal for the determination of semivolatile metal SRE. Spiking will be sufficient to ensure detection in the stack gas sample, and so that metal feed rate extrapolation can be reliably accomplished.

Chromium will be spiked as a representative metal for the determination of low volatility metal SRE. Spiking will be sufficient to ensure detection in the stack gas sample, and so that metal feed rate extrapolation can be reliably accomplished.

Based on a review of the organic constituents commonly found on the spent activated carbon routinely treated at WCAI, and based on the relative availability and handling safety considerations of these materials, WCAI will spike a mixture of organic compounds into the reactivation furnace to act as surrogates for the various classes of compounds routinely treated. This will ensure that the carbon fed during the test contains representative types, and higher than normal quantities, of organic compounds, in addition to those native to the spent carbon, and gives the feed materials the potential to produce a range of representative combustion products so that the risk assessment emissions data from the Performance Demonstration Test will be appropriate. In order to meet this objective, the following organic surrogates will be added to the furnace, in addition to the POHCs:

- Toluene
- 1,1,1-Trichloroethane
- Naphthalene

- Ethylene glycol.

These compounds represent aromatics, aliphatics, chlorinated compounds, non-chlorinated compounds, oxygenated compounds, as well as both volatile and semivolatile organics.

The spiked materials will be pumped into the reactivation furnace at the point where spent activated carbon enters the furnace. This location will ensure that all spiked materials enter the RF with the carbon. An injection manifold will be constructed to accommodate the spike material feed lines.

As shown on Table 4-1, and discussed above, the typical spent activated carbon metals concentration ranges from about 13 to 19 mg/kg. Spiking during the test will increase this concentration to between 240 to 280 mg/kg (over a fifteen-fold increase). Similarly, the total organic concentration of typical spent activated carbon is between 0.2 to 0.4 wt%, while the carbon used for the test will have the total organic concentration increased to approximately 3.5 to 5 wt% (over a ten-fold increase). The use of actual spent activated carbon will provide a variety of compounds which will produce representative emissions, and these compounds and their relative concentration may vary during the test (just as in normal operations) due to the limited ability to stockpile and blend. However, the addition of metals and organic surrogates is so much in excess of the expected native concentrations on the carbon, that any variability will be overshadowed by the spiking. Thus the overall composition of the carbon feed will be quite homogeneous during the three runs of the Performance Demonstration Test. Further, WCAI expects no significant variation in process operating conditions due to variability of the native carbon constituents, thus providing appropriate data for permitting decisions.

4.4.4.1 Chloride Content

The data presented in Table 4-1 include the target total chlorine/chloride content for the performance test carbon feed stream. The typical spent activated carbon contains varying amounts of organic chlorine associated with chlorinated organics adsorbed onto the carbon.

To demonstrate maximum total chlorine/chloride feed rate during the performance test, WCAI intends to spike chloride sources into the reactivation furnace. The chloride will be provided by the POHCs (chlorobenzene and tetrachloroethene), as well as the other chlorinated organic surrogates.

4.4.4.2 Metals Content

The data presented in Table 4-1 includes target performance test metal feed rates for the spiked and native metals.

Two approaches will be used in establishing the metals operating limits, based on a review of expected spent activated carbon compounds and the HWC MACT provisions for establishing metals feed rate limits:

WCAI will meet mercury limits which utilize the "Maximum Theoretical Emission Concentration" (MTEC) concept, where it is conservatively assumed that all feed mercury is emitted from the system. Based on the results of the performance test, WCAI will calculate a maximum mercury feed rate which will ensure compliance with the MTEC, and will continuously monitor the mercury feed rate to ensure that the limit is complied with. Since this approach does not take credit for actual removal across the APC system, it is the most conservative assumption for the low levels of mercury in the feeds.

Lead will be spiked during the test to determine the system removal efficiency (SRE) for semivolatile metals. Chromium will be spiked during the test to determine the SRE for low volatility metals. These SRE values will be used as the basis for establishing permit feed rate limits for the semivolatile and low volatility metals. The native metals content of the spent activated carbon feed is expected to be similar to that shown in Table 2-1.

4.4.5 POHC, Organic Surrogate, and Metal Spiking

A spiking system will be used for POHC, organic surrogate, and metals addition to the spent activated carbon feed. Three separate spiking systems will be used: one for the mixture of POHCs, one for the organic surrogate mixture, and one for the metals dispersion. Each spiking system will consist of a variable speed, positive displacement pump, (or equivalent system) that will transfer the spiking materials from containers onto the spent activated carbon just as it enters the RF. An electronic scale will be provided for each spiking material system so that a weighed amount of material will be metered into the RF and quantified for each test run. Technical grade POHCs and organic surrogates will be used as needed. Lead will be spiked as a dispersion of lead oxide, while chromium will be added as a dispersion of strontium chromate (a solid hexavalent chromium compound). Specifications for the spiking materials will be

provided by the spiking contractor. Calculations showing that the planned POHC feed rates are adequate to demonstrate the required DRE are presented in Attachment D. Metals spiking rates have been selected to allow for detection in the stack gas so that a rational SRE value can be determined. Based on the SRE, the metal feed rates will be extrapolated upwards to attain metals feed rate limits for the permit. Metal spiking rate calculations and anticipated permit feed rate limits, derived from extrapolation are shown in Attachment D.

WCAI will utilize the services of a spiking contractor to provide each of the spiking materials and to perform spiking operations during the test. The contractor's certification of composition of the spiking materials and the spiking logs will be used to determine the respective spike rates. Samples of the spiking materials will be collected and archived.

4.5 OPERATING CONDITIONS

Planned process operating conditions and feed rates are summarized in Table 4-2.

4.5.1 System Operation to Achieve Steady State Conditions

Carbon in the multiple hearth section of the RF has a residence time of 42 minutes. Test materials must be fed to the system for at least one residence time to ensure that the system is operating on test material only. There is no other "hold up" of feeds in the system and there is little in the way of "surge capacity" in the APC system, thus there is little "conditioning" necessary to bring the system to steady state conditions. However, since most process parameters are being monitored on an hourly rolling average basis, the process should be operated at test conditions (including spiking) for at least one hour prior to beginning a test run in order to allow the rolling averages to be established.

"Steady State" conditions will be achieved by bringing the system to the desired test conditions, and then operating the system at the desired test conditions (including any spiking operations) for a period of at least one hour prior to the beginning of each test run. Normal process fluctuations are expected during the test periods, just as during normal operations. Typically fluctuations of 10 to 20 percent of the "steady state" process value are considered normal for thermal treatment systems, however some parameters may experience even greater fluctuations while still being considered acceptable. WCAI operations personnel and the test

manager will closely monitor operations during the test periods and will make any necessary determinations regarding the acceptability of process fluctuations.

5.0 SAMPLING, ANALYSIS, AND MONITORING PROCEDURES

The following paragraphs discuss the planned sampling and analyses during the Performance Demonstration Test. The sampling procedures to be used during the performance test are located in Attachment A. Analytical methods are described in Attachment B. Quality Assurance and Quality Control procedures are described in the Quality Assurance Project Plan (QAPP) located in Attachment C.

5.1 SAMPLING LOCATIONS AND PROCEDURES

Sample collection points are shown on Figure 5-1. Sample collection locations, equipment, methods, and frequency are summarized in Table 5-1. The types of analyses planned for the performance test samples are outlined on Table 5-2. Analytical methods and procedures to be used for each sample are summarized in Table 5-3. Alternative methods may be used with the prior approval of the EPA.

5.1.1 Spent Activated Carbon Feed

Spent activated carbon feed samples will be collected during each test run as described in Attachment A. Spent carbon samples will be collected every 15 minutes and composited over the run. The feed samples collected will be analyzed for the parameters as indicated in Tables 5-2 and 5-3. Volatile organic analysis (VOA) samples will be collected from the run composite at the end of each run, and placed into a separate jar with minimum headspace. Analytical methods are presented in Attachment B.

5.1.2 Spiking Materials

POHC, Organic Surrogate, and Metals Spiking Feedstock Samples

POHC material will be pumped from portable containers into the RF, using metering pumps. The POHC injection point will be downstream of the point where spent activated carbon feed samples are collected. The POHC spiking rate will be determined using either flow meters or digital scales and logs of the differential weights at 10-minute intervals.

In addition to the POHCs, other organic surrogates will be spiked separately into the RF. These materials are being added to ensure that the test is conducted with a variety of organic compounds which have the potential to produce a wide variety of combustion products,

indicative of the range of materials to be treated at the WCAI Parker Facility. The organic surrogate spiking rate will be determined using either flow meters or digital scales and logs of the differential weights at 10-minute intervals.

Metals will be metered as necessary into the RF at an adjacent location to the POHC and organic surrogate spiking points. The metals will be supplied as aqueous solutions or as dispersions (aqueous or organic). The metal solution/dispersion injection point will be downstream of the ports where spent activated carbon feed samples are collected. The amount of each metal solution/dispersion spiked will be determined using either flow meters or digital scales and logs of the differential weights at 10-minute intervals.

WCAI will utilize the services of a spiking contractor to provide the POHCs, organic surrogates, and metals spiking materials and to conduct the spiking. The manufacturer's certification of composition of the spiking materials and the contractor's spiking logs will be used to determine the respective spike rates. Samples of the spiking materials will be collected once during the test and archived for analysis if required. The sampling procedure will be as described in Attachment A.

5.1.3 Process Makeup and Residue Streams

Makeup Water, Caustic, Scrubber Blowdown, and POTW Discharge Samples

Samples of the makeup water, caustic, scrubber blowdown, and POTW discharge streams will be collected during each test run as described in Attachment A. Samples of the makeup water and caustic will be collected once during the test. Samples of the scrubber blowdown and POTW discharge will be collected every 30 minutes during each test run. VOA samples of wet scrubber blowdown and POTW discharge will be collected and handled as discrete samples. VOA samples will be composited in the laboratory immediately prior to analysis. Grab samples of the scrubber blowdown and POTW discharge for the other analyses will be composited in the field to form one sample per run. Analytical methods are presented in Attachment B.

5.1.4 Stack Gas

5.1.4.1 Stack Gas Volatile Organics

A Volatile Organic Sampling Train (VOST) (SW-846 Method 0030), will be used to determine the stack gas emissions of the POHCs (chlorobenzene and tetrachloroethene) for DRE determination. Four pairs of VOST tubes will be collected during each sampling run, but only

three will be analyzed. The fourth tube pair will serve as an archive set in the case of breakage during shipment or laboratory handling. Attachment A presents a description of the stack gas VOST sampling method. The VOST tubes will be analyzed using SW-846 Method 8260.

In addition to the POHCs, the VOST samples will be analyzed for other volatile organic compounds. These analyses will be used to assess the emissions of volatile organic compounds for use in the risk assessment. The target volatile organic compounds are listed in Table 5-4. Analyses will be performed using SW-846 Method 8260. In addition to the target analyte list, analyses will include identifying non-target analyte peaks, which are referred to as tentatively identified compounds (TICs). A discussion of TIC identification and quantitation is presented in the QAPP (Attachment C). Analytical methods are presented in Attachment B.

5.1.4.2 Stack Gas Particulate, HCl, and Cl₂

The stack gas will be sampled for particulate, HCl, and Cl₂ according to EPA Method 26A. The sampling procedure is described in Attachment A. Particulate filters will be analyzed per EPA Method 5 procedures. Impinger solutions will be analyzed for chloride ion per SW-846 Method 9057 to determine the emissions of HCl and Cl₂. Analytical methods are presented in Attachment B.

5.1.4.3 Stack Gas Multiple Metals

Stack gas multiple metals will be collected using an EPA Method 29 sampling train according to the procedure in Attachment B. Metals will be analyzed by Inductively Coupled Plasma Spectroscopy/Mass Spectroscopy (ICP-MS) using SW-846 Method 6020. Mercury will be analyzed using Cold Vapor Atomic Absorption Spectroscopy (CVAAS) using SW-846 Method 7470. Target metal analytes are shown in Table 5-5.

5.1.4.4 Stack Gas Hexavalent Chromium

Stack gas hexavalent chromium samples will be collected using an SW-846 Method 0061 sampling train, as described in Attachment A. Analysis is according to SW-846 Method 7199, as described in Attachment B.

5.1.4.5 Stack Gas Semivolatile Organics, PAHs, Organochlorine Pesticides, and PCBs

The stack gas will be sampled to determine the emissions of the semivolatile organic compounds, for use in the risk assessment. Stack gas will be sampled for target semivolatile organic compounds (SVOCs), semivolatile TICs, polyaromatic hydrocarbons (PAHs),

organochlorine pesticides, and polychlorinated biphenyls (PCBs) using two separate SW-846 Method 0010 sampling trains. The extract from the analytical preparation of each Method 0010 sampling train will be split for analysis as follows:

Train 1: one portion for SVOC analysis by SW-846 Method 8270 [gas chromatograph/mass spectrometry (GCMS)], a second portion for organochlorine pesticides analysis by SW-846 Method 8081 (GC), and a final portion retained as an archive in case any of the analyses need to be repeated.

Train 2: one portion for high resolution gas chromatograph/high resolution mass spectrometry (HRGC/HRMS) PAH analysis, a second fraction for HRGC/HRMS analysis of PCBs, and a final portion retained as an archive in case any of the analyses need to be repeated.

Prior to testing, the XAD-2 resin of each train will be spiked with sampling surrogates of each target analyte group, as an added QC step. The sampling procedure is described in Attachment A.

The respective target SVOC, PAH, OC Pesticide, and PCB analytes for the Method 0010 samples are presented in Tables 5-6, 5-7, 5-8, and 5-9. In addition to the SVOC target analyte list, analyses will include identifying non-target analyte peaks (TICs) based on the nearest internal standard and library search.

5.1.4.6 Stack Gas Method 0023A (Dioxins and Furans)

Stack gas samples will be collected for PCDD/PCDF using a SW-846 Method 0023A sampling train. The sampling procedure is described in Attachment A. Analysis of PCDD/PCDF is conducted by high resolution GC/MS according to SW-846 Method 8290, and described in Attachment B. PCDD/PCDF target analytes are shown in Table 5-10.

5.1.4.7 Stack Gas Total Semivolatile and Nonvolatile Organics

For the risk assessment analysis, the performance test program includes determining the Total Organic Emissions (TOE) using the procedures in "Guidance for Total Organics" EPA/600/R-96/036, March 1996. The stack gas will be sampled for total semivolatile organic compounds (Boiling Points from 100°C to 300°C) and nonvolatile organic compounds (Boiling Points greater than 300°C) using a separate SW-846 Method 0010 sampling train. The sampling method is

described in Attachment A. No isotopically labeled sampling surrogates will be spiked to the XAD-2 resin used in this sampling train. The dichloromethane extracts of the pooled components of the sampling train will be used to determine the Total Chromatographable Organics (TCO) using Gas Chromatography/Flame Ionization Detector (GC/FID). The marker compounds are n-heptane and n-heptadecane because their boiling points are 98°C and 302°C, respectively.

The nonvolatile organics will be determined by a gravimetric procedure known as GRAV from the same pooled dichloromethane extract of the Method 0010 train components as the semivolatile organic components. The analytical methods are described in greater detail in Attachment B.

5.1.4.8 Stack Gas Total Volatile Organics

For the risk assessment analysis, stack gas will be sampled for total volatile organic compounds (Boiling Points <100°C). Tedlar bag samples of stack gas will be collected and measured for total volatile organics by field gas chromatograph (GC) according to SW-846 Method 0040. The SW-846 Method 0040 sampling procedure is described in Attachment A. Emphasis will be made on the identification of n-C₁ - C₇ hydrocarbons. In addition, the volatile organics collected in the condensate trap of the SW-846 Method 0040 will be analyzed by purge and trap GC/FID. The analytical procedure is described in Attachment B.

5.1.4.9 Particle Size Distribution

The risk assessment analysis requires the collection of particle size distribution data on the stack gas particulate emissions. A cascade impactor will be used to determine the particle size distribution. The cascade impactor will be operated at a single point of average velocity within the stack. The procedure to be used is described in Attachment A. Analysis of the cascade impactor filter substrates is the same as that used for particulate matter determination in Method 5.

WCAI has previously collected PSD information and is submitting this data set in lieu of testing. Refer to Section 9.0 and Attachment G for the PSD data.

5.2 ANALYTICAL PROCEDURES

Attachment B presents a description of the analytical methods to be used during the performance test.

5.3 MONITORING PROCEDURES

During the performance test, the stack gas will be continuously monitored by installed CEMS using the following procedures:

- Stack gas carbon monoxide by non-dispersive infrared (NDIR) analyzer
- Stack gas oxygen by paramagnetic analyzer

CEMS performance testing will be completed prior to conducting the PDT. Stack gas CO and O₂ monitors will be operated during the PDT according to the protocols of the Appendix to 40 CFR 63 Subpart EEE, and Performance Specification 4B of 40 CFR 60 Appendix B. The stack gas monitors will be checked daily during the performance test for calibration stability in accordance with WCAI's standard operating procedures.

Also during the performance test, the stack gas will be continuously monitored for total hydrocarbons, as propane (EPA Method 25A) using a portable monitor supplied by the stack testing contractor.

5.4 QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

Attachment C contains a Quality Assurance Project Plan for the Performance Demonstration Test.

5.5 EMISSION VALUES FOR USE IN RISK ASSESSMENTS

Many of the emissions determinations being made during the PDT are for risk assessment purposes rather than to demonstrate compliance with specific regulatory performance standards. WCAI suggests that the average emission rate determined from the PDT data be used for assessing risks, since the RF will be operated at "worst case" conditions during the PDT. It should also be noted that while the target analyte lists are extensive, approved and validated stack gas sampling and analytical methods are not currently available for several compounds identified as COPCs (Compounds of Potential Concern) in the risk assessment.

WCAI will estimate the emission rate of these compounds, by assuming that they are present in the spent activated carbon fed during the PDT at the “typical” concentration shown in the feedstream characterization (Table 2-2), and applying the average DRE determined for the test.

6.0 TEST SCHEDULE

6.1 PLANNED TEST DATE

Following approval of the test plan, WCAI needs to accumulate carbon necessary for conducting the test, and needs to schedule the delivery of spiking materials, and consider the availability of sampling and analytical contractors. The RF unit is currently operating, thus no startup or shakedown period is required, however WCAI may conduct limited preliminary testing prior to conducting the formal PDT. The pre-test will involve operating the RF system at the same conditions proposed for the PDT. The scope of any pre-test sampling and analysis will be a subset of the PDT sampling and analyses. Depending on the results of the initial pretest, additional pretests may be performed. The preliminary testing will be used to confirm that the proposed PDT conditions are attainable and appropriate for meeting the objectives of the testing program. Preliminary testing will also allow WCAI and its contractors to identify and correct any difficulties with the sampling, analytical, or QA/QC procedures specified in this test plan.

Currently WCAI believes that, depending on contractor and consultant schedules, it can be ready to conduct preliminary testing within 8 to 10 weeks of PDTP approval, and to conduct the PDT within 5 to 6 weeks after completion of the pre-test. WCAI will notify EPA at least 30 days before the planned date for starting the Performance Demonstration Test. The performance testing will be conducted over a period of approximately 5 consecutive days.

Day 1 – Equipment setup and system readiness confirmation

Day 2 – Test Run 1

Day 3 – Test Run 2

Day 4 – Test Run 3

Day 5 – Contingency/equipment demobilization.

Each of the three test run days are planned to be identical. A detailed daily schedule for these three days is presented in Table 6-1.

6.2 DURATION OF EACH TEST

The performance test will consist of three replicate sampling runs at a single test condition. The RF will be fed test materials 1 to 2 hours before a sampling run, and will be stabilized at test

conditions at least 1 hour before a sampling run. This will establish steady operation at process test conditions. Each test run is anticipated to have a duration of 4 to 6 hours, including port changes.

6.3 QUANTITY OF SPENT CARBON TO BE REACTIVATED

Spent activated carbon will be fed to the RF for up to an estimated 6 to 9 hours per day over a period of 3 testing days, assuming no interruptions. Allowing for 12 hours of spent activated carbon feed each test day (as a contingency), plus one extra contingency day (12 additional hours), the approximate amount of spent activated carbon used for testing purposes is 144,000 pounds.

6.4 DETAILED SCHEDULE OF PLANNED TEST ACTIVITIES

A planned schedule for a typical testing day is presented in Table 6-1.

6.5 PRELIMINARY TESTING

WCAI may conduct preliminary testing prior to the formal Performance Demonstration Test. The purpose of any such preliminary testing will be to verify that the planned Performance Demonstration Test operating conditions, as well as the planned spiking, sampling, and/or analytical methods are appropriate and yield acceptable results. A further goal of preliminary testing is to ensure that any test team interaction, coordination, or logistics issues are addressed prior to conducting the formal test.

Tables 4-1 and 4-2 show the anticipated feed materials, compositions, feed rates, and operating conditions planned for the test. Preliminary testing, if conducted, will be targeted to achieve the same conditions as shown in Tables 4-1 and 4-2. Process and emissions sampling and analysis for the Performance Demonstration Test are summarized in Tables 5-1, 5-2, and 5-3. Preliminary testing, if conducted, will utilize all, or a portion, of these same sampling and analytical methods.

7.0 OPERATING PERMIT OBJECTIVES

Based on the results of the performance test, WCAI will propose operating limits for the RF system that assure continuing compliant performance. Parameters will be established from the operating conditions demonstrated during the test; established regulatory requirements; manufacturer's recommendations; good operating practice, or on the results of the risk assessments. If the required performance objectives of the test are achieved, the RF system should be allowed to operate under the conditions proposed in this section.

Table 7-1 summarizes the anticipated operating limits. In order to achieve the desired conditions and demonstrate operations at the limits shown in Table 7-1, the interlock setpoints for certain operating parameters must be set somewhat wider during testing periods. The recommended interlock setpoints during testing periods are presented in Table 7-2. The following sections present a discussion of each parameter.

7.1 CONTROL PARAMETERS

WCAI anticipates limits on a number of operational control parameters which must be maintained as an indication that the RF system will continue to operate in compliance with all regulatory standards. Control parameters are grouped into categories:

- Group A1 parameters are continuously monitored and recorded, and are interlocked with the automatic waste feed cutoff system. Group A1 parameter limits are established from test operating data, and are used to ensure that system operating conditions are equal to or are more rigorous than those demonstrated during the test. During the test periods (pretest and performance test), interlocks for Group A1 parameters will be operational, but will be set at values which will allow the desired operating limits to be demonstrated.
- Group A2 parameters are continuously monitored and recorded, and are interlocked with the automatic waste feed cutoff system. Group A2 parameter limits are established based on regulatory requirements rather than on the test operating conditions, e.g., the maximum stack CO concentration. Interlocks for Group A2 parameters will be operational during the test periods, without modification to the interlock setpoints.
- Group B parameters are continuously monitored and recorded, but are not required to be interlocked with the automatic waste feed cutoff system. Operating records are required to ensure that established limits for these parameters are not exceeded. The Group B parameter limits are established based on the operation of the system during the test.

- Group C parameters are continuously monitored and recorded, but are not required to be interlocked with the automatic waste feed cutoff system. Group C parameter limits are based on manufacturer's recommendations, operational safety, and good operating practice considerations rather than on the test operating conditions, e.g., the minimum packed bed scrubber pressure differential.

7.2 DEVELOPMENT OF PERMIT LIMITS

The following sections describe how each control parameter limit will be established.

In addition to establishing specific operating limits, WCAI has limits on the types of waste which can be accepted in the RF. Since WCAI will demonstrate performance while treating a Class 1 (most thermally stable) compound, it is expected that WCAI will be permitted to treat spent activated carbon having EPA waste codes as represented in the facility's most recent RCRA Part A permit application. Specific prohibitions are expected for wastes containing greater than 50 mg/kg of PCBs and those wastes listed with the waste codes F020, F021, F022, F023, F026 or F027.

7.2.1 Group A1 Parameters

Group A1 parameter limits are based on the results of the performance test. The following parameters are proposed as Group A1 parameters for the RF system.

7.2.1.1 Maximum Spent Carbon Feed Rate

The performance test will be conducted in order to demonstrate the maximum feed rate of spent activated carbon. The spent activated carbon feed rate will be monitored on a continuous basis. The maximum allowable spent activated carbon feed rate will be established from the mean of the average feed rates demonstrated during each run of the performance test. The feed rate limit will be monitored as 1-hour block average.

7.2.1.2 Minimum Afterburner Temperature

The performance test will be conducted to demonstrate the minimum afterburner temperature with maximized combustion gas flow (minimum residence time), since these conditions are least favorable for DRE. Organic emissions are also being measured for risk assessment purposes since it is expected to represent worst case conditions for organic emissions. Combustion gas temperature will be monitored in the afterburner on a continuous basis. Based on successful

demonstration of DRE during the performance test, the minimum temperature limit will be based on the average of the minimum hourly rolling average temperature values demonstrated during each run of the performance test. The permit limit is expected to be administered as an hourly rolling average.

7.2.1.3 Minimum Venturi Scrubber Differential Pressure

The performance test will be conducted to demonstrate the minimum venturi scrubber differential pressure. Venturi scrubber differential pressure will be monitored on a continuous basis. Based on successful demonstration of particulate control during the performance test, the minimum venturi scrubber differential pressure limit will be based on the mean of the minimum hourly rolling average values demonstrated during each run of the performance test. The permit limit is also expected to be an hourly rolling average value.

7.2.1.4 Minimum Quench/Venturi Scrubber Recycle Liquid Flow Rate

The performance test will be conducted to demonstrate the minimum quench/venturi scrubber recycle flow and maximum stack gas flow, thus establishing a *de facto* minimum liquid to gas ratio. Quench/Venturi scrubber flow and stack gas flow will both be monitored on a continuous basis. Based on successful demonstration during the performance test, the minimum quench/venturi scrubber recycle liquid flow rate limit will be based on the mean of the hourly rolling average values demonstrated during each run of the performance test.

7.2.1.5 Minimum Packed Bed Scrubber pH

The performance test will be conducted to demonstrate the minimum packed bed scrubber pH at maximum total chlorine/chloride feed rate. Scrubber pH will be monitored on a continuous basis. Based on successful demonstration of HCl and Cl₂ control during the performance test, the minimum packed bed scrubber pH limit will be based on the mean of the minimum hourly rolling average pH values demonstrated during each run of the performance test. The permit limit will be administered as an hourly rolling average.

7.2.1.6 Minimum Packed Bed Scrubber Recycle Liquid Flow Rate

The performance test will be conducted to demonstrate the minimum packed bed scrubber recycle flow rate and maximum stack gas flow, thus establishing a *de facto* minimum liquid to gas ratio. Packed bed scrubber recycle flow and stack gas flow will both be monitored on a continuous basis. Based on successful demonstration of HCl and Cl₂ control during the performance test, the minimum packed bed scrubber recycle liquid flow rate limit will be based

on the mean of the hourly rolling average values demonstrated during each run of the performance test.

7.2.1.7 Minimum Scrubber Blowdown Flow Rate

The performance test will demonstrate minimum scrubber blowdown flow rate, in order to demonstrate worst case conditions for solids buildup in the scrubbing system. In order to conserve water, WCAI recycles most of the liquid from the air pollution control system. However, in order to prevent the buildup of dissolved solids in the recycled water, a certain amount of the water must be purged (or blown down) from the system. As water is purged from the system, fresh makeup water is added. The minimum scrubber blowdown flow rate limit will be based on the mean of the hourly rolling average values demonstrated during each run of the performance test.

7.2.1.8 Minimum WESP Secondary Voltage

Although the HWC MACT regulations do not require any indicator of performance in an electrically enhanced emissions control device, WCAI believes that it is appropriate to establish a performance indicator. Accordingly, WESP secondary voltage (expressed as KVDC) will be used as the indicator of continuing WESP performance. The minimum value will be established from the mean of the minimum hourly rolling average secondary voltage values demonstrated during each run of the performance test. The secondary voltage value will be based on an hourly rolling average.

7.2.1.9 Maximum Combustion Gas Velocity (Stack Gas Flow Rate)

The stack gas flow rate (expressed as actual cubic feet per minute) will be used as the indicator of combustion gas velocity. The maximum stack gas flow rate will be established from the mean of the maximum hourly rolling average stack gas flow rates demonstrated during each run of the performance test. The combustion gas velocity is proposed as an hourly rolling average limit to dampen normal variations in flow.

7.2.2 Group A2 Parameters

Group A2 parameter limits are interlocked with the automatic waste feed cutoff system, but are not based on the results of the performance test. The following parameters are proposed as Group A2 parameters for the RF system.

7.2.2.1 Maximum Stack Gas CO Concentration

The maximum hourly rolling average stack gas CO concentration will be maintained at or below 100 ppmv corrected to 7% oxygen (dry basis) during the PDT. WCAI expects to obtain a permit limit specifying a maximum allowable stack gas carbon monoxide concentration of 100 ppmv hourly rolling average corrected to 7% oxygen, dry basis, based on the regulatory limit.

7.2.3 Group B Parameters

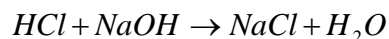
7.2.3.1 Maximum Chlorine/Chloride Feed Rate

WCAI will feed chlorine/chloride at the maximum anticipated rate during the performance test. Assuming that the stack gas HCl/Cl₂ concentration meets the applicable standard, the final limit for total chlorine/chloride feed rate should be expressed as a 12-hour average based on the mean of the chlorine/chloride feed rate values demonstrated during each run of the test.

During routine operations, WCAI has found the chlorine feed rate to be somewhat variable based on observed caustic use in the scrubbing system. Further, accurate chlorine feed concentration data on spent activated carbon is difficult to obtain and relate to a "real time" measure of chlorine feed rate. WCAI therefore proposes to demonstrate continuing compliance with the chlorine feed rate limit by monitoring the change in total dissolved solids in the RF scrubber blowdown.

WCAI has performed an evaluation of the total amount of chlorine feed as it relates to TDS in the scrubber blowdown. This evaluation demonstrates that compliance with the facility's existing wastewater discharge limit of delta 1400 mg/L for the rise in total dissolved solids (TDS) can be used to ensure compliance with the facility's proposed 12-hour rolling average limit for the feed rate of total chlorine and chloride. Attachment F contains a summary of the evaluation of chlorine feed rate versus TDS, and presents calculations to support the conclusions. The following presents a brief summary of the evaluation.

Sodium hydroxide (NaOH) is added to the recirculated scrubber solutions for the quench/venturi and packed bed scrubbers to neutralize and control the emissions of hydrogen chloride (HCl) from the carbon reactivation furnace. HCl is readily dissolved in water, where chloride ions react with sodium ions to form salt.



The NaCl formed as a result of this reaction manifests itself as dissolved solids in the scrubber liquid. The pH of the recirculated scrubber solutions for the quench/venturi and packed bed scrubbers are controlled to make sure a sufficient amount of sodium hydroxide is continuously added to the scrubber water for reaction with the hydrogen chloride.

The amount of sodium chloride formed is dependent upon the chloride content and the feed rate of the spent activated carbon. At a chloride feed rate equal to that proposed as the permit limit (and as will be demonstrated during the test) the increase in TDS in WCAI's water discharge is slightly in excess of its waste water discharge limit. Thus, compliance with the waste water TDS discharge restrictions ensures compliance with the proposed chlorine feed rate limit.

7.2.3.2 Maximum Mercury Feed Rate

Due to the low amounts of mercury expected in the spent activated carbon, WCAI will comply with the mercury standard by calculating and complying with a 12-hour rolling average Maximum Theoretical Emission Concentration (MTEC), conservatively assuming no mercury removal across the APC system. The MTEC is complied with as a maximum mercury feed rate limit. This limit will be calculated from the performance test data by using the stack gas flow rate and oxygen concentration, and the maximum allowable stack gas mercury concentration based on the HWC MACT regulations. The feed rate limit is determined assuming that all mercury is emitted.

7.2.3.3 Maximum Semivolatile Metals Feed Rate

WCAI will determine the feed rate and emission rate of the semivolatile metals cadmium and lead during the performance test. A maximum semivolatile metal feed rate will be established for the total combined cadmium and lead feed rates as a 12-hour rolling average. This limit will be based on the mean of the average semivolatile metals feed rates, and the emission rate of semivolatile metals, demonstrated during each run. The total semivolatile metal feed rate during the test will be supplemented by spiking of lead as needed. The test results will be extrapolated upwards to the allowable HWC MACT limit, using the equations presented in Section 7.2.5 below.

7.2.3.4 Maximum Low Volatility Metals Feed Rate

WCAI will determine the feed rate and emission rate of the low volatility metals arsenic, beryllium, and chromium during the performance test. A maximum low volatility metal feed rate will be established for the total combined arsenic, beryllium, and chromium feed rates as a 12-hour rolling average. This limit will be based on the mean of the average low volatility metals feed rates, and the emission rate of low volatility metals, demonstrated during each run. The total low volatility metal feed rate during the test will be supplemented by spiking of chromium as needed. The test results will be extrapolated upwards to the allowable HWC MACT limit, using the equations presented in Section 7.2.5 below.

7.2.4 Group C Parameters

Group C parameters are determined from information other than the test results. These parameters and how the limits are to be established are described below.

7.2.4.1 Minimum Packed Bed Scrubber Pressure Differential

A minimum pressure drop across the packed bed scrubber will be established as a limit, based on past operating experience.

7.2.5 Extrapolation of Metals Feed Rate Limits

As allowed by the HWC MACT regulations, WCAI wishes to feed representative metals to the RF system during the PDT and to establish feed rate limits by extrapolating upward from the test results. WCAI proposes to feed lead and chromium at only a fraction of the maximum desired permit feed rates during the test. These metals are representative of the semivolatile and low volatility metal groups, respectively. Since these metals are representative of the metal volatility groups, the test data can be used to calculate a system removal efficiency (SRE) for each of these metals which can then be applied to their respective metal volatility group. System removal efficiency will be calculated using the following equation:

$$SRE = \left[1 - \frac{\dot{m}_{i,out}}{\dot{m}_{i,in}} \right] \times 100\%$$

where:

$\dot{m}_{i,in}$ = mass feed rate of metal i.

$\dot{m}_{i,out}$ = mass emission rate of metal i.

The calculated system removal efficiency for each metal volatility class can be used to establish feed rate limits for each group using the following equation:

$$\dot{m}_{i,in,max} = \frac{\dot{m}_{i,out,MACT}}{\left(1 - \frac{SRE}{100}\right)}$$

where:

$\dot{m}_{i,in,max}$ = maximum allowable feed rate of metal i.

$\dot{m}_{i,out,MACT}$ = mass emission rate of metal i at MACT regulatory limit.

If necessary, the test data can also be used to establish appropriate risk-based feed rate limits for other individual metals of concern based on their volatility and risk-based emission limits.

Metals will be added to the spent activated carbon feed as a stable dispersion of finely divided metal-containing solids. Lead will be fed in its oxide form. Chromium will be fed in the form of strontium chromate, which is a solid hexavalent chromium compound. Metals or their oxides are the most common form of the metals expected to be fed to the process, and were thus chosen as the feed form for the test. Chromium is being fed in a hexavalent form to produce a worst case feed for the risk assessment testing.

The purpose of feeding only two metals, and for upward extrapolation from the test results, is to minimize the number and quantity of metals added to the RF solely for the purpose of demonstrating the maximum desired feed rate. WCAI has examined its spent activated carbon characterization data (shown in Table 2-1) and has determined a desired feed rate limit for each metal volatility group. The proposed metal feed rates for the PDT are approximately one third to one half of these limits. This level of spiking was chosen for the following reasons:

- Spiking at less than the maximum desired feed rate decreases the emissions associated solely with the PDT.
- Spiking at approximately one third to one half of the desired feed rate minimizes the degree to which test results will need to be extrapolated, thus eliminating inaccuracies which might be associated with a larger degree of extrapolation.

- The anticipated emissions resulting from the planned spiking rates should be detectable using the specified sampling and analytical methods so that an accurate system removal efficiency for both metals can be determined. (See calculations in Attachment D).

8.0 TEST REPORT

The final Performance Demonstration Test report will be submitted to EPA within 90 days after completion of the test. The final report will be a comprehensive test report that contains a discussion of the test objectives; sampling, analysis, and QA/QC activities performed; summaries of process operating conditions; the results of the test determinations; and proposed permit conditions. The planned outline of the report is shown in Figure 8-1.

Specific determinations to be made based on the test results include, but are not limited to the following:

8.1 REGULATORY COMPLIANCE DETERMINATIONS

8.1.1 Destruction and Removal Efficiency for the Designated POHCs.

The DRE determination will be made using the following equation:

$$DRE = \left[1 - \frac{W_{out}}{W_{in}} \right] \times 100$$

where:

DRE = Destruction and Removal Efficiency (%)

W_{in} = Mass feed rate of POHC

W_{out} = Stack gas mass emission rate of POHC.

8.1.2 Particulate Matter Emission Concentration

The particulate matter emission concentration will be determined in terms of milligrams of filterable particulate matter per dry standard cubic meter of stack gas, corrected to 7 percent oxygen by volume, dry basis.

8.1.3 Metal Emission Concentrations

The stack gas emission concentration of mercury, semivolatile metals (total combined cadmium and lead), and low volatility metals (total combined arsenic, beryllium, and chromium) will be

determined in terms of micrograms of metal per dry standard cubic meter of stack gas, corrected to 7 percent oxygen by volume, dry basis.

8.1.4 System Removal Efficiency for Specific Metal Groups

In addition to determining the metal emission concentrations, for purposes of extrapolating metal emission rates and feed rates upwards to determine the appropriate metal feed rate limits, WCAI will determine the System Removal Efficiency (SRE) of Semivolatile Metals (lead and cadmium combined) and of Low Volatility Metals (arsenic, beryllium, and chromium combined). The formula for SRE is analogous to that used for DRE:

$$SRE = \left[1 - \frac{M_{out}}{M_{in}} \right] \times 100$$

where:

SRE = System Removal Efficiency (%)

M_{in} = Mass feed rate of metal (or metal group)

M_{out} = Stack gas mass emission rate of metal (or metal group).

8.1.5 Hydrogen Chloride and Chlorine Emission Concentration

The total combined stack gas emission concentration of hydrogen chloride and chlorine will be determined in terms of parts per million hydrogen chloride equivalents by volume, corrected to 7 percent oxygen by volume, dry basis.

8.1.6 Polychlorinated Dibenzo-p-Dioxin and Polychlorinated Dibenzofuran Emission Concentration

The stack gas emission concentration of PCDD/PCDF will be determined in terms of nanograms of 2,3,7,8-TCDD toxic equivalents (TEQ) per dry standard cubic meter of stack gas, corrected to 7 percent oxygen by volume, dry basis.

8.1.7 Carbon Monoxide Emission Concentration

The concentration of carbon monoxide in the stack gas will be continuously monitored and reported in terms of parts per million by volume, corrected to 7 percent oxygen by volume, dry basis.

8.1.8 Total Hydrocarbon Emission Concentration

During each test run where DRE is being determined, the stack gas total hydrocarbon emission concentration will also be determined in terms of parts per million propane by volume, corrected to 7 percent oxygen by volume, dry basis.

8.2 OTHER STACK GAS EMISSION DETERMINATIONS

In addition to the regulatory compliance emission determinations, the following stack gas emission determinations will be made:

Stack Gas Parameter	Units
Stack gas flow rate	dscfm, dscm/min
Stack gas velocity	ft/s, m/s
Stack gas temperature	°F, °C
Stack gas moisture content	vol%
Stack gas oxygen concentration	vol%, dry
Stack gas carbon dioxide concentration	vol%, dry
Stack gas dry molecular weight	lb/lb-mol
Particulate matter emission rate	lb/h, g/s
Hydrogen chloride emission rate	lb/h, g/s
Chlorine emission rate	lb/h, g/s
Metals emission rates (Al, Sb, As, Ba, Be, Cd, Cr (total), Cr (VI), Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)	lb/h, g/s
PCDD/PCDF emission rate (each 2,3,7,8-substituted congener, and total TEQ)	lb/h, g/s
Speciated volatile organic compound emission rate (each target analyte plus each TIC)	lb/h, g/s
Speciated semivolatile organic compound emission rate (each target analyte plus each TIC)	lb/h, g/s
Total volatile organics emission rate (subset of TOE)	lb/h, g/s
Total semivolatile organics emission rate (subset of TOE)	lb/h, g/s
Total nonvolatile organics emission rate (subset of TOE)	lb/h, g/s
PCB emission rate (each target analyte)	lb/h, g/s
Organochlorine pesticides emission rate (each target analyte)	lb/h, g/s
PAH emission rate (each target analyte)	lb/h, g/s
Particle size distribution	Mass fraction of various particle size ranges

8.3 FEED AND EFFLUENT DETERMINATIONS

The following feed material and effluent determination will be made:

Feed Parameter	Units
Spent carbon feed rate	lb/h, kg/h
Spent carbon chlorine/chloride concentration	mg/kg
Spent carbon metals concentration (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)	mg/kg
Spent carbon POHC concentration	mg/kg
Spent carbon volatile organic concentration	ug/kg
Spent carbon semivolatile organic concentration	ug/kg
Makeup water metals concentration (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)	mg/l
Makeup water volatile organic concentration	ug/l
Makeup water semivolatile organic concentration	ug/l
Caustic solution metals concentration (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)	mg/l
Caustic solution volatile organic concentration	ug/l
Caustic solution semivolatile organic concentration	ug/l
Total feed rate of chlorine/chloride	lb/h
Total feed rate of metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)	lb/h
Total feed rate of each POHC	lb/h

Effluent Parameter	Units
Scrubber blowdown volatile organic concentration	ug/kg
Scrubber blowdown semivolatile organic concentration	ug/kg
Scrubber blowdown metals concentration (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)	mg/l
POTW discharge volatile organic concentration	ug/l
POTW discharge semivolatile organic concentration	ug/l
POTW discharge metals concentration (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)	mg/l

8.4 PROCESS DATA

The following process data points will be continuously monitored and recorded. One minute values for each parameter listed will be presented in an appendix to the report, while the average, minimum, and maximum values recorded during each run will be summarized within the body of the report.

Process Parameter	Units
Spent carbon feed rate	lb/h
Afterburner temperature	°F
Venturi scrubber pressure differential	in. w.c.
Quench/Venturi scrubber liquid flow rate	gpm
Packed bed scrubber pH	pH
Packed bed scrubber liquid flow rate	gpm
Packed bed scrubber pressure differential	in. w.c.

Process Parameter	Units
Scrubber blowdown flow rate	gpm
WESP secondary voltage	kVDC
Stack gas flow rate	acfm

8.5 REPORTING OF INTERMEDIATE RESULTS

EPA has requested that WCAI report certain intermediate data to EPA during the test and prior to submittal of the final test report. WCAI will comply with those requests as follows.

8.5.1 Daily Process Data

WCAI will provide EPA's on-site observer with either computer disks or hard copy printouts (at the option of the EPA observer) representing the one-minute updated process operating data for the parameters described in Table 7-1. These data, from the previous day's test activities, will be provided at the beginning of the following day. Prior to the first day of testing, WCAI will provide the previous day's data for these same monitors, as well as their most recent calibration results.

8.5.2 Unfavorable Performance Demonstration Test Results

If WCAI determines, during the course of compiling the Performance Demonstration Test data, that any of the test emission targets were not met, or if other unfavorable results have occurred, EPA will be notified as soon as practical following WCAI's confirmation of such an occurrence. WCAI and EPA will work together to develop appropriate corrective actions to resolve any such situation.

8.5.3 Modification of Planned Performance Test Operating Conditions

Should preliminary testing of the RF system, or other information lead WCAI to propose a change of target process operating conditions or to modify the test protocol after approval of the test plan, WCAI will implement such changes through the use of a Corrective Action Request (CAR) as described in Section 14.2 of the QAPP (Attachment C). Such CAR will require approval of WCAI, the test manager, and EPA.

9.0 DATA IN LIEU OF TESTING

WCAI has conducted a number of previous tests on the carbon reactivation furnace and is confident in its ability to meet all emission requirements, however most of those tests were not conducted under conditions which would support all of the desired permit conditions as described in this test plan. Thus, data from those previous tests is not being submitted in lieu of the currently planned compliance testing. WCAI does however have particle size distribution data collected in November 2001, which it believes is appropriate for use during the risk assessment, and has included in Attachment G.

WCAI believes that it is appropriate to utilize the existing particle size distribution (PSD) data in lieu of gathering the same data during the Performance Demonstration Test, since PSD data is used for risk assessment modeling of long-term impacts from the facility. This data set was generated under typical operating conditions without spiking of metals or ash, thus the size distribution data in Attachment G is representative of normal operations. It is possible that spiking of finely divided metals during the Performance Demonstration Test could alter the size distribution of the emitted particles compared to normal operations. Further, use of the current PSD data set will also eliminate one sampling train during the Performance Demonstration Test, which is already extremely complex.

ATTACHMENT A
SAMPLING PROCEDURES

Table A-1. Spent Activated Carbon Sampling Procedure

Sample name:	Spent Activated Carbon
Sampler:	Process sampling team
Locations:	Feed Conveyor
Equipment:	Amber glass bottle (4L wide mouth) with Teflon-lined lid Amber glass bottles (1L wide mouth) with Teflon-lined lid Amber glass jar (250 mL wide mouth) with Teflon-lined lid Stainless steel scoop Gloves, eye protection, hard hat
Frequency:	15-minute intervals during each test run
Procedure Summary:	<p>The steel scoop is used to collect approximately equal size samples of the material from the feed conveyor at each time interval and to transfer to the 4L glass bottle.</p> <p>The time each grab sample is taken is recorded on the sample collection sheet.</p> <p>At the end of each run, fill four 1L bottles and one 250 mL jar from the 4L bottle as follows:</p> <ul style="list-style-type: none">1L – Semivolatile organics1L – Properties1L – Metals1L – Archive250 mL – Volatile organics. <p>A sample number and label with date and sample name is attached to each bottle.</p> <p>Sample coordinator accepts custody of samples and records numbers and collection data in field logbook.</p> <p>Samples are placed on ice in shipping container that is stored in the sample holding area separate from the container supply area.</p>
Reference:	“ASTM E 300-92 (1996) - Sections 27-34, Standard Practice for Sampling Industrial Chemicals”, American Society for Testing and Materials, Annual Book of ASTM Standards, West Conshohocken, Pennsylvania.

Table A-2. Spiking Material Sampling Procedure

Sample name:	POHC spiking material Organic surrogate spiking material Metals spiking material
Sampler:	Process sampling team
Locations:	Sample tap on spiking system feed line
Equipment:	1-liter amber glass bottles with Teflon-lined lids 250-mL amber glass bottles with Teflon-lined lids 40-mL amber glass (VOA vials with plastic screw caps and Teflon septa Waste container Gloves, eye protection, respiratory protection if needed.
Frequency:	Once during test when used
Procedure Summary:	<p><u>Metals Spiking Material Sample Collection:</u> The sample tap is purged by allowing a small amount of liquid to flow into the waste container. A 1-liter glass bottle is filled from the sample tap. The time the sample is taken and the approximate volume of the sample are recorded on the sample collection sheet.</p> <p><u>POHC Spiking Material:</u> The sampling tap is purged by allowing a small amount of liquid to flow into the waste container. One pair of VOA vials is filled so there is no headspace when the caps are placed on the vials. One 250-mL glass bottle is then filled from the sample tap. Each pair of VOA vials and the sample bottles are labeled with the sample name, date, run number, and time. Each sample number and the time collected are recorded on the sample collection sheet.</p> <p><u>Organic Surrogate Sampling Material:</u> The sampling tap is purged by allowing a small amount of liquid to flow into the waste container. One pair of VOA vials is filled so there is no headspace when the caps are placed on the vials. One 250-mL glass bottle is then filled from the sample tap. Each pair of VOA vials and the sample bottles are labeled with the sample name, date, run number, and time. Each sample number and the time collected are recorded on the sample collection sheet.</p> <p><u>All samples:</u> The Sample Custodian accepts custody of all samples and records numbers and collection data in a field logbook. Samples are sealed and placed in a shipping container that is stored in the sample holding area separate from the container supply area. Samples for organic analyses are chilled to 4°C; all other samples do not require chilling.</p>
References:	“ASTM E 300-92 (1996) - Sections 11-26, American Society for Testing and Materials”, Annual Book of ASTM Standards, West Conshohocken, Pennsylvania.

Table A-3. Makeup Water Sampling Procedure

Sample name:	Makeup Water
Sampler:	Process sampling team
Locations:	Sample tap on supply line
Equipment:	Amber glass bottle (4 L) with Teflon-line lid Amber glass bottles (1-L) with Teflon-lined lids Amber glass VOA vials (40-mL) with plastic screw caps and Teflon septa
Frequency:	Once, at the beginning of the test.
Procedure Summary:	<p>The sample tap is purged by allowing a small amount of liquid to flow into the waste container. A grab sample is collected by filling a 4-liter glass bottle from the tap. Two VOA vials are then filled completely full (no headspace) from the sample tap.</p> <p>The sample in the 4-liter bottle is subsequently transferred to three 1-liter bottles, as follows::</p> <ul style="list-style-type: none">1L – Semivolatile organics1L – Metals1L – Archive. <p>The 4-liter bottle and any excess samples are discarded as appropriate for the material.</p> <p>Record the sampling time on sample collection sheet and approximate volume of final samples.</p> <p>The sample bottles and vials are labeled with sample number, date, sample name, and test-run number.</p> <p>Sample coordinator accepts custody of samples and records numbers and collection data in field logbook.</p> <p>Samples are placed on ice in shipping container that is stored in the sample holding area separate from the container supply area.</p>
Reference:	“ASTM E 300-92 (1996) - Sections 11-26, Standard Practice for Sampling Industrial Chemicals”, American Society for Testing and Materials, Annual Book of ASTM Standards, West Conshohocken, Pennsylvania.

Table A-4. Caustic Sampling Procedure

Sample name:	Caustic Solution (<i>Purchased product; not a waste feed stream</i>)
Sampler:	Process sampling team
Locations:	Sample tap on caustic supply line
Equipment:	4-liter glass bottle 1-liter amber glass bottles with Teflon-lined lids Amber glass VOA vials (40-mL) with plastic screw caps and Teflon septa Waste liquid container Gloves, eye protection, splash protection
Frequency:	Once at the beginning of the test.
Procedure Summary:	<p>The sample tap is purged by allowing a small amount of liquid to flow into the waste container. A grab sample is collected by filling a 4-liter glass bottle from the tap. Two VOA vials are then filled completely full (no headspace) from the sample tap.</p> <p>The caustic sample in the 4-liter bottle is subsequently transferred to three 1-liter bottles, as follows:</p> <ul style="list-style-type: none">1L – Semivolatile organics1L – Metals1L – Archive. <p>The 4-liter bottle and any excess samples are discarded as appropriate for the material.</p> <p>Record the sampling time on sample collection sheet and approximate volume of final samples.</p> <p>The sample bottles and vials are labeled with sample number, date, sample name, and test-run number.</p> <p>The Sample Custodian accepts custody of samples and records numbers and collection data in a field logbook.</p> <p>Samples are placed on ice in a shipping container that is stored in the sample holding area separate from the container supply area.</p>
Reference:	"ASTM E 300-92 (1996) - Sections 11-26, Standard Practice for Sampling Industrial Chemicals", American Society for Testing and Materials, Annual Book of ASTM Standards, West Conshohocken, Pennsylvania.

Table A-5. Scrubber Blowdown Water and POTW Discharge Water Sampling Procedure

Sample name:	Scrubber blowdown POTW discharge
Sampler:	Process sampling team
Locations:	Sample tap on pipeline
Equipment:	Glass graduated cylinder 4-liter glass bottle 1-liter amber glass bottles with Teflon-lined lids Amber glass VOA vials (40-mL) with plastic screw caps and Teflon septa Waste container Gloves, eye protection
Frequency:	30-minute intervals during each test run
Procedure Summary:	<p>The sample tap is purged by allowing a small amount of liquid to flow into the waste container. The graduated cylinder is rinsed with liquid and the rinse is discarded to the container. Approximately 200-mL of sample is collected in the graduated cylinder and transferred to a 4-liter glass bottle at each time interval.</p> <p>Two VOA vials are filled (no headspace) from the sample tap at each time interval.</p> <p>The time each grab and VOA vial sample is taken is recorded on the sample collection sheet. The approximate total volume of the grab samples is also recorded.</p> <p>At the end of the test run, the grab samples are mixed in the 4-liter bottle to form a composite sample. The composite sample is then transferred to 1-liter bottles as follows:</p> <ul style="list-style-type: none">1 - Total metals1 - Semivolatiles1 - Archive <p>Each sample bottle and vial is labeled with sample number, date, sample name, and test-run number.</p> <p>The 4-liter bottle and any excess sample are discarded to appropriate waste containers.</p> <p>The Sample Custodian accepts custody of samples and records sample numbers and collection data in a field logbook.</p> <p>Samples are placed on ice in a shipping container that is stored in the sample holding area separate from the container supply area.</p>
Reference:	“ASTM E 300-92 (1996) - Sections 11-26, Standard Practice for Sampling Industrial Chemicals”, American Society for Testing and Materials”, Annual Book of ASTM Standards, West Conshohocken, Pennsylvania.

Table A-6. Method 0030 Stack Gas Volatile Organic Sampling Procedure

Sample name: Stack Gas Method 0030 (VOST)

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 0030 volatile organic sampling train (VOST) as shown in Figure 1 of this table; Tenax and Tenax/charcoal sorbent cartridges sealed in glass culture tubes with Teflon lined screw caps; amber glass VOA vials (40 ml) with plastic screw caps and Teflon septa; screw capped glass container.

Frequency: Continuous with replacement of sorbent cartridge pairs every 40 minutes for a total sampling time of at least 160 minutes. Four pairs of sorbent cartridges are sampled during each test run.

Procedure

Summary: Stack gases are sampled at a controlled rate of 1.0 liters/minute (L/min) to sample volatile organic compounds (VOCs) on sorbent resin and charcoal, and in stack gas condensate. The mass of each target VOC sampled on the sorbents and condensate is quantitatively determined by GC/MS analysis. (Note: This procedure is used to sample VOCs that have boiling points ranging from approximately 35°C to over 100°C.)

The sampling train includes a stainless steel probe with borosilicate or quartz glass liner, a heat-traced Teflon sample line (if used), an isolation valve, a water-cooled glass condenser, a Tenax cartridge, a condensate collection trap, a second water-cooled glass condenser, a Tenax/charcoal cartridge, and a silica gel cartridge. A rotameter, vacuum pump, and dry gas meter complete the train. A submersible pump is used to circulate ice bath water through the condensers.

The sampling train components and reagents are prepared according to the procedures specified in Method 0030. As described in the method, the train glassware is cleaned with nonionic detergent in an ultrasonic bath, rinsed with organic-free (HPLC) water, and dried in an oven. The sorbent material (Tenax resin and charcoal) is Soxhlet extracted, vacuum dried, loaded into clean glass tubes, and thermally conditioned with organic-free nitrogen. The tubes are sealed with clean metal caps. Each cartridge (glass tube, sorbent, glass wool, and end caps) is permanently marked with an identification number and direction of sample flow, and sealed in a glass culture tubes that contain clean charcoal. The cleanliness of the sorbent in the cartridge set is checked by analysis of at least one pair of the cartridges that have been spiked with benzene and toluene as described in the method. The dry gas meter is calibrated within 30 days before conducting the test program.

In the laboratory, the cartridges are stored in a cooler at or near 4 °C (39°F) in an area free from volatile organic material. During pretest preparation, one pair of cartridges is selected from the cartridge set and identified as a laboratory blank, which is retained at the laboratory for analysis with the field samples. For transfer to the test site, the cartridges are packed separate from other test supplies and placed on cold packs in insulated containers to keep their temperature at or near 4°C for the duration of the trip. The laboratory blank cartridges are stored in the same manner as the field cartridges. At the test site, the insulated containers are stored in an area as free as possible from volatile organic materials and the cartridges are kept cold in the insulated containers until needed for a test run.

Table A-6. Method 0030 Stack Gas Volatile Organic Sampling Procedure (Continued)

The VOST and a small insulated cooler containing 5 to 8 pairs of sorbent cartridges are taken to the sampling location, where the train is assembled in a manner that minimizes the potential for contamination from air or by direct contact. One pair of sorbent cartridges is installed in the train and the end caps are placed in a clean screw-capped glass container. No sealant grease is used in assembling the train.

A leak-check of the train assembly is performed by closing the isolation valve at the inlet to the first condenser, establishing ice bath water flow through the condensers, and using the pump to create a vacuum that is 25 cm (10 in.) Hg above normal operating pressure (total of approximately 38 cm Hg). The train leak rate must be less than 0.25 cm (0.1 in) Hg for 1 minute before starting the sampling run. The train is returned to atmospheric pressure by admitting ambient air through charcoal at the train inlet.

The VOST is operated according to the procedures provided in Method 0030. The train controls are set to maintain a temperature of 130°C to 150°C (266°F to 302°F) at the sample probe outlet and less than 20°C (68°F) at the inlet to the first cartridge. The stack gas is sampled at a rate 0.5 L/min for 40 minutes to sample a nominal volume of 20 L. The sampling train operating conditions are recorded as required on field data sheets.

At the end of the sampling run, the isolation valve is closed and the leak-check is repeated at the highest vacuum encountered during the run. The sorbent cartridges are removed from the train, the end caps replaced, labeled, and returned to the culture tubes, which are then placed on ice packs in a separate insulated container. A new pair of cartridges is then installed in the train and the procedures for leak checking, sampling, and recovery are repeated as described above. Four pairs of cartridges are sampled during each test run. (Note: Additional cartridge pairs are sampled if problems occur with the cartridges or the train fails the post-sampling run leak-check.)

At the end of each test run (4 cartridge pairs), any water collected in the condensate trap is transferred into a VOA vial(s), the trap is rinsed with minimum amounts of HPLC water, and the rinse is added to the vial. If there is not enough condensate to fill a VOA vial, organic-free water is added to eliminate headspace. The vials are labeled with sample name, date, and test run number. (Note: For high moisture content gases, the condensate may be recovered after collection of each cartridge pair.)

Field blank samples are collected once during each test run. The end caps are removed from a new pair of cartridges and they are allowed to remain open in the sampling area for approximately 10 minutes to simulate handling of the test cartridges.

The VOST operator delivers the test cartridges, condensate samples (if any), and the field blank cartridges for each test run to the Sample Coordinator, who makes the appropriate notations in the field log book, and packs the samples for shipment to the laboratory. For each shipment of cartridges during the test program, the Sample Coordinator includes one pair of new cartridges as trip blank samples.

Samples are stored at or near 4°C in shipping packages, which are stored in an area away from other samples that may have high concentrations of VOCs. If shipped by truck, the samples are stored away from other chemicals or from where automotive exhaust fumes could become concentrated.

Reference: "Method 0030 - Volatile Organic Sampling Train", Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.

Table A-6. Method 0030 Stack Gas Volatile Organic Sampling Procedure (Continued)

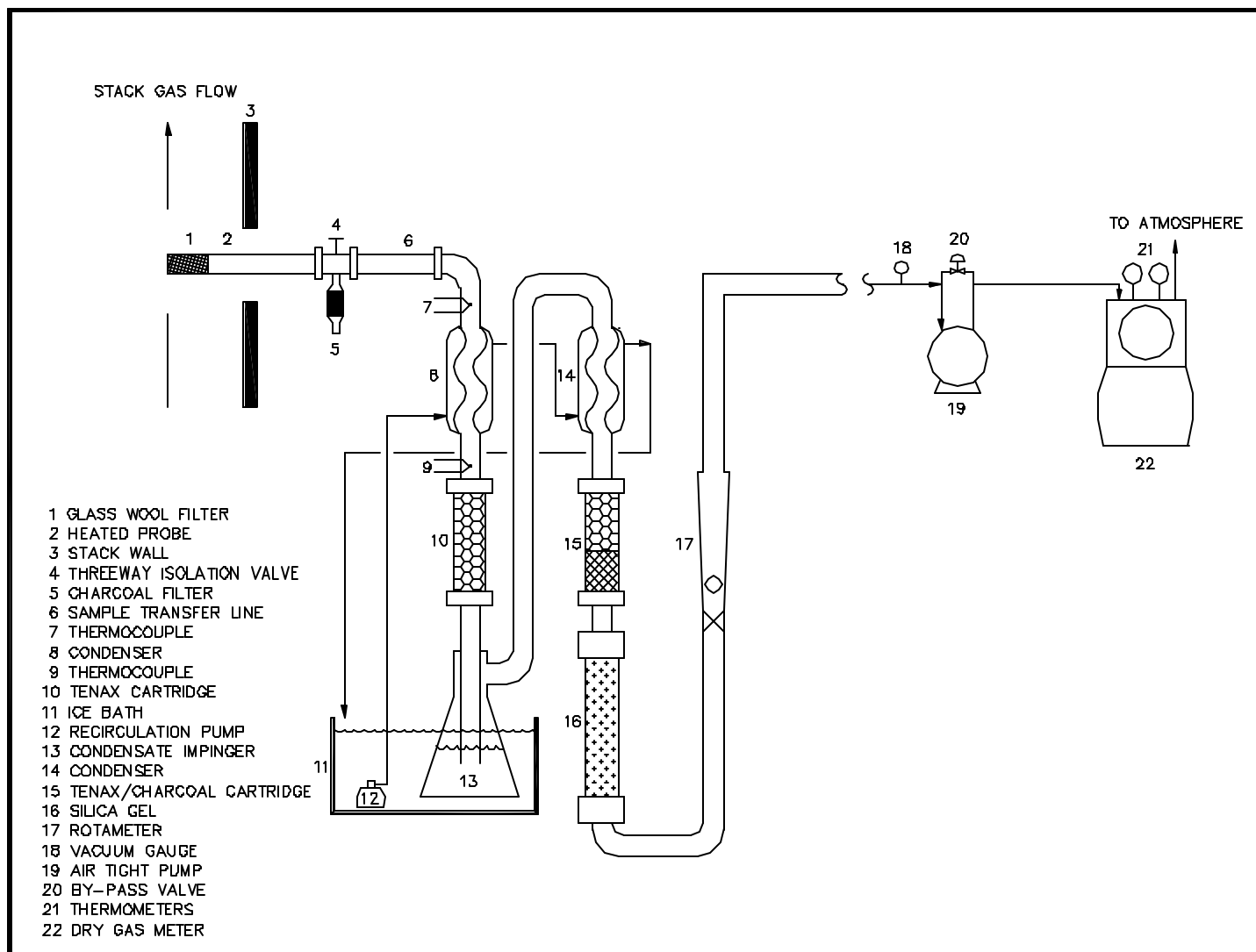


Figure 1. Method 0030 Sample Train for Volatile Organics

Table A-6. Method 0030 Stack Gas Volatile Organic Sampling Procedure (Continued)

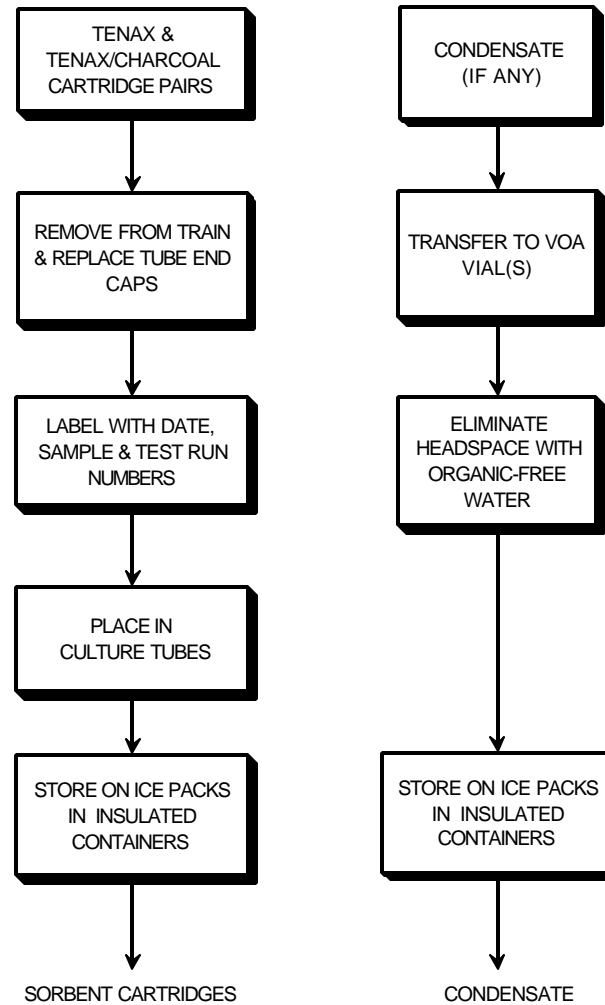


Figure 2. Method 0030 Volatile Organic Sample Recovery

Table A-7. Method 26A Stack Gas Particulate Matter, Hydrogen Halide, and Halogen Sampling Procedure (Isokinetic)

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:	<p>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.</p> <p>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.</p> <p>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 ($\geq 50^{\circ}\text{C}$) 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.</p> <p>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.</p> <p>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.</p>

Table A-7. Method 26A Stack Gas Particulate Matter, Hydrogen Halide, and Halogen Sampling Procedure (Isokinetic) (Continued)

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.

Table A-7. Method 26A Stack Gas Particulate Matter, Hydrogen Halide, and Halogen Sampling Procedure (Isokinetic) (Continued)

- 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.

Table A-7. Method 26A Stack Gas Particulate Matter, Hydrogen Halide, and Halogen Sampling Procedure (Isokinetic) (Continued)

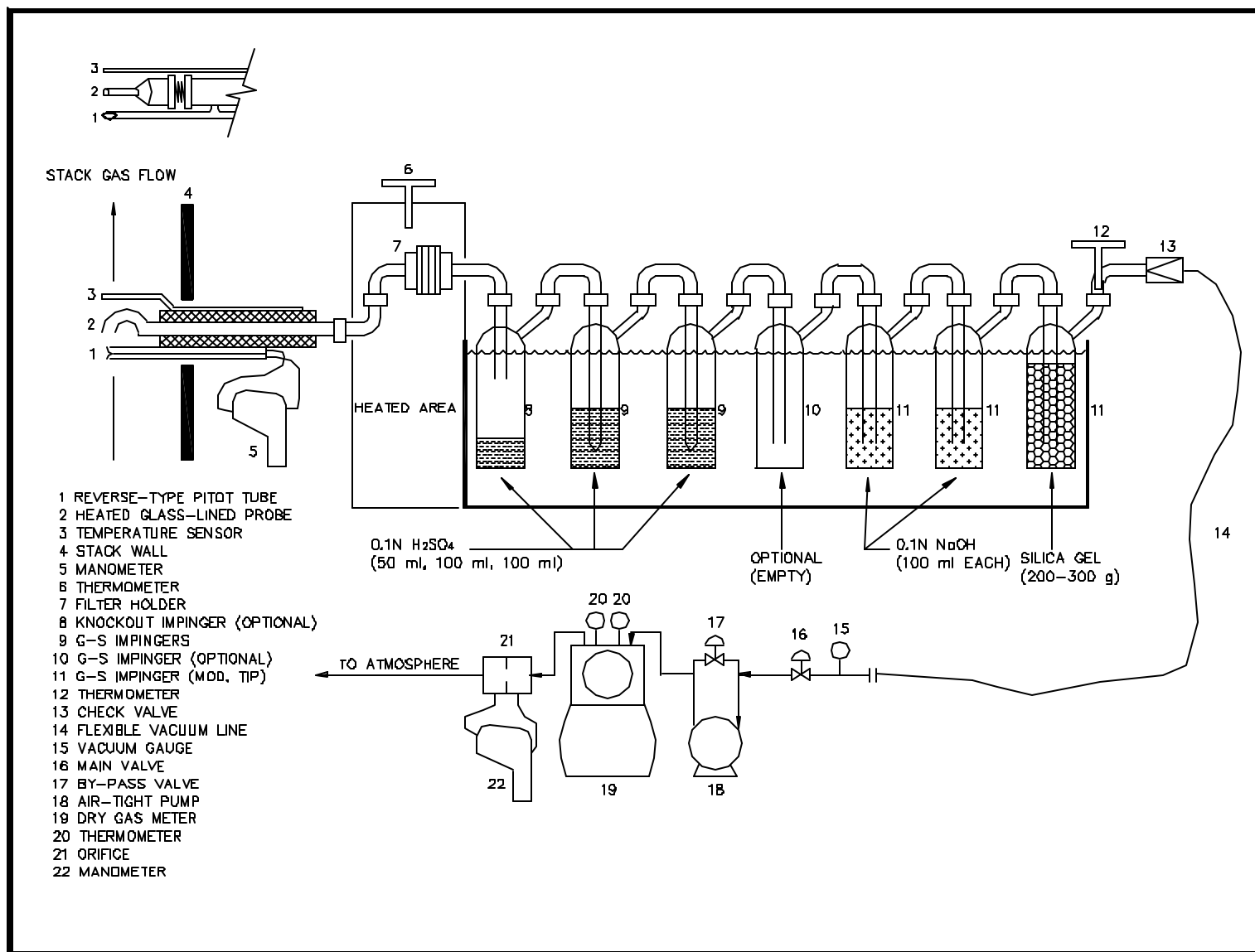


Figure 1. Method 26A Sample Train for Particulate Matter, Hydrogen Halide, and Halogen

Table A-7. Method 26A Stack Gas Particulate Matter, Hydrogen Halide, and Halogen Sampling Procedure (Isokinetic) (Continued)

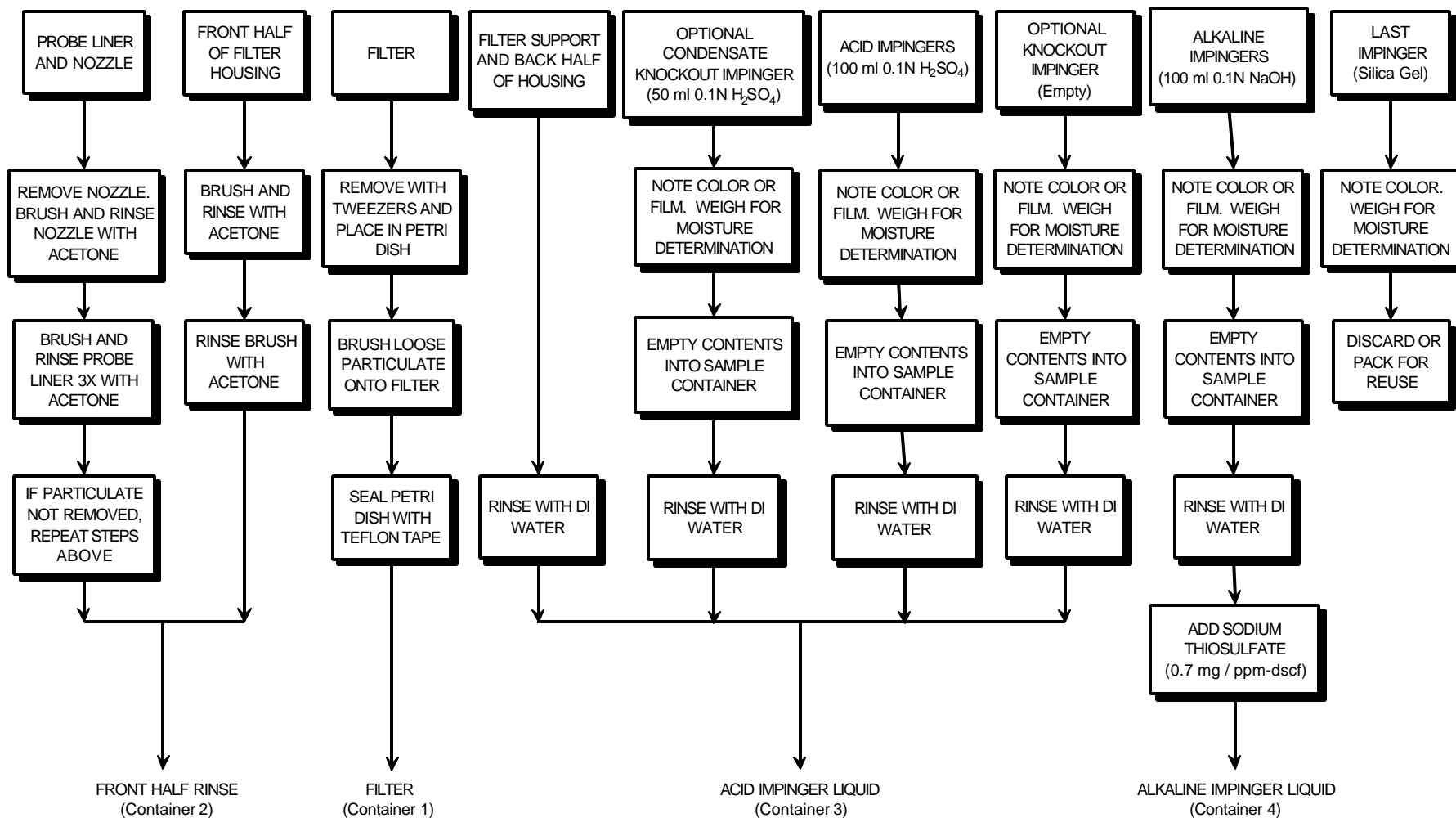


Figure 2. Method 26A Particulate Matter, Hydrogen Halide, and Halogen Sample Recovery

Table A-8. Method 29 Stack Gas Multiple Metals Sampling Procedure
(Includes Mercury, No Particulate)

Sample name:	Stack Gas Method 29 (MM, 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 of three test runs.
Procedure Summary:	<p>Gases are sampled isokinetically to collect the metals Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Mn, Ni, Se, Ag, Tl, V, and Zn on a particulate filter and in absorbing solutions. The filter and absorbing solutions are recovered for subsequent metals analyses by inductively coupled plasma emission spectroscopy (ICP) and/or graphite furnace AAS (GFAAS), and cold vapor AAS (CVASS). [Note: This procedure does <u>not</u> include collection of particulate matter (PM) samples for determination of PM concentration.]</p> <p>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, an empty impinger, two acidified potassium permanganate 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.</p> <p>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.</p> <p>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% HNO₃/10% H₂O₂ solution. The carryover impinger is empty and the next two impingers each contain 100 ml of 4% KmnO₄/10% H₂SO₄ solution. The last impinger contains 200 to 300 g of indicating silica gel.</p> <p>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.</p>

Table A-8. Method 29 Stack Gas Multiple Metals Sampling Procedure
(Includes Mercury, No Particulate) (Continued)

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 $\pm 10\%$ 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 a 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 $\text{HNO}_3/\text{H}_2\text{O}_2$ 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 $\text{HNO}_3/\text{H}_2\text{O}_2$ impingers, and all connecting glassware are rinsed with exactly 100 ml of 0.1N nitric acid, and the rinses are added to Container 4.
- Carryover impinger – The liquid contents of the initially empty impinger is measured to the nearest 0.5 ml and placed into a separate sample bottle (Container 5A). The impinger is then rinsed with exactly 100 ml of 0.10N nitric acid solution and the rinse is added to Container 5A.
- Acidified Permanganate Impingers -- The liquid contents of the acidified permanganate impingers are measured to the nearest 0.5 ml and placed into a separate sample bottle (Container 5B). These impingers and any connecting glassware are then rinsed a minimum of three times using a total of exactly 100 ml of fresh acidified potassium permanganate solution, and the rinses are added to Container 5B, being careful to also transfer any loose precipitated materials into the container. Triple rinsing of the acidified permanganate impingers is then repeated using a total of exactly 100 ml of water. The water rinses are also placed into Container 5B. If visible deposits remain in the impingers following the water rinses, they are rinsed with 25 ml of 8N hydrochloric acid, and the rinse is placed into a separate container (Container 5C) which contains 200 ml of water.

Table A-8. Method 29 Stack Gas Multiple Metals Sampling Procedure
(Includes Mercury, No Particulate) (Continued)

- 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 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; 200 ml of the acidified hydrogen peroxide solution, and 100 ml of the acidified potassium permanganate solution. If the acidified potassium permanganate impingers from any run are rinsed with HCl, then a 25 ml blank sample of the 8N HCl solution is also collected and added to 200 ml of water in a separate sample bottle.

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.
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Table A-8. Method 29 Stack Gas Multiple Metals Sampling Procedure (Includes Mercury, No Particulate) (Continued)

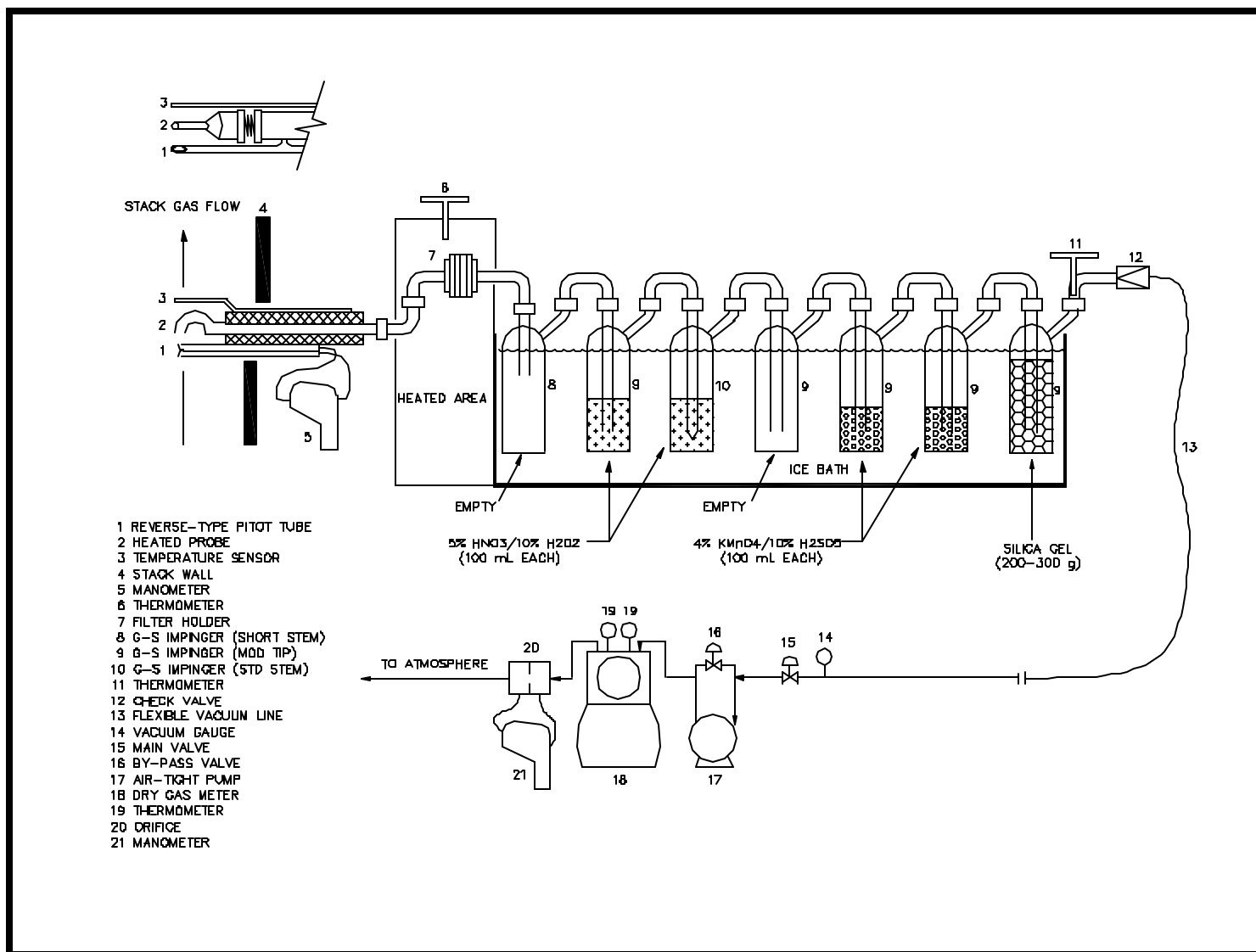


Figure 1. Method 29 Sample Train for Multiple Metals (Including Hg) (No Particulate)

Table A-8. Method 29 Stack Gas Multiple Metals Sampling Procedure (Includes Mercury, No Particulate) (Continued)

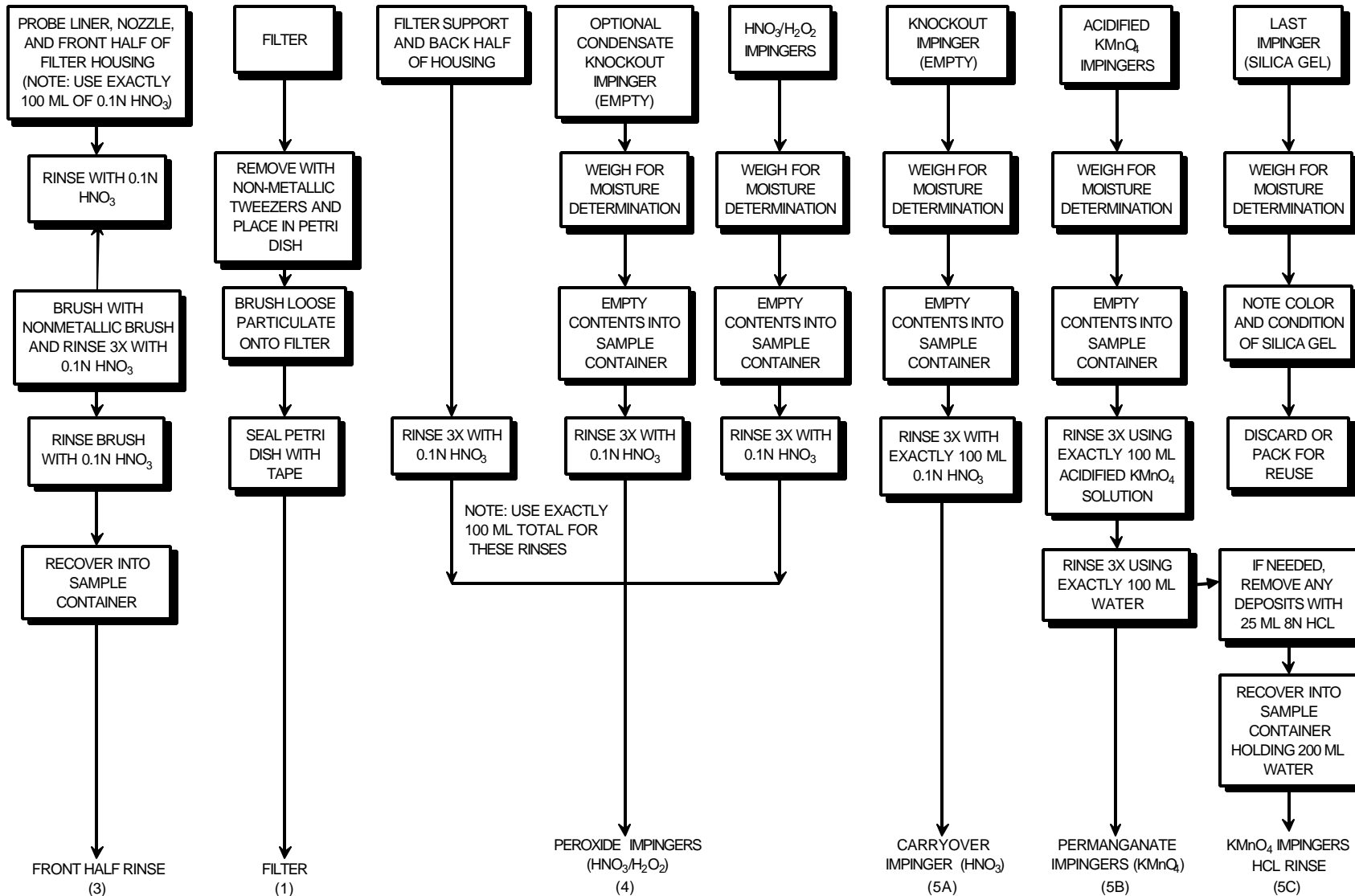


Figure 2. Method 29 Multiple Metals (Including Hg) (No Particulate) Sample Recovery

Table A-9. Method 0061 Stack Gas Hexavalent Chromium Sampling Procedure

Sample name:	Stack Gas Method 0061 (Cr ⁺⁶)
Sampler:	Stack sampling team
Locations:	Exhaust stack
Equipment:	Method 0061 hexavalent chromium sampling train as shown in Figure 1 of this table, cleaned polyethylene sample bottles with leak-free screw caps, graduated cylinder, balance, nitrogen cylinder and purge line, and sample filtration apparatus.
Frequency:	Continuous for a minimum of 120 minutes during each test run.
Procedure Summary:	Stack gases are sampled isokinetically to collect hexavalent chromium in an absorbing solution. The hexavalent chromium content of the solution sample is quantitatively determined by ion chromatography employing a post-column reactor (IC/PCR).

The Method 0061 sampling train consists of a glass or Teflon probe nozzle, a Teflon sample line, a Teflon aspirator (or peristaltic pump), four Teflon impingers, and a glass impinger. The first three impingers contain potassium hydroxide absorbing solution, the fourth is empty, and the fifth contains silica gel. All impingers are placed in an ice bath. An air-tight gas pump, dry gas meter, and a gas flow manometer complete the train.

During operation, the gas pump draws the stack gas sample through the nozzle, probe, and the chilled impingers, which serve to remove moisture from the gas and to capture hexavalent chromium. The aspirator (or pump) circulates reagent solution from the first impinger and injects it into the sample line, which returns the solution to the impinger along with the gas sample. The absorbing solution is injected at or near the probe outlet in order to capture and stabilize the hexavalent chromium before it can convert (reduce) to the trivalent oxidation state as it travels through the sample line to the impingers. [Note: If the stack gas temperature is greater than 200°F, 3 to 4 feet of the sample and absorbent lines may be coiled into the ice bath in order to prevent boiling of the absorbing solution.]

All train components, reagents, and cleaning solutions are prepared according to the procedures specified in the reference method. 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 reagent 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 reagent 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. The first impinger contains 150 ml of a 0.1 M solution of potassium hydroxide. Impingers 2 and 3 each contain 75 ml of 0.1 M potassium hydroxide solution. Impinger 4 is initially empty and serves to collect any liquid carryover from upstream impingers. Impinger 5 contains 200 to 400 g of indicating silica gel weighed to the nearest 0.5 g. [Note: The absorbing solution strength in the first impinger may be increased to 0.5 M (or higher if necessary) to maintain a high pH (>8.5) in the solution throughout sampling.]

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

Table A-9. Method 0061 Stack Gas Hexavalent Chromium Sampling Procedure (Continued)

(cyclonic flow is checked only on the first day of testing). An Orsat analyzer or a continuous emission monitoring system (CEMS) is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to Method 3, 3A, or 3C procedures. The stack gas moisture content is determined according to Method 4.

The sample train is operated according to Method 5 procedures for pretest, port change, and post-test leak checks, isokinetic sampling, and data recording. The train leak rate must be less than or equal to 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 $\pm 10\%$ of 100%. The sample probe temperature is maintained less than 200°F in order to prevent boiling of the absorbing solution.

After sampling, the probe is removed from the stack and a post-test leak check is performed and the nozzle covered. After cooling, the probe nozzle is wiped to remove external particulate matter and capped. The sampling train assembly is disconnected from the umbilical cord and transported to the sample recovery area. The sample recovery and sample train cleanup procedures are summarized below.

- Impinger 1 pH Check - The pH of the reagent solution in the first impinger is determined using a pH indicator strip. If the solution pH is less than 8.5, the solution is discarded and the test is repeated with appropriate adjustment of the absorbing solution strength. If the solution pH is equal to or greater than 8.5, sample recovery proceeds as follows.
- Post-Test Nitrogen Purge - Nitrogen is bubbled through the impinger train at a rate of approximately 10 liters per minute for 30 minutes as a safeguard against the conversion of hexavalent chromium to the trivalent oxidation state.
- Impinger Solution - The liquid contents of the first four impingers are volumetrically measured to the nearest 1 mL or weighed to the nearest 1 g and placed into a polyethylene sample bottle (Container 1). The impingers, probe nozzle, absorbent circulation system, and connecting tubing are then rinsed four times with distilled, deionized water and the rinses are added to the Container 1.
- Silica Gel -- The silica gel contents of impinger 5 are weighed to the nearest 0.5 g.

Immediately following recovery, the impinger solution (Container 1) is filtered through a 0.45 micrometer acetate filter (or equal) to remove insoluble matter. The sample container is rinsed three times with distilled deionized (DI) water, and the rinses are passed through the filter. The filter and reservoir are then rinsed three times with DI water and these rinses are also passed through the filter and added to the sample. The final sample volume is measured to the nearest 1 mL, and returned to the original polyethylene sample bottle. After sample filtration, the filter and reservoir are rinsed once with 0.1 M nitric acid and once with DI water. These rinses are discarded.

A schematic diagram of the sample recovery and filtration procedures is provided in Figure 2.

Reagent blank samples are collected of the potassium hydroxide absorbing solution(s) used in the sample trains and the reagent water used in rinsing the sample train. The blank volume the absorbing solution is approximately 300 mL. The reagent water volume is equal to the volume of water used in rinsing the train.

Table A-9. Method 0061 Stack Gas Hexavalent Chromium Sampling Procedure (Continued)

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 0061, Determination of Hexavalent Chromium Emissions from Stationary Sources," SW-846, Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates.

"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, and 5: Appendix A, Test Methods, New Source Performance Standards, 40 CFR 60.

Table A-9. Method 0061 Stack Gas Hexavalent Chromium Sampling Procedure (Continued)

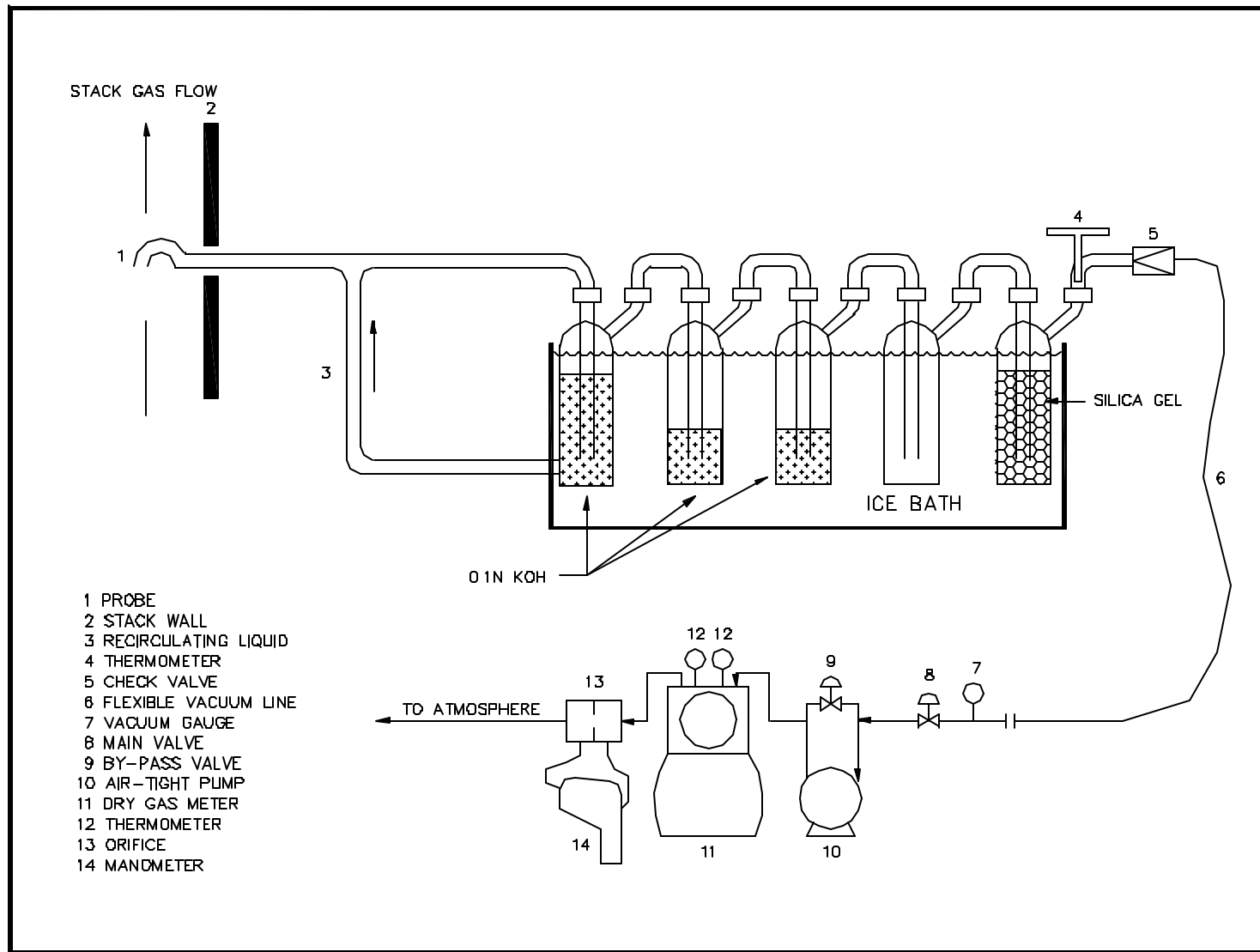


Figure 1. Method 0061 Sample Train for Hexavalent Chromium

Table A-9. Method 0061 Stack Gas Hexavalent Chromium Sampling Procedure (Continued)

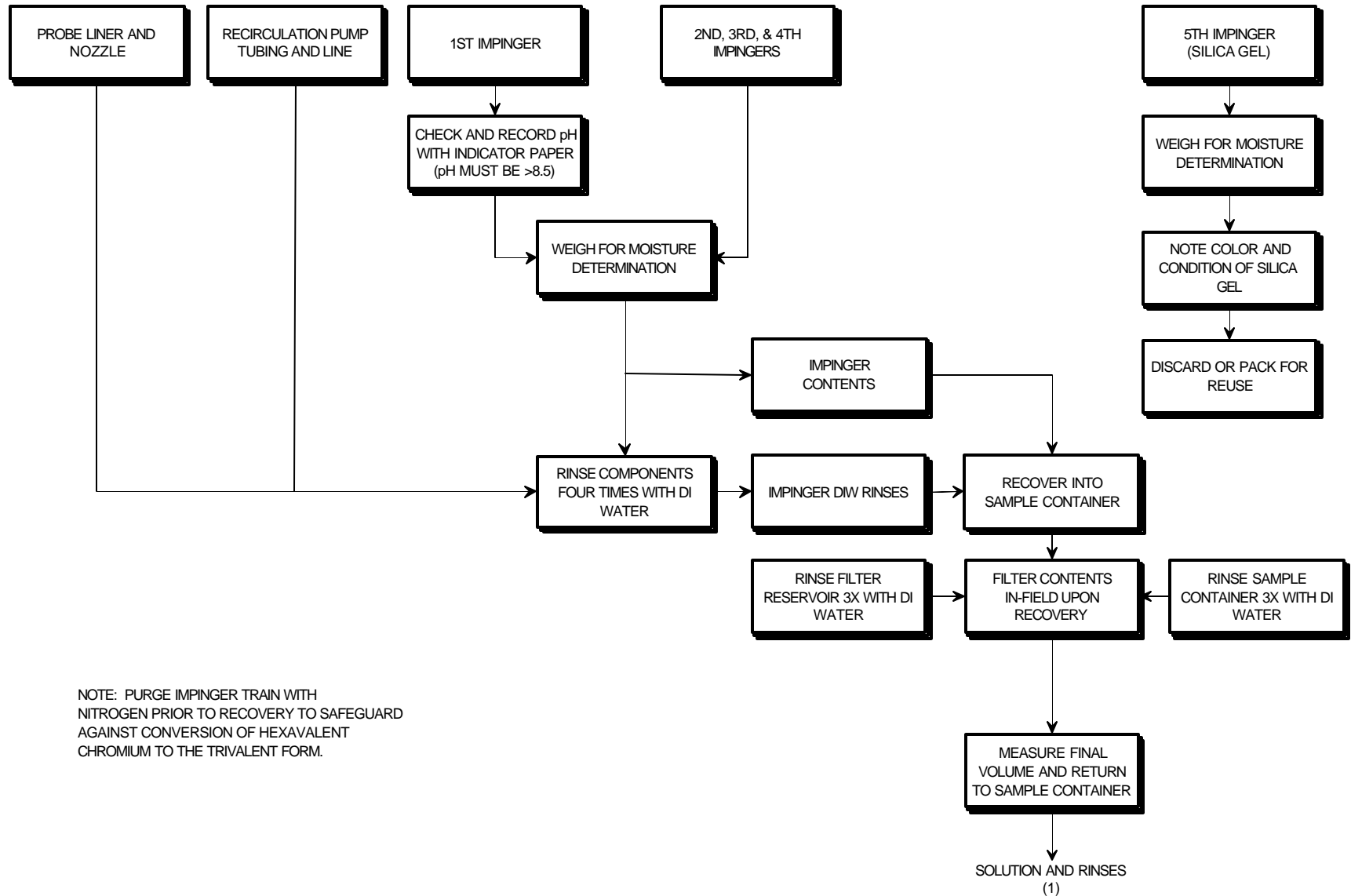


Figure 2. Method 0061 Hexavalent Chromium Sample Recovery

Table A-10. Method 0010 Stack Gas Semivolatile Organic and Organochlorine Pesticides Sampling Procedure

Sample name:	Stack Gas Method 0010 (SVOC & OCP)
Sampler:	Stack sampling team
Locations:	Exhaust stack
Equipment:	Method 0010 sampling train as shown in Figure 1 of this table; aluminum foil; amber glass sample bottles with Teflon-lined lids; tare weighed particulate filters in sealed petri dishes; 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 semivolatile organics on a particulate filter, adsorbent resin, and in impinger solutions. The train component samples are recovered in the field and shipped to an analytical laboratory where the mass amounts of semivolatile organics present are determined by extraction, concentration, and analysis by GC/MS.

The Method 0010 train includes a heated probe and particulate filter, a water-cooled condenser with an integral adsorbent resin trap, a condenser cooling water system, a condensate knockout impinger, two deionized water impingers, an empty impinger, and a silica gel impinger. All impingers are placed in an ice bath.

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 ($\geq 50^{\circ}\text{C}$) 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 450°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 XAD-2 adsorbent resin is prepared according to the procedures specified in Appendix A of Method 0010, which are summarized as follows:

- XAD-2 resin is cleaned by water rinses followed by soxhlet extractions with water, methanol, and methylene chloride, then dried using a flow of clean inert gas. An extract is prepared from a portion of the XAD-2 resin and 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.
- The adsorbent trap is loaded with approximately 40 g of the XAD-2 resin. The resin is spiked with an isotope labeled surrogate for the semivolatile organics and organochlorine pesticides analysis and the open end of the resin trap is packed with clean glass wool. 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 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

Table A-10. Method 0010 Stack Gas Semivolatile Organic and Organochlorine Pesticides Sampling Procedure (Continued)

methylene chloride. A clean and inspected particulate filter is placed in the filter holder. Before each sampling run, the Sample Coordinator supplies the 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 deionized water, and Impinger 5 contains 200 to 300 grams of indicating silica gel. 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 apparatus or a continuous emission monitor system (CEMS) is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to Methods 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 procedures given in Method 0010. The sample probe exit and filter assembly temperatures are maintained at $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$, and the gas sample entering the adsorbent module is maintained at or below 68°F . The sampling rate is adjusted during the test to maintain isokinetic conditions at the probe nozzle within $\pm 10\%$ 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 procedures are summarized below.

- Particulate Filter -- The particulate filter is removed from its holder and placed into its original petri dish (Container 1) which is sealed with Teflon tape and placed in a plastic bag.
- Front Half Rinse -- The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware is brushed and rinsed three times with methanol/methylene chloride (1:1 v/v). All rinses are placed into a glass sample bottle (Container 2).
- 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 (Container 3) and stored in an insulated cold chest.
- Condensate Knockout Impinger -- The condensate collected in Impinger 1 is measured to the nearest milliliter or weighed to the nearest 0.5 g. and transferred a glass sample bottle (Container 4). The back half of the particulate filter holder is also inspected for condensate and recovered, if observed, by measuring or weighing and transferring to Container No. 4 as described above.
- Back Half Rinse -- The back half of the filter holder, the condenser, and the connecting line between the holder and the condenser are rinsed

Table A-10. Method 0010 Stack Gas Semivolatile Organic and Organochlorine Pesticides Sampling Procedure (Continued)

three times with methanol/methylene chloride (1:1 v/v). The condensate knockout impinger (Impinger 1) is rinsed with methanol/methylene chloride (1:1 v/v). The rinses are then placed into a glass sample bottle with a Teflon lined lid (Container 5).

- Impinger water -- The contents of 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. If no color or film is present, the impinger water may be discarded. If color or film is observed, the impinger water is recovered and added to the condensate sample (Container 4).
- Silica Gel -- The silica gel contents of impinger 5 are weighed to the nearest 0.5 g.

Once during the test program, a blank train is prepared, set up at the sampling location, and leak tested at the beginning and end of one of the runs. The particulate filter holder and probe is heated for the duration of the sampling period, but no gas will pass through the train. The nozzle is capped with aluminum foil and the exit end of the last impinger is sealed with a cap. The train will remain assembled at the sampling location for a period equivalent to one test run. The blank train samples are recovered using the procedures described above. A Sample of the methanol/methylene chloride is collected once during the test as a reagent blank. An unused particulate filter and unused XAD-2 resin trap are also collected during the test as trip blanks. (Note: The reagent and trip blanks are archived in the event that blank sample analyses indicate a potential contamination problem.)

All of the sample containers is 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 0010 - Modified Method 5 Sampling Train”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates
 - “Method 0010 – Appendix A – Preparation of XAD-2 Adsorbent Resin”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates
 - “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 through 5: Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.
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Table A-10. Method 0010 Stack Gas Semivolatile Organic and Organochlorine Pesticides Sampling Procedure (Continued)

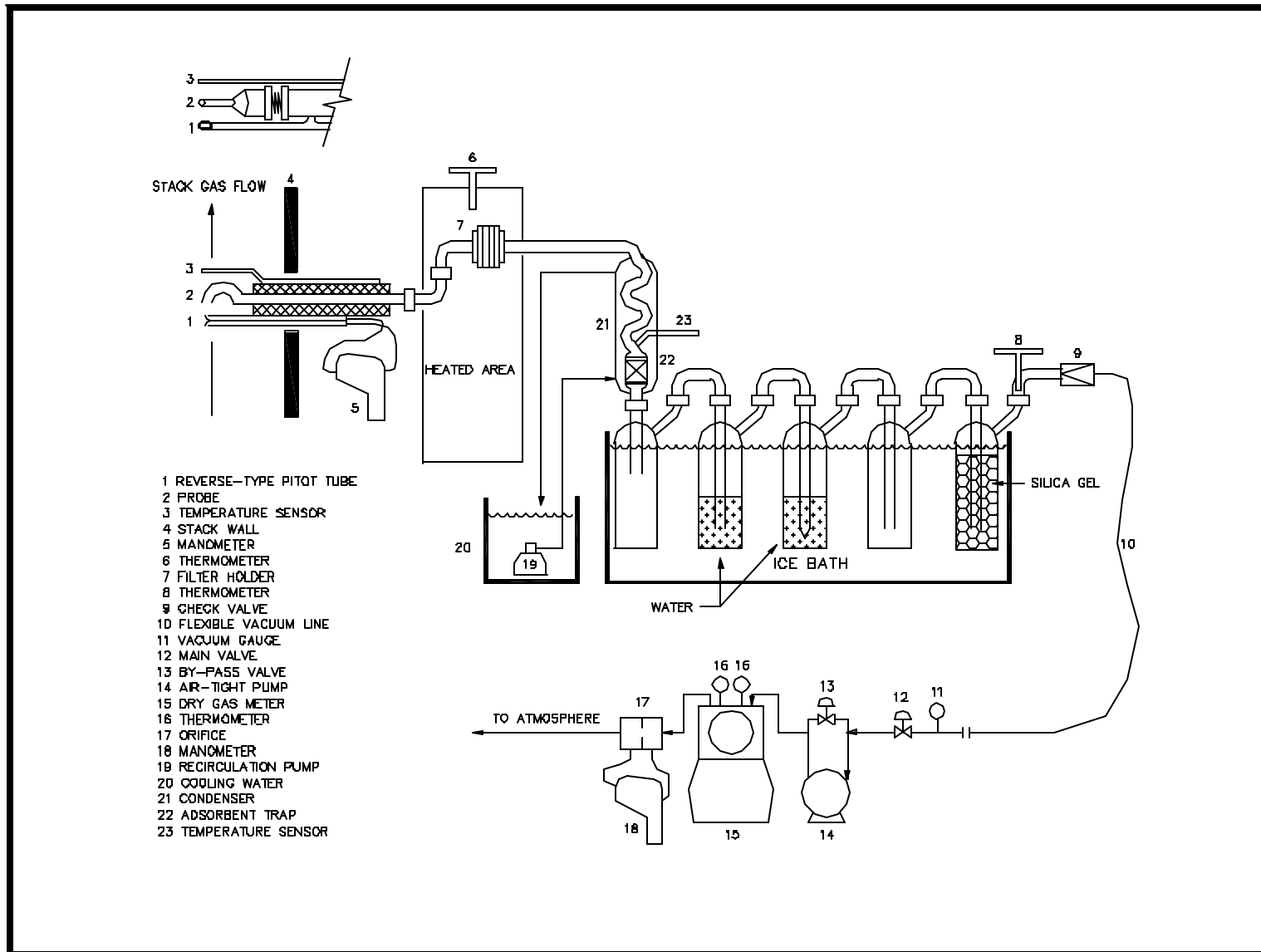


Figure 1. Method 0010 Sample Train for Semivolatile Organics

Table A-10. Method 0010 Stack Gas Semivolatile Organic and Organochlorine Pesticides Sampling Procedure (Continued)

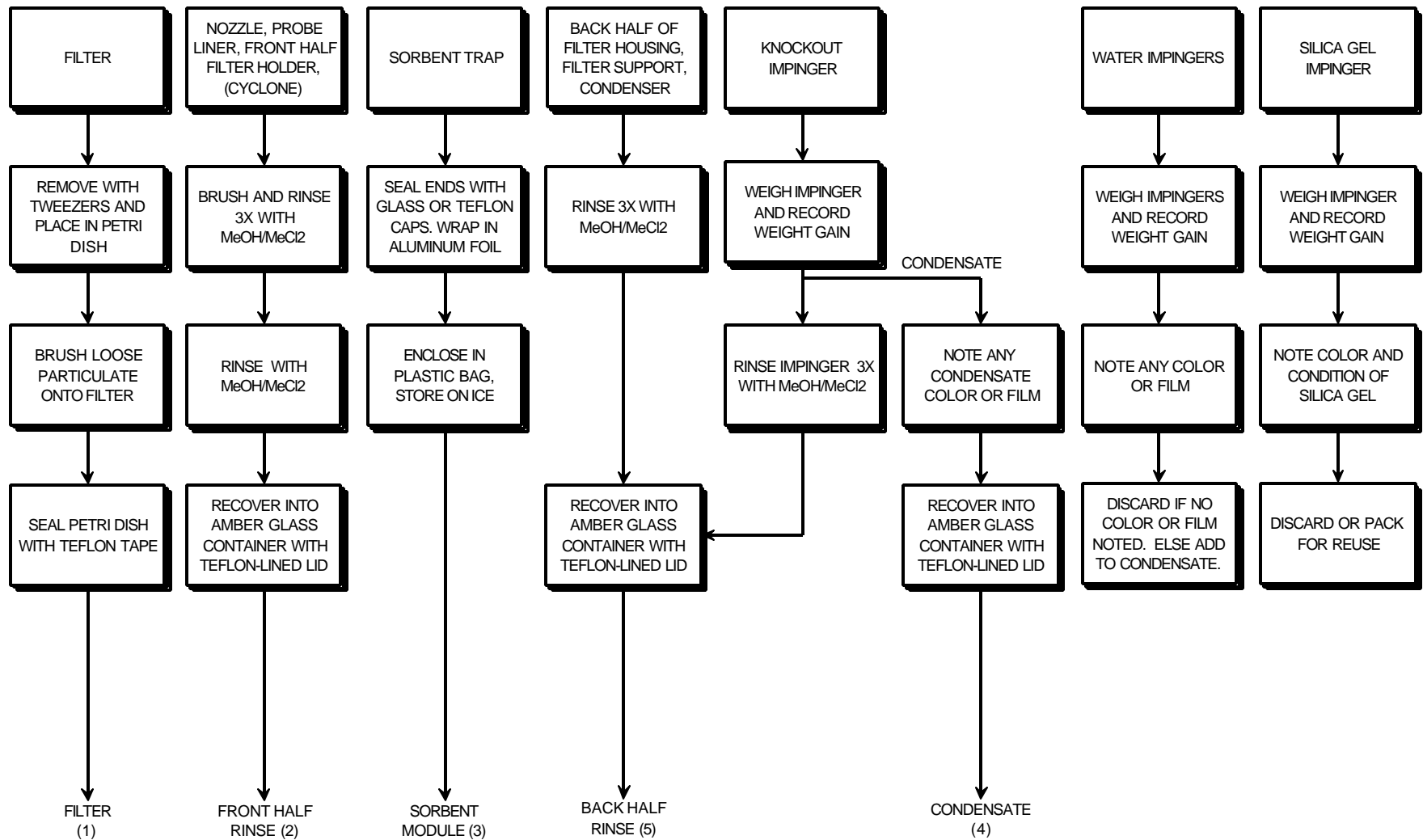


Figure 2. Method 0010 Semivolatile Organics Sample Recovery

Table A-11. Method 0010 Stack Gas Polyaromatic Hydrocarbons and Polychlorinated Biphenyls Sampling Procedure

Sample name:	Stack Gas Method 0010 (PAH & PCB)
Sampler:	Stack sampling team
Locations:	Exhaust stack
Equipment:	Method 0010 sampling train as shown in Figure 1 of this table; aluminum foil; amber glass sample bottles with Teflon-lined lids; tare weighed particulate filters in sealed petri dishes; 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 semivolatile organics on a particulate filter, adsorbent resin, and in impinger solutions. The train component samples are recovered in the field and shipped to an analytical laboratory where the mass amounts of semivolatile organics present are determined by extraction, concentration, and analysis by GC/MS.

The Method 0010 train includes a heated probe and particulate filter, a water-cooled condenser with an integral adsorbent resin trap, a condenser cooling water system, a condensate knockout impinger, two deionized water impingers, an empty impinger, and a silica gel impinger. All impingers are placed in an ice bath.

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 ($\geq 50^{\circ}\text{C}$) 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 450°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 XAD-2 adsorbent resin is prepared according to the procedures specified in Appendix A of Method 0010, which are summarized as follows:

- XAD-2 resin is cleaned by water rinses followed by soxhlet extractions with water, methanol, and methylene chloride, then dried using a flow of clean inert gas. An extract is prepared from a portion of the XAD-2 resin and 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.
- The adsorbent trap is loaded with approximately 40 g of the XAD-2 resin. The resin is spiked with an isotope labeled surrogate for the PAH and PCB analysis and the open end of the resin trap is packed with clean glass wool. 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 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

Table A-11. Method 0010 Stack Gas Polyaromatic Hydrocarbons and Polychlorinated Biphenyls Sampling Procedure (Continued)

methylene chloride. A clean and inspected particulate filter is placed in the filter holder. Before each sampling run, the Sample Coordinator supplies the 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 deionized water, and Impinger 5 contains 200 to 300 grams of indicating silica gel. 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 apparatus or a continuous emission monitor system (CEMS) is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to Methods 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 procedures given in Method 0010. The sample probe exit and filter assembly temperatures are maintained at $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$, and the gas sample entering the adsorbent module is maintained at or below 68°F . The sampling rate is adjusted during the test to maintain isokinetic conditions at the probe nozzle within $\pm 10\%$ 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 procedures are summarized below.

- Particulate Filter -- The particulate filter is removed from its holder and placed into its original petri dish (Container 1) which is sealed with Teflon tape and placed in a plastic bag.
- Front Half Rinse -- The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware is brushed and rinsed three times with methanol/methylene chloride (1:1 v/v). All rinses are placed into a glass sample bottle (Container 2).
- 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 (Container 3) and stored in an insulated cold chest.
- Condensate Knockout Impinger -- The condensate collected in Impinger 1 is measured to the nearest milliliter or weighed to the nearest 0.5 g. and transferred a glass sample bottle (Container 4). The back half of the particulate filter holder is also inspected for condensate and recovered, if observed, by measuring or weighing and transferring to Container No. 4 as described above.
- Back Half Rinse -- The back half of the filter holder, the condenser, and the connecting line between the holder and the condenser are rinsed

Table A-11. Method 0010 Stack Gas Polyaromatic Hydrocarbons and Polychlorinated Biphenyls Sampling Procedure (Continued)

three times with methanol/methylene chloride (1:1 v/v). The condensate knockout impinger (Impinger 1) is rinsed with methanol/methylene chloride (1:1 v/v). The rinses are then placed into a glass sample bottle with a Teflon lined lid (Container 5).

- Impinger water -- The contents of 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. If no color or film is present, the impinger water may be discarded. If color or film is observed, the impinger water is recovered and added to the condensate sample (Container 4).
- Silica Gel -- The silica gel contents of impinger 5 are weighed to the nearest 0.5 g.

Once during the test program, a blank train is prepared, set up at the sampling location, and leak tested at the beginning and end of one of the runs. The particulate filter holder and probe is heated for the duration of the sampling period, but no gas will pass through the train. The nozzle is capped with aluminum foil and the exit end of the last impinger is sealed with a cap. The train will remain assembled at the sampling location for a period equivalent to one test run. The blank train samples are recovered using the procedures described above. A Sample of the methanol/methylene chloride is collected once during the test as a reagent blank. An unused particulate filter and unused XAD-2 resin trap are also collected during the test as trip blanks. (Note: The reagent and trip blanks are archived in the event that blank sample analyses indicate a potential contamination problem.)

All of the sample containers is 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 0010 - Modified Method 5 Sampling Train”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates
 - “Method 0010 – Appendix A – Preparation of XAD-2 Adsorbent Resin”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates
 - “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 through 5: Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.
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Table A-11. Method 0010 Stack Gas Polyaromatic Hydrocarbons and Polychlorinated Biphenyls Sampling Procedure (Continued)

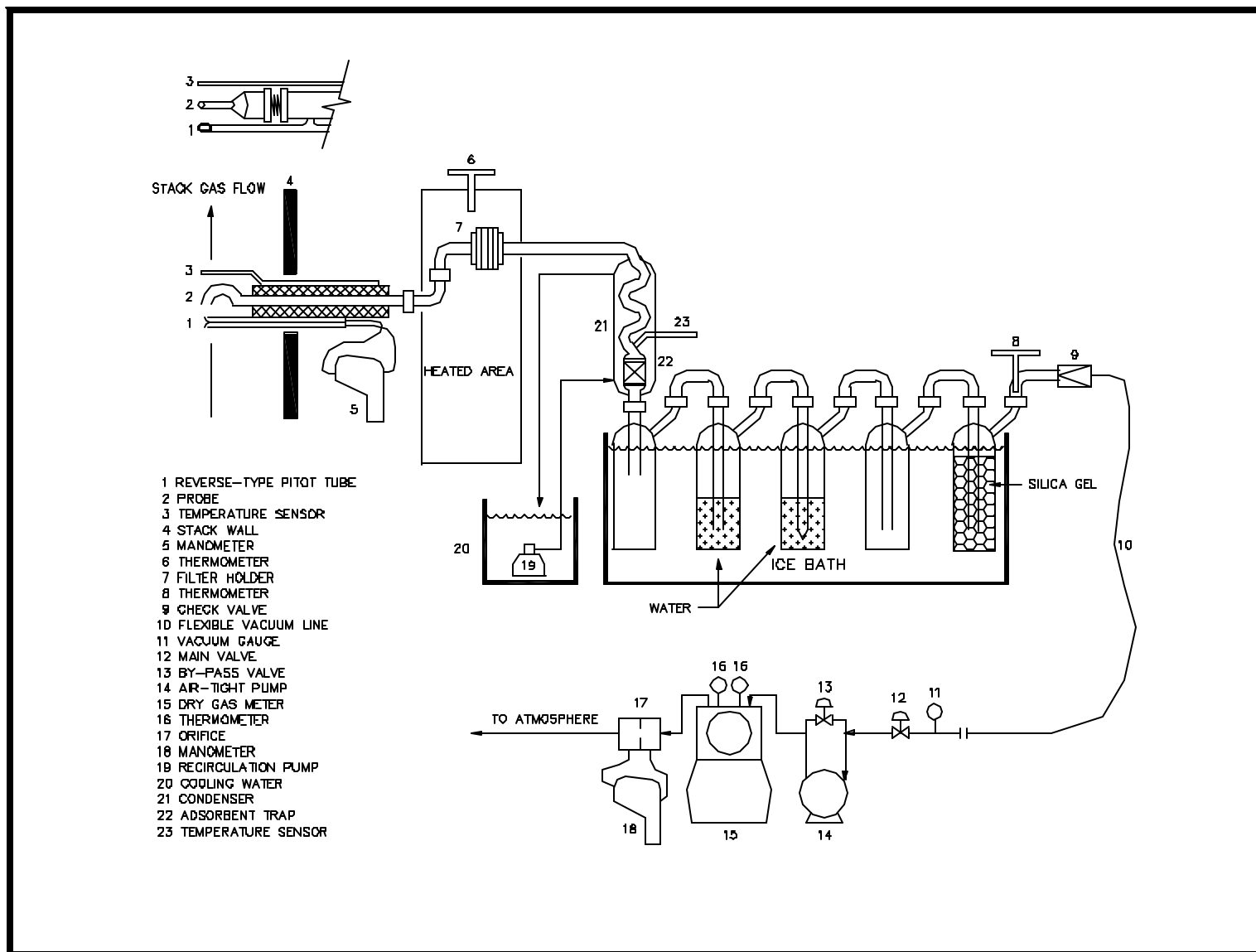


Figure 1. Method 0010 Sample Train for Semivolatile Organics

Table A-11. Method 0010 Stack Gas Polyaromatic Hydrocarbons and Polychlorinated Biphenyls Sampling Procedure (Continued)

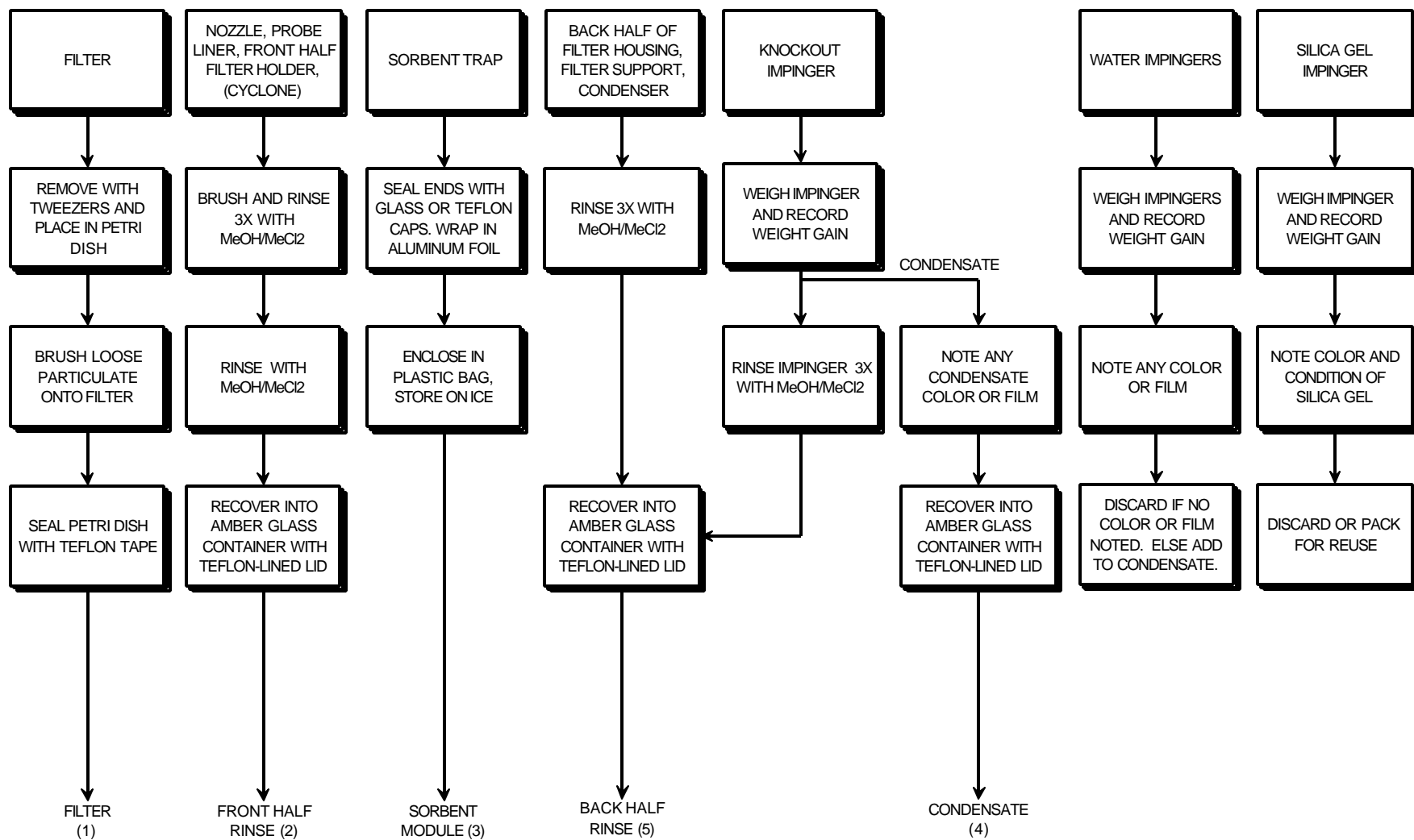


Figure 2. Method 0010 Semivolatile Organics Sample Recovery

Table A-12. Method 0010 Stack Gas Total Semivolatile and Nonvolatile Organic Sampling Procedure

Sample name: Stack Gas Method 0010 (Total SVOC & NVOC)

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 0010 sampling train as shown in Figure 1 of this table; aluminum foil; amber glass sample bottles with Teflon-lined lids; tare weighed particulate filters in sealed petri dishes; 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 semivolatile and non-volatile organics on a particulate filter, adsorbent resin, and in impinger solutions. The train component samples are recovered in the field and shipped to an analytical laboratory where the mass amount of total semivolatile organics present is subsequently determined by chromatograph/flame ionization detector (GC/FID) analysis of the combined sample extract. The mass amount of total non-volatile organics present is determined by gravimetric analysis after evaporating of a portion of the combined sample extract.

The Method 0010 train includes a glass nozzle, heated probe and particulate filter, a water cooled condenser, adsorbent resin trap, a condenser cooling water system, a condensate knockout impinger, two deionized water impingers, an empty impinger, and a silica gel impinger. All impingers are placed in an ice bath. An air tight 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 ($\geq 50^{\circ}\text{C}$) 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 XAD-2 adsorbent resin is prepared according to the procedures specified in Appendix A of Method 0010, which are summarized as follows:

- XAD-2 resin is cleaned by water rinses followed by soxhlet extractions with water, methanol, and methylene chloride, then dried using a flow of inert gas. An extract is prepared from a portion of the XAD-2 resin and 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 adsorbent trap is loaded with approximately 40 g of the XAD-2 resin and the open end of the resin trap is packed with clean glass wool. The ends of the adsorbent trap are capped and the trap is wrapped in aluminum foil, sealed in a plastic bag, and stored in an insulated cold chest. The resin is stored at less than 120°F at all times. (Note: No surrogates are added to the resin because the sample analysis does not involve the identification of individual organic compounds.)

Table A-12. Method 0010 Stack Gas Total Semivolatile and Nonvolatile Organic Sampling Procedure
(Continued)

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 clean and inspected particulate filter is placed in the filter holder. Before each sampling run, the Sample Coordinator supplies the 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 deionized water, and Impinger 5 contains 200 to 300 grams of indicating silica gel. 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 apparatus or a continuous emission monitoring system (CEMS) is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to 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 procedures given in Method 0010. The sample probe exit and filter assembly temperatures are maintained at $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$, and the gas sample entering the adsorbent module is maintained at or below 68°F . The sampling rate is adjusted during the test to maintain isokinetic conditions at the probe nozzle within $\pm 10\%$ 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 procedures are summarized below.

- Particulate Filter -- The particulate filter is removed from its holder and placed into its original petri dish (Container 1) which is sealed with Teflon tape and placed in a plastic bag.
- Front Half Rinse -- The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware is brushed and rinsed three times with methanol/methylene chloride (1:1 v/v). All rinses are placed into a glass sample bottle (Container 2).
- 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 (Container 3) and stored in an insulated cold chest.
- Condensate Knockout Impinger -- The condensate collected in Impinger 1 is measured to the nearest milliliter or weighed to the nearest 0.5 gram and transferred to a glass sample bottle (Container 4). The back half of the particulate filter holder is also inspected for condensate and recovered, if observed, by measuring or weighing and transferring to Container 4 as described above.

Table A-12. Method 0010 Stack Gas Total Semivolatile and Nonvolatile Organic Sampling Procedure
(Continued)

- Back Half Rinse -- The back half of the filter holder, the condenser, and the connecting line between the holder and the condenser are rinsed three times with methanol/methylene chloride (1:1 v/v). The condensate knockout impinger (Impinger 1) is rinsed with methanol/methylene chloride (1:1 v/v). The rinses are then placed into a glass sample bottle with a Teflon lined lid (Container 5).
- Impinger water -- The contents of 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 noted on the sample recovery sheet. If no color or film is present, the impinger water may be discarded. If color or film is observed, the impinger water is recovered and added to the condensate sample (Container 4).
- Silica Gel -- The silica gel contents of impinger 5 are weighed to the nearest 0.5 g.

Once during the test program, a blank train is prepared, set up at the sampling location, and leak tested at the beginning and end of one of the runs. The particulate filter holder and probe is heated for the duration of the sampling period, but no gas will pass through the train. The nozzle is capped with aluminum foil and the exit end of the last impinger is sealed with a cap. The train remains assembled at the sampling location for a period equivalent to one test run. The blank train samples are recovered using the procedures described above. Samples of the methanol/methylene chloride are collected once during the test as a reagent blank. A blank filter and an XAD-2 resin trap are also collected once during the test.

All of the sample containers is 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 0010 - Modified Method 5 Sampling Train”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates
 - “Method 0010 – Appendix A – Preparation of XAD-2 Adsorbent Resin”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates
 - “Guidance for Total Organics”, EPA/600/R-96/036
 - “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 through 5: Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.
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Table A-12. Method 0010 Stack Gas Total Semivolatile and Nonvolatile Organic Sampling Procedure (Continued)

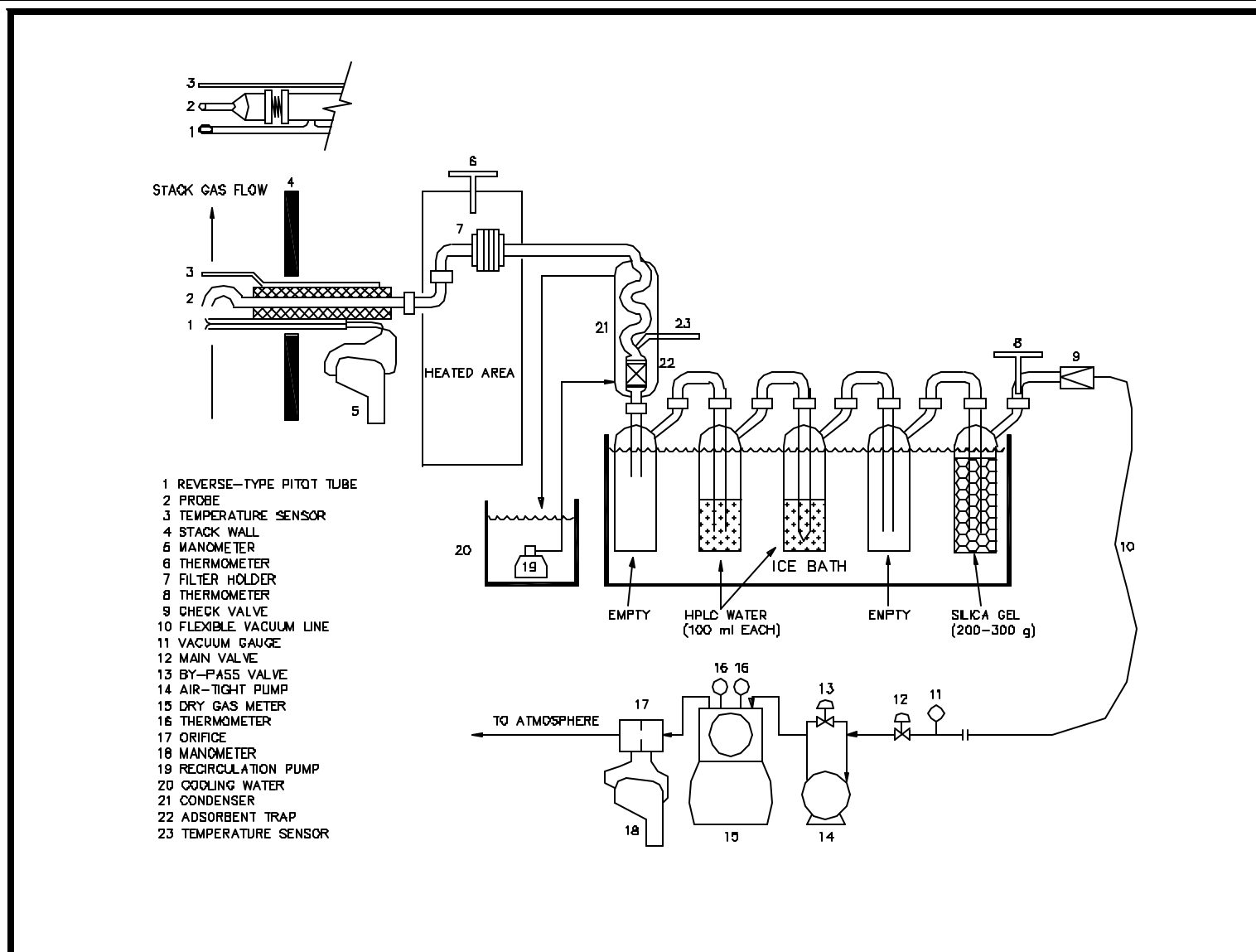


Figure 1. Method 0010 Sample Train for Semivolatile and Nonvolatile Organics

Table A-12. Method 0010 Stack Gas Total Semivolatile and Nonvolatile Organic Sampling Procedure (Continued)

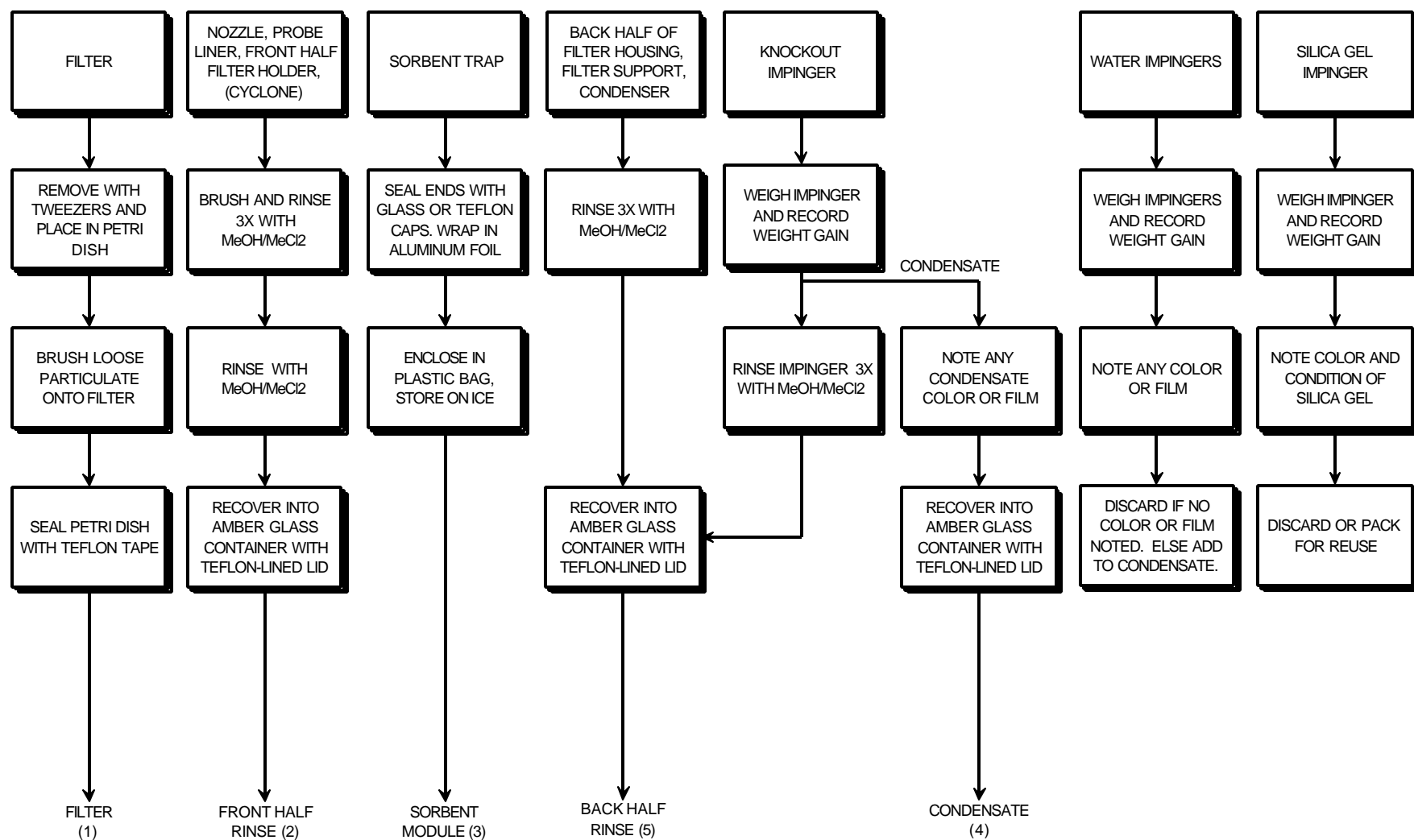


Figure 2. Method 0010 Semivolatile and Nonvolatile Organics Sample Recovery

Table A-13. Method 0023A Stack Gas PCDD/PCDF Sampling Procedure

Sample name:	Stack Gas M0023A
Sampler:	Stack sampling team
Locations:	Exhaust stack
Equipment:	Method 0023A sampling train as shown in Figure 1 of this table; tare weighed glass fiber particulate filters (without organic binders) sealed in glass petri dishes; prepared XAD-2 adsorbent resin traps; aluminum foil; amber glass bottles with Teflon-lined lids; scale balance; glass graduated cylinder.
Frequency:	Continuous during each test run. Minimum sample time for each run is determined based on analytical detection limits, sampling rates, and desired in-stack concentration detection limits.

Procedure

Summary: Stack gases are sampled isokinetically to collect PCDD/PCDF on a particulate filter and on XAD-2 adsorbent resin. The mass of PCDD/PCDF collected is quantitatively determined by HRGC/HRMS analysis of the samples recovered from the train components.

The Method 0023A train includes a glass probe nozzle, a heated sample probe with a borosilicate or quartz glass probe liner, heated particulate filter holder with Teflon frit filter support, a water cooled condenser, a water cooled adsorbent resin trap, a condensate knockout impinger, two water impingers, an empty impinger (optional), and a silica gel impinger. A water pump is provided to circulate ice bath water through the condenser and around the adsorbent 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 ($\geq 50^{\circ}\text{C}$) 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 clean aluminum foil or clean glass plugs until sample train assembly.

The particulate filters and the XAD-2 adsorbent resin are prepared according to Method 0023A procedures summarized as follows:

- The filters are cleaned by soxhlet extraction using toluene. Following extraction, each filter is dried in a stream of clean dry inert gas and stored in a glass petri dish sealed with Teflon tape.
- XAD-2 resin is cleaned by rinsing twice with organic-free (HPLC) water followed by soxhlet extractions with HPLC water, methanol, methylene chloride, and toluene, and then dried using a flow of clean dry 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 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

Table A-13. Method 0023A Stack Gas PCDD/PCDF Sampling Procedure (Continued)

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.

- A sample of the XAD-2 resin used in preparing the traps is collected as a laboratory method blank. The method blank is retained in the laboratory for analysis with the field samples.

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 g 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 g of indicating silica gel. All impingers are weighed separately to nearest 0.5 g 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 apparatus or a continuous emission monitoring system (CEMS) is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to Method 3, 3A, or 3C procedures. The stack gas moisture content is determined according to Method 4.

A sample train leak check is performed before each test run according to Method 0023A. The probe inlet is plugged, the probe and filter holder heaters are set to maintain 248 °F, and a vacuum of 15 inches Hg is applied to the system. A lower vacuum may be used, but must not be exceeded during the test run. The leak rate must be ≤ 0.02 cfm or less than 4% of the average sampling rate before starting the test run.

The stack gas sampling is conducted following the procedures given in Method 0023A. The sample probe exit and filter assembly temperatures are maintained at $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$, 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 $\pm 10\%$ of 100%. A leak check is conducted during the test run whenever the sampling probe is removed from a sample port, such as during the stack traverse port change.

After sampling, the probe is removed from the stack and a post-test leak check is conducted. The test run is voided if the leak rate exceeds 0.02 cfm or 4% of the average sampling rate (whichever is less).

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 particulate filter is removed from its holder and placed into its original petri dish (Container No. 1) which is sealed with Teflon tape and placed in a plastic bag.

Table A-13. Method 0023A Stack Gas PCDD/PCDF Sampling Procedure (Continued)

- Front Half Rinse -- 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 with acetone and rinsed three times with methylene chloride and two times with toluene. All rinses are placed into a glass sample bottle (Container No. 2). The final liquid level is marked on the sample container.
- XAD-2 Adsorbent Resin Trap -- The XAD-2 adsorbent resin trap is removed from the train, and both ends are capped. The trap is then labeled, covered with aluminum foil, sealed in a plastic bag and stored on ice in an insulated cold chest (Container No. 3).
- Back Half Rinse -- 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 followed by two rinses with methylene chloride and two rinses with toluene. The back half rinses are placed into a glass sample bottle (Container No. 4). The final liquid level is marked on the sample container.
- Impinger water -- The liquid contents of the impingers are measured to the nearest milliliter or weighed to the nearest 0.5 g and are then discarded. 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 train recovery and cleanup procedures is provided in Figure 2. Following sample recovery, the sample train may be used at the same sampling location.

Field blank samples are collected from a blank sampling train once during the test program. A complete train is assembled from glassware that has not been used to collect any field samples. The train is placed in the sampling area, brought up to operating temperature, leak tested, and the nozzle is capped with aluminum foil or Teflon tape and the exit end of the last impinger is sealed with a glass cap. The train remains idle (no sample gas flow) for the duration of the test run and the leak test is repeated. The field blank samples are recovered using the same procedures described above for the stack gas samples.

Reagent blank samples are collected from each of the acetone, methylene chloride, and toluene wash bottles used in the recovery of the stack gas samples. (Note: The reagent blank samples are archived for analysis in the event that a field blank analytical result indicates a potential contamination problem.)

A glassware blank sample (proof blank) may be recovered once during the test program from a set of sampling train glassware that has not been used to collect any field samples. A probe liner, filter holder, condenser coil, and impinger set are assembled as if for sampling and then recovered using the same procedures described above for the stack gas samples. (Note: The collection of this sample is optional because its purpose is to check the effectiveness of the glassware cleaning procedure. The proof blank samples are archived for analysis in the event that a field blank analytical result indicates a potential contamination problem.)

Table A-13. Method 0023A Stack Gas PCDD/PCDF Sampling Procedure (Continued)

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: SW-846 Method 0023A, "Sampling Method for Polychlorinated Dibenzo-p-Dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Emissions from Stationary Sources", Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.

"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 3A – Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrument Analyzer Procedure)"

"Method 3C – Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen from Stationary Sources"

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

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

Reference for Methods 1 through 5: Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.

Table A-13. Method 0023A Stack Gas PCDD/PCDF Sampling Procedure (Continued)

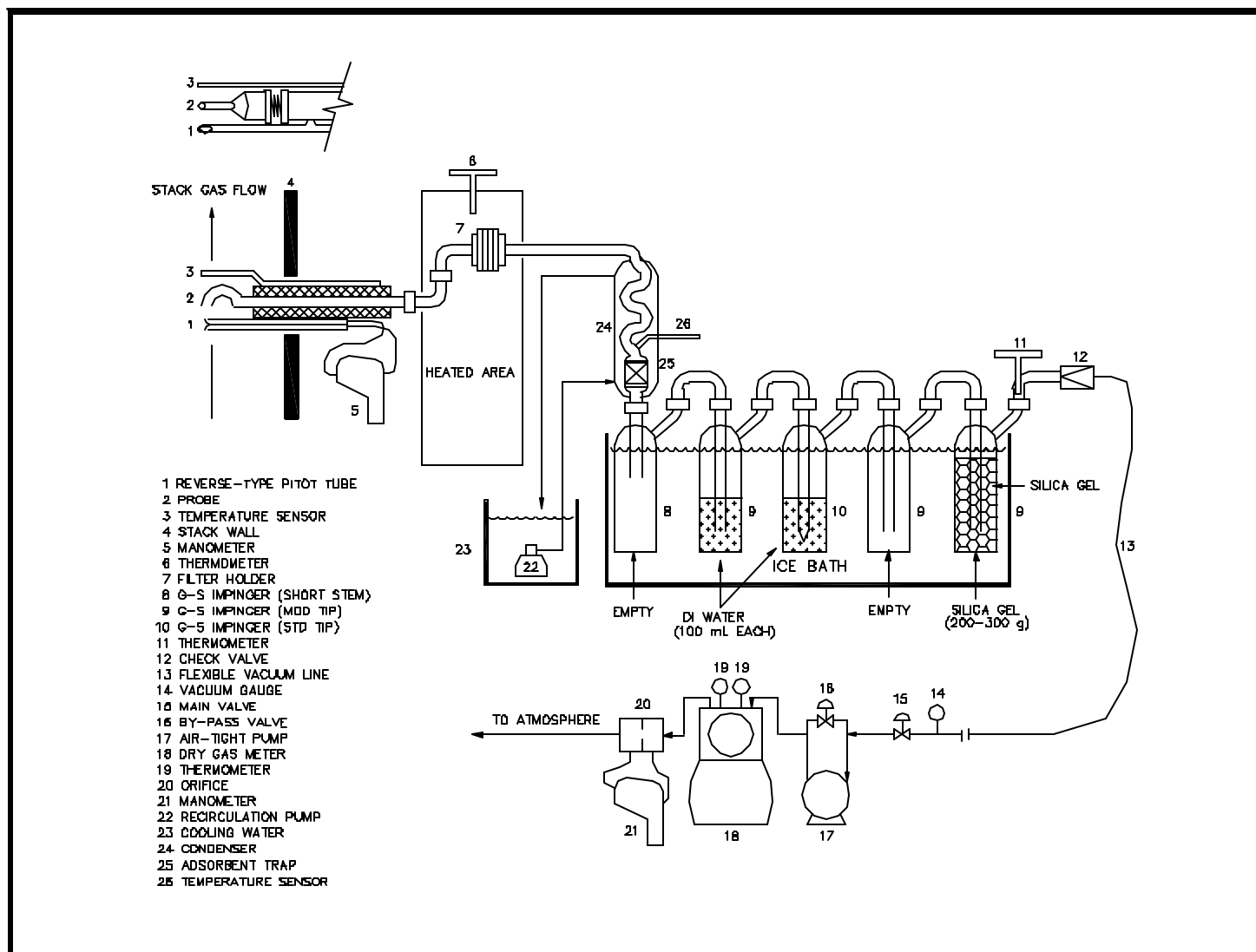


Figure 1. Method 0023A Sample Train for PCDD/PCDF

Table A-13. Method 0023A Stack Gas PCDD/PCDF Sampling Procedure (Continued)

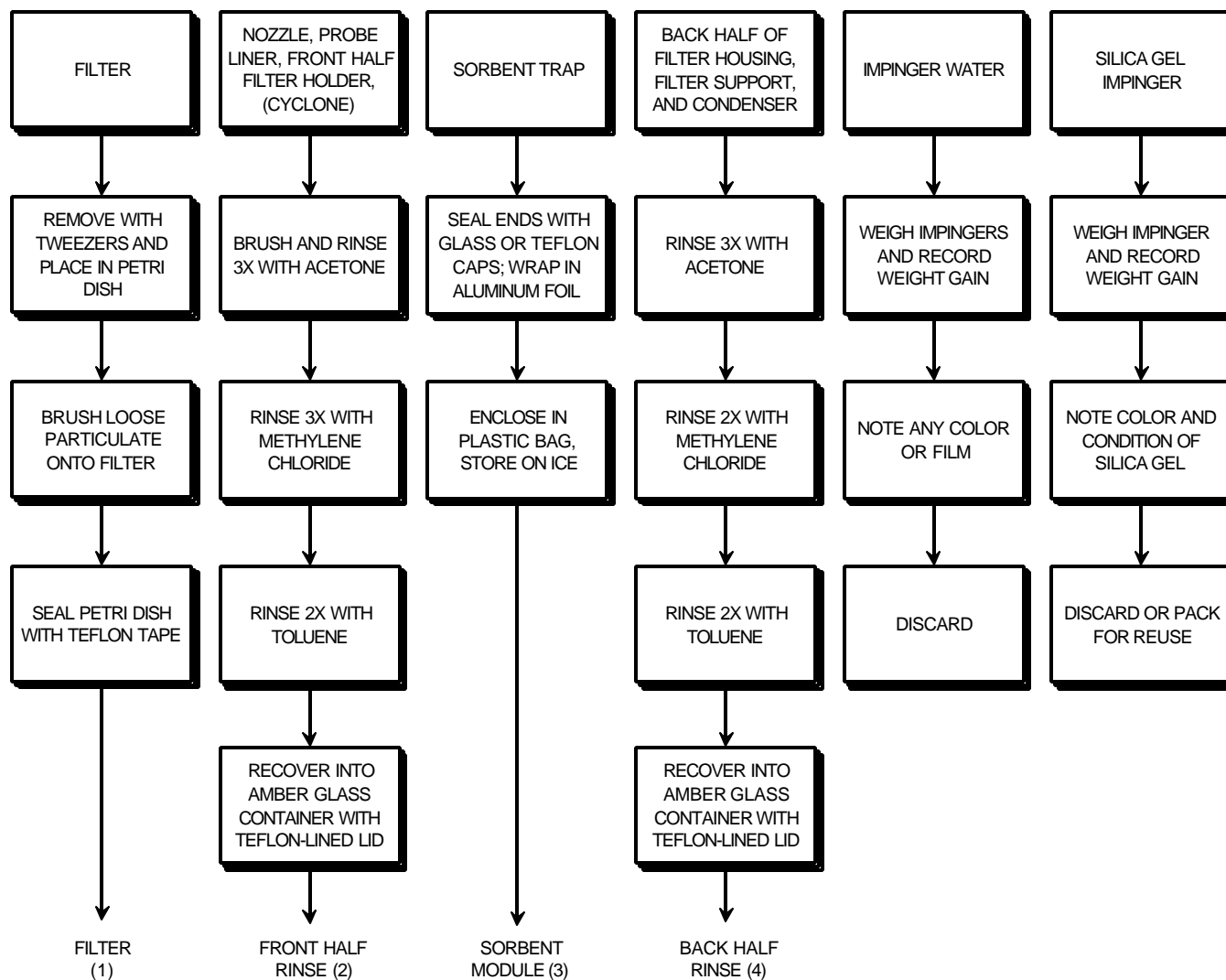


Figure 2. Method 0023A PCDD/PCDF Sample Recovery

Table A-14. Method 0040 Stack Gas Volatile Organic Compound Sampling Procedure

Sample name:	Stack Gas Method 0040 (Total VOC)
Sampler:	Stack sampling team
Location:	Stack Sampling Port
Equipment:	Method 0040 sampling train as shown in Figure 1 of this table; quartz fiber particulate filters; 25-35 liter Tedlar (or equivalent) sample bags; glass VOA vials (40 mL) with plastic screw caps and Teflon septa; graduated glass cylinder.
Frequency:	Continuous to fill over 80% of the sample bag during each test run.
Procedure Summary:	<p>Stack gases are withdrawn at a controlled rate, cooled to condense moisture, and collected in a flexible bag. The stack gas condensate samples and the dry gas samples in the flexible bags are analyzed by GC/FID for total VOCs following the procedures presented in "Guidance for Total Organics" referenced below. (Note: The total mass of VOCs is based on analyses for C₁-C₇ n-alkane compound groups and not on individual VOC analytes. Individual major n-alkanes are methane, ethane, propane, butane, hexane, and heptane.)</p> <p>The sampling train includes a glass-lined stainless steel probe, a heated glass or Teflon filter holder with a quartz filter, a 3-way isolation valve, a water-cooled glass condenser and condensate trap, a second 3-way isolation valve, an air tight evacuation container, a Tedlar sample bag, silica gel and charcoal tubes, and a VOST control console. The control console includes a needle valve, vacuum pump, and a flow meter. A third 3-way isolation valve is included in the purge line to allow clean air to enter the system when performing leak checks.</p> <p>The sampling train components and reagents are prepared according to the procedures specified in Method 0040. As described in the method, the train glassware is cleaned with nonionic detergent and hot water in an ultrasonic bath, rinsed three times each with distilled deionized water, nitric acid, and distilled deionized water, and dried in an oven. The Teflon lines, fittings, and sample bags are cleaned in a similar manner except that they are allowed to air dry in an area that is free of organic compounds. The rigid containers are hand washed and allowed to dry in air.</p> <p>The gas sample bags are given positive pressure leak checks before each sampling run. For a leak check, the bag is connected to a water manometer and pressurized to 2 to 4 inches of water column and the bag is allowed to stand for 10 minutes. Any displacement of the manometer water indicates a leak. The rigid bag container is leak tested in the same manner.</p> <p>All sample bags are flushed three times with high purity nitrogen and then filled with the nitrogen. The nitrogen gas in each bag is analyzed for an acceptable level of VOC contamination, which is a response five times the method detection limit or one-half of the level of concern, whichever is less. Nitrogen flushes are repeated until an acceptable level is achieved. The number of bags submitted for analysis may be reduced if analytical results indicate that all bags have an acceptable level of contamination.</p> <p>Two laboratory blank samples consisting of nitrogen filled bags are retained in the laboratory using the same storage methods as the field samples. These samples are analyzed to help identify the source of contamination when field and trip blanks contain high concentrations of contaminants.</p>

Table A-14. Method 0040 Stack Gas Volatile Organic Compound Sampling Procedure
(Continued)

The sample train is operated according to procedures specified in Method 0040. The sample probe end is located at the centroid of the stack, the sample and vacuum lines are connected as indicated in Figure 1. The sample pump is started with the needle valve adjusted to yield a flow that will fill over 50% of the bag during the test period. After the probe, sample line, moisture condenser, and sample bag have been purged several times with the stack gas, the vacuum line connection is changed from the container to the sample bag and the bag is evacuated until the rotometer indicates no flow. [Note: It may be necessary, depending on the stack gas moisture content, to heat the moisture condenser and trap to prevent condensation during purging.]

A leak check of the sampling train is performed after the bag evacuation. The bag isolation valve is closed, the end of the probe is sealed, and a vacuum of 5 in. Hg is applied to the system. After the vacuum has stabilized, the leak rate is timed for 1 minute. A leak rate less than 0.1 in. Hg/min must be achieved before starting the test run.

The pump vacuum and the sample lines are changed back to their original positions (Figure 1). The sample probe and filter assembly heater is set to maintain $130^{\circ}\text{C} \pm 5^{\circ}\text{C}$ (266°F to 284°F) and the ice bath pump is turned on to circulate water through the condenser. The gas pump is then turned on to begin sample collection. The sampling rate during the run is maintained proportional to the stack gas velocity. The rigid container is protected from sunlight during the test run.

After collecting the sample, the pump is stopped, the probe removed from the stack, and the leak check is repeated at the highest vacuum attained during the test run. The sample recovery procedures are summarized in Figure 2 of this table. The bag container is removed to the sample holding location and protected from sunlight. The sample bags are stored in rigid, opaque containers in a restricted area. A sample of the condensate water is collected in a VOA vial with no headspace and any excess is discarded. If needed to eliminate headspace, organic-free (HPLC) water is added to the vial. The condensate samples are stored on ice or in a refrigerated container at 0°C (32°F). [Note: All samples are analyzed within 72 hours unless pretests have been conducted to show that significant deterioration ($>20\%$) does not occur for longer periods of storage.]

The particulate filter is removed and discarded. The silica gel trap is checked for moisture saturation and replaced if needed to protect the sample pump.

Field blank samples of gas and condensate are collected at least once per test day and for each emission source. A gas blank sample is prepared by drawing high-purity air or nitrogen from a compressed gas cylinder through the sampling system into a sample bag, which is recovered in the same manner as the stack gas samples. A condensate blank sample is prepared by filling a VOA vial with HPLC water. The blank samples are transported along with the stack gas samples.

A field spike sample is collected once for every 10 field samples or at least once per test program. The spike sample is prepared according to the procedures described in Section 8.2.5 of Method M0040, which include injecting isotope labeled analogs of the compounds of interest into a bag while a stack gas sample is being collected. The field spike samples are transported with the stack gas samples.

References: Method 0040 - Sampling of Principle Organic Hazardous Constituents from Combustion Sources using Tedlar Bags", Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986 and Updates.

Table A-14. Method 0040 Stack Gas Volatile Organic Compound Sampling Procedure
(Continued)

“Guidance for Total Organics” Draft Final Report, EPA/600/R-96/036 (PB97-118533),
November, 1996.

Table A-14. Method 0040 Stack Gas Volatile Organic Compound Sampling Procedure (Continued)

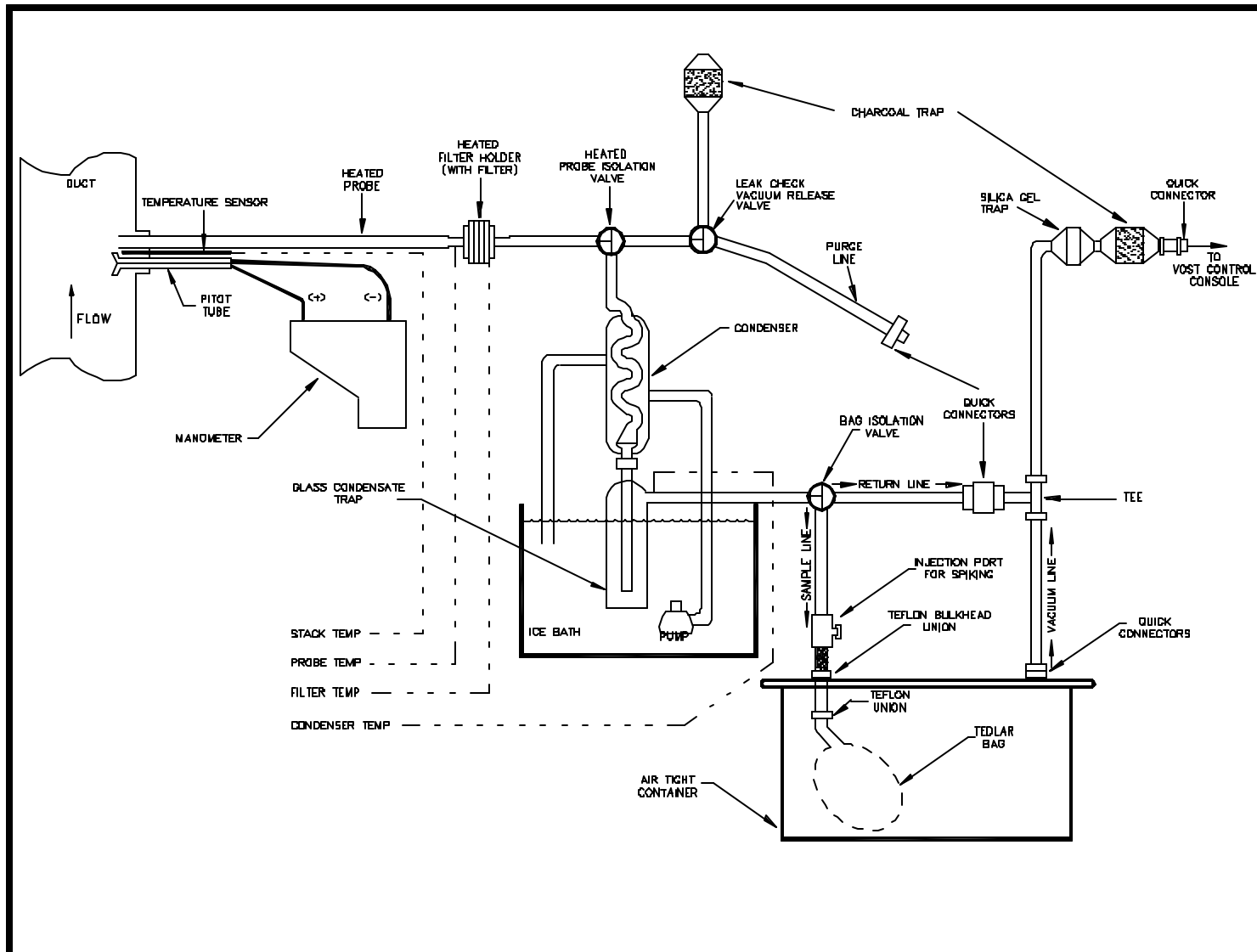


Figure 1. Method 0040 Sample Train for Volatile Organic Compounds

Table A-14. Method 0040 Stack Gas Volatile Organic Compound Sampling Procedure (Continued)

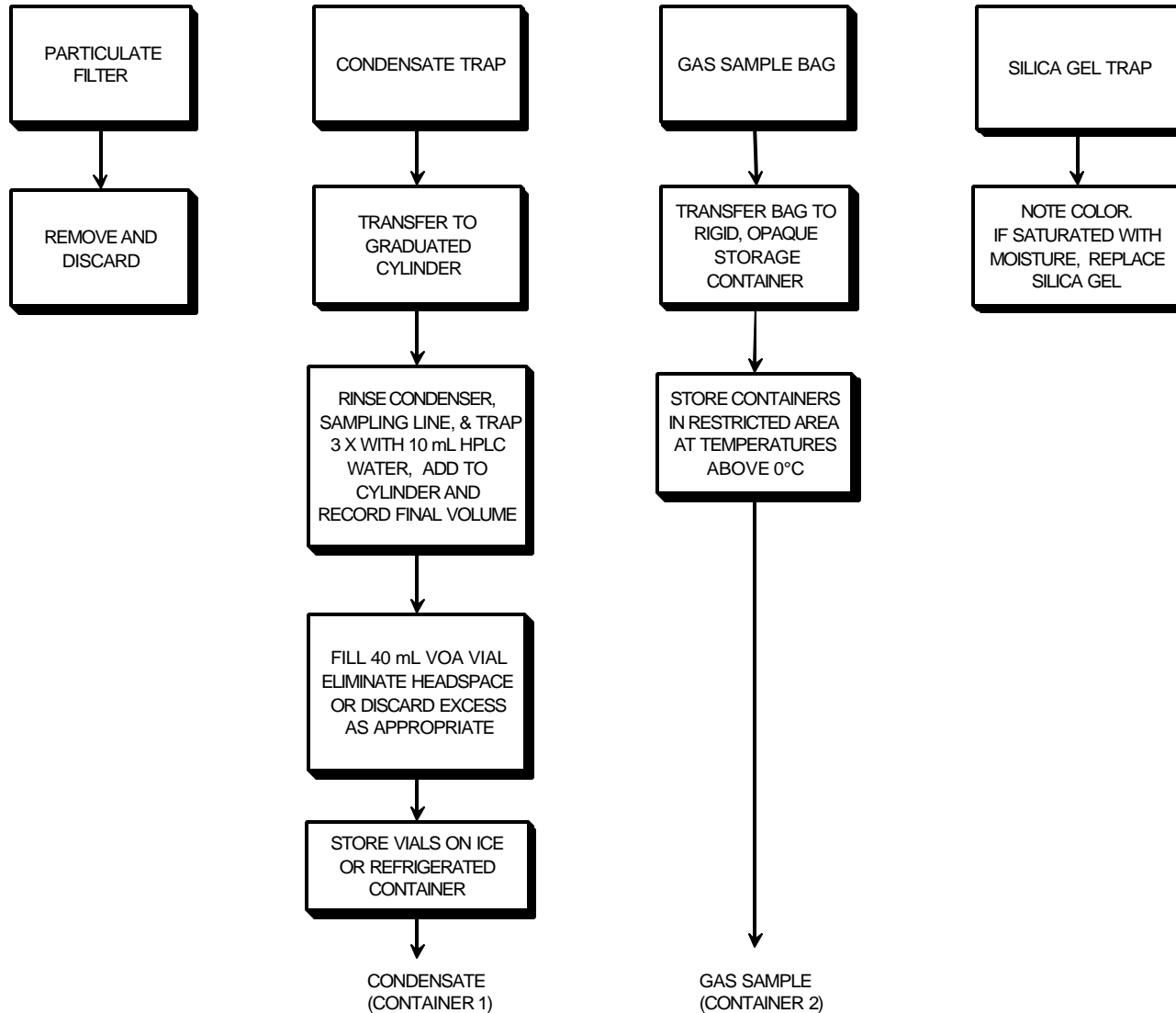


Figure 2. Method 0040 Volatile Organic Compound Sample Recovery

Table A-15. Cascade Impactor Stack Gas Particle Size Distribution Sampling Procedure

Sample name:	Stack Gas Cascade Impactor
Sampler:	Stack sampling team
Locations:	Exhaust stack
Equipment:	Cascade impactor; preweighed quartz fiber substrates; modified EPA Method 5 sampling train; petri dishes; balance; graduated cylinder.
Frequency:	Once per test run.
Procedures:	Stack gases will be sampled at a single point of average velocity. A constant sampling rate is used at near isokinetic conditions to collect particulate matter on preweighed cascade impactor substrates. The particle size distribution will be determined gravimetrically.

The sample will be withdrawn from the stack at a point of average velocity. Candidate stack sampling point locations are determined in accordance with EPA Method 1. An initial traverse is made with a pitot tube at each potential sample point location following EPA Method 2 to establish stack gas velocity profile and temperature. Stack gas mean molecular weight is determined using an Orsat analyzer per EPA Method 3 and CARB Method 501. EPA Method 4 will be followed to determine the stack gas moisture content. Following the initial traverse, a point of average velocity will be selected for collection of the particle size distribution sample. Nozzle size, sample flow rate, run time, and impactor stage configuration are determined according to CARB Method 501 procedures and equipment manufacturer's instructions.

The modified EPA Method 5 sampling train utilizes a cascade impactor with preweighed quartz fiber substrates, a condenser or impinger train immersed in an ice bath, and a drying column. EPA Method 5 procedures are used for pre-test and post-test leak checks. Prior to sampling, the impactor is inserted into the stack and is allowed to come to equilibrium temperature with the stack gas.

During sampling operations, gases are withdrawn from the stack through the cascade impactor at a constant rate. Particulate matter is inertially impacted on the preweighed substrates. A backup filter is used to collect any particulate matter that escapes the cascade impactor. A chilled condenser and a drying column are used to remove moisture from the stack gas. A dry gas meter and a calibrated orifice are used to measure the gas flow.

The sample train will be operated according to CARB Method 501 procedures and equipment manufacturer's instructions, however only a single point of average velocity will be sampled. The sampling rate will be set such that the sample is drawn into the cascade impactor within an isokinetic variation of $\pm 20\%$ at the point selected for sampling. Determination of isokinetic variation will be based on the stack gas velocity, temperature, and molecular weight determinations made during the initial stack traverse. Moisture determination will be based on the actual particle size distribution sample.

After sampling, the sampler is removed from the stack. The post-test hot leak check referenced in CARB Method 501 is not performed in order to avoid disturbing the particulate matter collected on the substrates. The impactor assembly is gently disconnected from the probe and allowed to cool. The nozzle should be covered and the impactor oriented in an upright position. The probe is disconnected from the sampling

Table A-15. Cascade Impactor Stack Gas Particle Size Distribution Sampling Procedure (Continued)

train, and the train components are transported, along with the sampler, to the sample recovery area.

After the sampler has cooled to where it can be handled without gloves, the substrates are carefully removed and placed into individually labeled petri dishes. Residue from the nozzle, body, and the inside and outside of the gas exit tube is brushed onto the substrate from the first impactor stage. Residue from the impactor walls is brushed onto the appropriate substrate. By convention, all particulate matter collected on surfaces between one substrate and another is considered to be part of the catch of the second, or lower, substrate. The individual petri dishes are then sealed. The moisture collected in the impingers (or condenser) and the drying tube is measured by weighing to the nearest 0.5 gram.

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

- References:
- CARB Method 501, Determination of Size Distribution of Particulate Matter from Stationary Sources, State of California Air Resources Board, Adopted March 23, 1988, Amended September 12, 1990.
 - EPA Methods 1, 2, 3, 4, and 5, Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.
 - Cascade impactor manufacturer's instructions.
-

Table A-15. Cascade Impactor Stack Gas Particle Size Distribution Sampling Procedure (Continued)

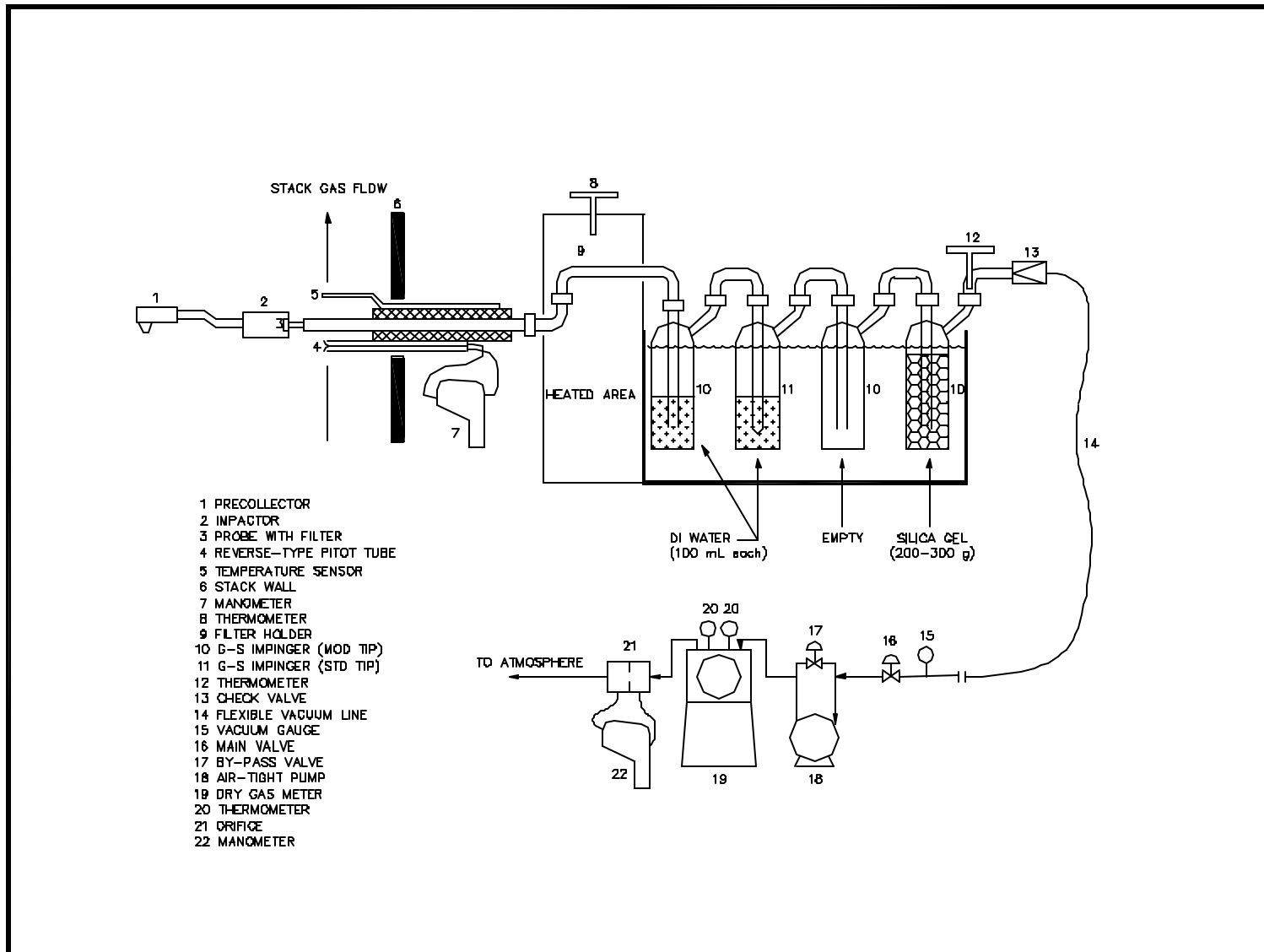


Figure 1. CARB 501 Sample Train for Particle Size Distribution

Table A-15. Cascade Impactor Stack Gas Particle Size Distribution Sampling Procedure (Continued)

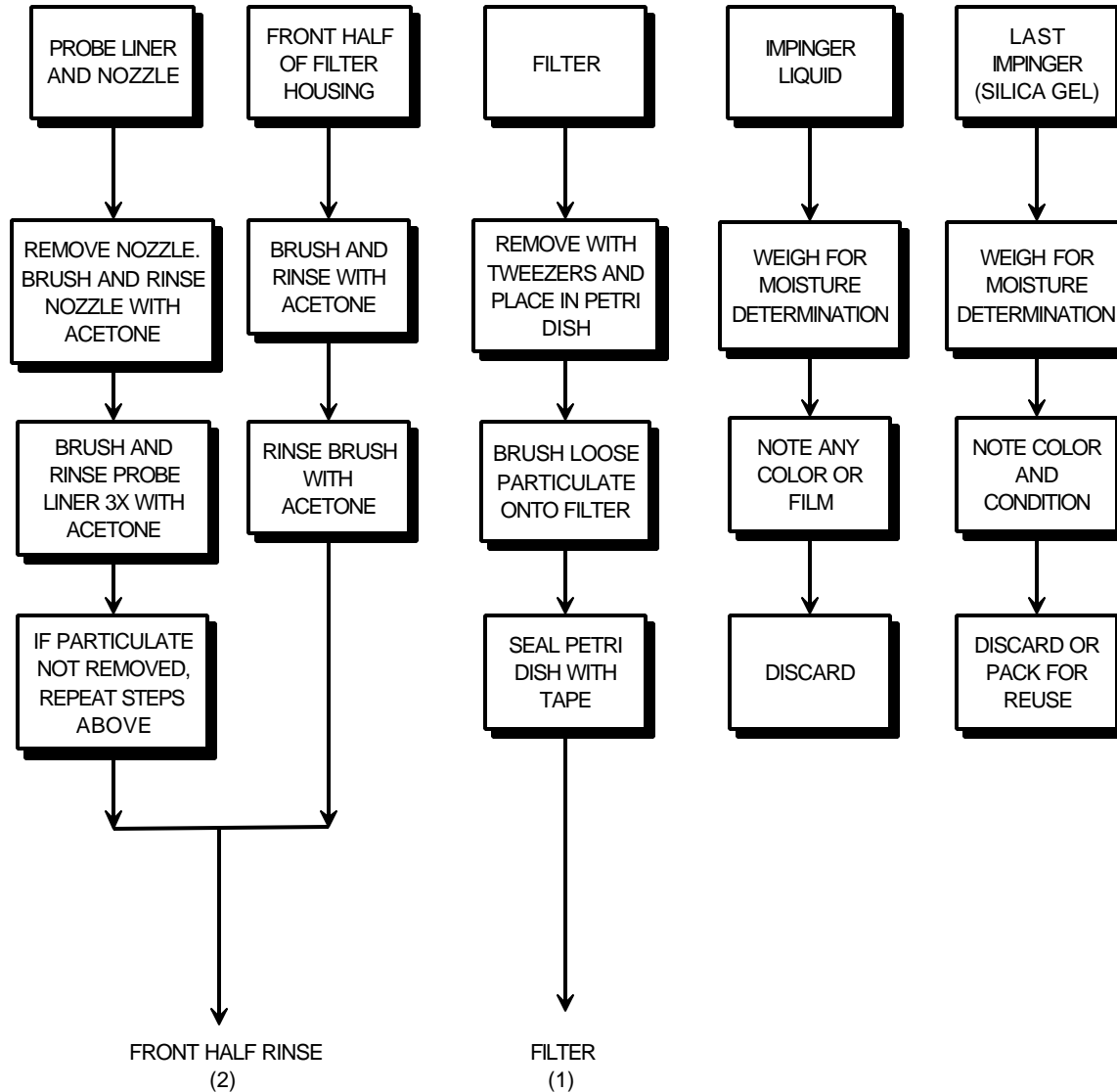


Figure 2. CARB 501 Particle Size Distribution Sample Recovery

Table A-16. Method 25A for Total Hydrocarbons using Continuous Emissions Monitor Procedure

Sample name: Stack gas THC CEMS
Inlet gas THC CEMS

Sampler: Exhaust stack: Monitoring system operator
RTO inlet duct: Stack sampling team

Components: Stack gas extraction probe, heated sample line, filter, THC gas analyzers

Locations: Exhaust stack
RTO inlet duct

Frequency: Continuous during each sampling run; three runs to complete test.

Procedures: Gas samples are drawn into the probe, transported through a heated sample line, filtered to remove particulate matter, and analyzed directly.

Both THC monitors shall be four point calibrated before and after each run using the following calibration gas concentrations:

- Zero
- 25 to 35% of instrument range
- 45 to 55% of instrument range
- 80 to 90% of instrument range.

Calibration error shall be within 5% of standard. Calibration drift during each test run shall be within 3% of span (high and low range).

References: "Method 25A – Determination of Total Gaseous Organic Concentrations using a Flame Ionization Analyzer", Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.

ATTACHMENT B
ANALYTICAL PROCEDURES

Table B-1. Analysis of Total Metals using Microwave Assisted Digestion of Sediments, Sludges, Soils, and Oils (Method 3051)

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 ($23 \pm 2^{\circ}\text{C}$ measured to $\pm 0.05^{\circ}\text{C}$) into a fluorocarbon (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 $\pm 0.05^{\circ}\text{C}$.

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 ($\pm 10\text{W}$), 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 $\pm 10\text{W}$, 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 ± 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 $170 - 180^{\circ}\text{C}$ 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

Table B-2. Analysis Multiple Metals Samples using SW-846 Method 6020 (ICP-MS)

Matrices: Spent activated carbon
Makeup water
Caustic
Scrubber blowdown
POTW discharge

Procedure Summary: **SW-846 Method 6020** Prior to analysis, the manufactures instructions should be followed to provide the appropriate operating configuration of the instrument's computer and the instrument's proper operating parameters. Allow the at least 30 minutes to equilibrate prior to analyzing and samples or standards. This must be verified by analyzing a tuning solution at least four times with relative standard deviations of $\leq 5\%$ for the analytes in the tuning solution. Mass calibration and resolution checks in the mass regions of interest are required with a requirement that the mass calibration differ no more than 0.1 amu from the true value.

To begin analysis of samples, 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. The system should be rinsed between each standard. The calibration must be verified by analyzing a calibration verification solution (ICV or CCV) immediately following the calibration. This verification solution and calibration blank should be reanalyzed after every tenth sample. When the measurement exceeds $\pm 10\%$ of the accepted value, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the new calibration verified. Any samples analyzed under out-of-control calibration must be reanalyzed. An interference check sample should also be run at the beginning of each analytical run or once every twelve hours to verify the magnitude of interferences and the adequacy of corrections.

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 (post digestion 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 at least 100 times the concentration in the method blank) 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. One dilution test must be included for each matrix in a batch.

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

Table B-3. Analysis of Mercury Samples using SW-846 Method 7470 (CVAA)

Matrices: Spent activated carbon
Makeup water
Caustic
Scrubber blowdown
POTW discharge

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.

Table B-4. Total Chlorine/Chloride Analysis

Matrices: Spent activated carbon

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.

Table B-5. Elemental Analysis of Waste Feed

Matrices:	Spent Activated Carbon
Procedure	<p>The carbon, hydrogen, oxygen, nitrogen, sulfur, ash, and moisture in the sample are determined by the procedures described in ASTM D 3176 which incorporates the following constituent analyses:</p> <p>Carbon and hydrogen are determined by burning a weighed quantity of sample and fixing the products of combustion in an absorption train, according to the procedures in ASTM D 3178. This method gives the total hydrogen content of the sample, including hydrogen due to moisture. The data will be corrected for moisture.</p> <p>Sulfur is determined by ignition and precipitation as barium sulfate according to the procedures described in ASTM Method D 3177.</p> <p>Nitrogen is determined by destructive digestion of the sample in a hot catalyzed mixture of sulfuric acid and potassium sulfate according to the procedures described in ASTM D 3179.</p> <p>Ash is determined by the weighing of the residue remaining after a rigidly controlled burning of the sample in accordance with the procedures described in ASTM Method D 3174 (Separate analytical procedure provided).</p> <p>Oxygen is determined by subtracting from 100 the sum of the other components. This method gives a total oxygen content of the sample, including oxygen due to moisture. The data will be corrected for moisture.</p> <p>Moisture is determined by establishing the loss of weight of the sample when heated under rigidly controlled conditions, in accordance with the procedures described in ASTM Method D 3173. Where volatile materials may be present in significant quantities, a Karl-Fischer (ASTM E 203) or co-distillation method will be substituted.</p>
Quality Control:	One laboratory blank will be analyzed for every sample batch.
References:	ASTM D 3176 and E 203, American Society for Testing and Materials, Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series.

Table B-6. Analysis of Volatile Organics in Solids, Semi-solids and Liquids

Matrices: Spent activated carbon
Makeup water
Caustic
Scrubber blowdown
POTW discharge

Procedure Summary: Organic and solid or semi-solid samples will be dispersed in methanol as appropriate. Aqueous samples generally do not require methanol dispersion.

Surrogates and internal standards will be added.

The sample will be processed through purge-and-trap procedures and analyzed by GC/MS according to SW-846 Method 8260.

References: Methods 5030, 5035, and 8260, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986 and Updates.

Table B-7. Analysis of Semi-Volatile Organics in Solid, Semi-solid and Liquid Samples

Matrices: Spent activated carbon
Makeup water
Caustic
Scrubber blowdown
POTW discharge

Procedure Summary: Extract solid samples using SW-846 Method 3540 or 3550 as appropriate. Extract liquid samples using SW-846 Method 3510 or 3520 as appropriate. Add internal standards and surrogates. Process through cleanup as necessary and proceed with GC/MS analysis per SW-846 Method 8270.

References: Method 8270, Semi-Volatile Organics by GC/MS, SW-846, 3rd ed., 1986 and updates.
Method 3510, Separatory Funnel Liquid-Liquid Extraction, SW-846, 3rd ed., 1986 and updates.
Method 3520, Continuous Liquid Extraction, SW-846, 3rd ed., 1986 and updates.
Method 3540, Soxhlet Extraction, SW-846, 3rd ed., 1986 and updates.
Method 3550, Ultrasonic Extraction, SW-846, 3rd., 1986 and updates.

Table B-8. Analysis of VOST (Method 0030) Samples for Volatile Organic Compounds Using Methods 5041A and 8260B

Matrices: VOST sorbent resins (Tenax and Tenax/Charcoal)

Procedure
Summary:

Instrument Calibration

The GC/MS system is hardware-tuned using the 4-bromofluorobenzene (BFB) standard to meet the criteria specified in Method 8260B (Table 4). The purge-and-trap device and the cartridge desorber are assembled and connected to the GC/MS system as specified in Methods 5030B (Section 4.6) and 5041A (Section 4.0), respectively. A methanolic solution of internal, surrogate, and initial calibration standards is prepared according to Method 8260B (Section 5.0). The calibration standards include five system performance check compounds (SPCC) and 6 calibration check compounds (CCC), and other compounds of interest as specified in the test plan. The standards are spiked onto individual Tenax® tubes and Tenax®/charcoal tubes using the flash evaporation technique described in Method 5041A (Section 7.7). For the initial calibration, the SPCCs, CCCs, and specified compounds of interest are present in five different concentrations. All Vost tubes used in the test program are from the same resin preparation batch. (Note: When the analysis of Tenax® and Tenax®/charcoal tube pairs is specified in the test plan, the calibration is performed using tube pairs.)

The GC/MS system is then calibrated by desorbing the standards from each of the VOST tubes using the same procedures as for the test samples. A 5-point calibration curve is generated and the mean response factor (RF) and relative standard deviation (RSD) of the RF are calculated for each target analyte. The mean RF of the five SPCCs and the RSD of the six CCCs are evaluated against the method criteria. The calibration curve is accepted if the SPCCs meet the minimum mean RF assigned by the method (Method 8260B, Section 7.3.5.4), and if the CCC results have an RSD less than 30%. All specified compounds of interest must have RSDs less than 15%. If the performance criteria are not met, the results are evaluated, corrective action is taken, and the calibration procedures are repeated until the criteria are met. (Note: The calibration data may also be evaluated using the one of the options described in Section 7.5 of Method 8000. The calibration curve is accepted only if a listed option is applied).

The instrument calibration is verified at the beginning of each twelve-hour shift by introducing the BFB standard, a midrange calibration standard, and a method blank. If any of the standards do not meet the criterion set forth in Method 5041A (Section 7.15), corrective action must be taken before sample analysis.

Sample Analysis

VOST tube samples are analyzed separately. VOST tube contents are spiked with surrogate and internal standards, thermally desorbed, bubbled through organic-free water, and trapped on an analytical adsorbent trap. Target analytes are identified and quantified according to the procedures in Method 8260B (Section 7.0). Samples are analyzed from lower to higher concentration. If system contamination is suspected after an unusually high recovery of an analyte, a method blank is used to establish that the system is free from contamination. If analyte recovery exceeds the calibration range, an additional calibration standard is added to bracket the point. Alternately, the analytical result may be flagged as extrapolated beyond the calibration range.

The analysis is extended to tentatively identify and quantify as many non-target analytes as possible. The tentatively identified compounds (TICs) include all non-target peaks that are at least 10% of the nearest internal standard. The identification and quantification are made using procedures in Method 8260B (Sections 7.6 and 7.7). This includes forward and reverse library searches using a response factor of 1 for the nearest internal standard, unless a specific response factor can be determined and used for quantification.

Table B-8. Analysis of Volatile Organics in VOST Samples using SW-846 Method 5041 (Continued)

Quality
Control:

Method blanks are analyzed with each set of field test samples. The blank Tenax® and Tenax®-charcoal tubes are reserved from the same resin preparation batch as the field test samples.

Field blanks are analyzed with each set of field test samples. At least one pair of field blanks is included with each six pairs of sample tubes. (Field blanks are exposed to the test environment by removing the tube end caps for the period of time required to replace one pair of test samples during a test run.)

Trip blanks are analyzed the same as field blank and test samples. At least one pair of blank tubes is included with each shipment of tubes to and from the site. (Trip blanks have not had the tube caps removed at any time during shipment or storage in the laboratory or at the test site.)

Laboratory control samples are analyzed with each analytical batch (field test, method blank, field blank, and trip blank samples) for determination of analytical accuracy.

References:

“Method 0030 - Volatile Organic Sampling Train”

“Method 5030B – Purge-and-Trap for Aqueous Samples”

“Method 5041A - Protocol for Analysis of Sorbent Cartridges from Volatile Organic Sampling Train: Wide-bore Capillary Column Technique”

“Method 8000B – Determinative Chromatographic Separations”

“Method 8260B – Volatile Organic Compounds by Gas Chromatography/mass Spectrometry (GC/MS)”

All of the above methods are from: “Test Methods for Evaluating Solid Waste”, SW-846, Third Edition, November 1986, and Updates”.

Table B-9. Analysis of Volatile Organics in VOST Condensate Samples using SW-846 Method 8260

Matrices: VOST condensate (water)

Procedure

Summary: Prior to analysis, the purge-and-trap unit should be assembled and connected as specified in Method 5030.

Prior to calibration, the system must be hardware tuned to meet the BFB criteria. Calibration of the instrument must be performed using the same introduction technique as the same. Calibration standards should be prepared from the secondary dilution standards. The initial calibration must contain at least five different calibration standards. The mean response factor (RF) and relative standard deviation (RSD) of the response factor are calibrated for each target analyte. Prior to using the calibration, the mean RF of the five system performance check compounds (SPCC) and the RSD of the six calibration check compounds (CCC) are evaluated against the method criteria. If the SPCCs do not meet the minimum assigned mean response factor or if the CCCs report an RSD greater than 30%, the curve should not be used. All target compounds must report RSDs less than 15% or one of the calibration options listed in section 7.0 of method 8000 must be applied to the compound. If one of the listed calibration options is not applied, the curve should not be used.

Calibration verification should be performed at the beginning of each twelve-hour shift. To verify the calibration, a BFB standard is introduced to the system followed by a midrange calibration standard and a method blank. If any of the standards do not meet the criterion set forth in section 7.4 of this method, corrective action must be taken prior to sample analysis.

Condensate samples contents will be spiked with surrogate and internal standards and introduced through to the GC/MS via a purge-and-trap unit. An inert gas is bubbled through a portion of the sample at ambient temperature transferring the volatile components from an aqueous phase to a vapor phase. The vapor is then swept into the sorbent column. If sample concentrations exceed the calibration range, the sample should, if sufficient volume is available, be diluted and reanalyzed. If insufficient volume is available for reanalysis, the results may be flagged as extrapolated beyond the calibration range.

Target analytes are identified and quantified according to the procedures in SW-846 Method 8260. The analysis will be extended to identify and quantify as many non-target analytes as possible. These tentatively identified compounds (TICs) will include all non-target peaks that are at least 10% of the nearest internal standard, and the identification and quantitation will be made using EPA procedures for TICs. This includes forward and reverse library searches using a response factor of 1 for the nearest internal standard, unless a specific response factor can be determined and used for quantitation.

Quality Control:

One purge-and-trap method blanks should be analyzed with each group of 20 or less sample analyzed on the same instrument during the same shift.

Field blanks should be prepared with the samples and taken to the field. The caps of the field blanks are removed in the field during recovery of the sample. At least one pair of field blanks should be included during each sampling event.

Trip blanks are treated like field blanks except that the caps are not removed. At least one pair of blank tubes must be included with the shipment of tubes to the site.

Table B-9. Analysis of Volatile Organics in VOST Condensate Samples using SW-846 Method 8260
(Continued)

Laboratory control samples (LCS) and matrix spike/matrix spike duplicates (MS/MSD) must be analyzed to determine accuracy of the analysis and should be included with each analytical batch.

References: Method 0030, Volatile Organic Sampling Train, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.
Method 8260, Volatile Organics by GC/MS, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.

Table B-10. Analysis of Particulate Matter in M5 Samples (Front Half Rinse Only)

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, desiccated 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.

Table B-11. Analysis of Hydrogen Chloride and Chlorine in M26A Samples

Matrices: Acid Impinger Liquid - sulfuric acid solution for HCl analysis (Container 3)
Alkaline Impinger Liquid - sodium hydroxide solution for Cl₂ 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 Cl₂.

References: Method 9057, Protocol for the Analysis of Samples from HCl/Cl₂ Emission Sampling Trains, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates.

Table B-12. Preparation of Method 29 or Method 0060 Stack Gas Samples for Multiple Metals Analyses (Including Mercury)

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)
- Knockout impinger contents plus nitric acid rinse (Container 5A)
- Acidified permanganate impinger solutions plus water rinse (Container 5B)
- Acidified permanganate impinger HCl rinse plus water (Container 5C)
- Acetone reagent blank (Container 7)
- 0.1N nitric acid reagent blank (Container 8A)
- Deionized water blank (Container 8B)
- Acidified H₂O₂ impinger solution blank (Container 9)
- Acidified KmnO₄ impinger solution blank (Container 10)
- 8N HCl reagent blank (Container 11)
- Particulate filter blanks (Container 12)

Procedure
Summary:

Particulate filter (Container 1) – When the determination of particulate matter (PM) is required by the test plan, the filter is first dried to constant weight and weighed to the nearest 0.1 mg following the procedures specified in Method 5.

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 HNO₃ 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. When the determination of particulate matter (PM) is required by the test plan, the residue is dried to constant weight and the results are recorded to the nearest 0.1 mg. The front-half acetone rinse residue is then dissolved with 10 mL HNO₃ and combined with the front-half nitric acid rinse in Container 3 before it is prepared for digesting.

Front-half nitric acid rinse (Container 3) – The rinse sample pH is checked and adjusted to <2 with HNO₃ 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 1A for multiple metals analysis. A 50 mL aliquot of Fraction 1A is transferred to a new sample bottle, which is labeled as Analytical Fraction 1B for mercury analysis.

Acidified Peroxide Impinger Solutions (Container 4) - The sample volume is measured to the nearest 0.5 mL and labeled Sample Fraction 2A for multiple metals analyses. A 75 to 100 mL aliquot is transferred from Fraction 2A into a new sample bottle, which is labeled Sample Fraction 2B for mercury analysis.

- Sample Fraction 2A - 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.

Table B-12. Preparation of Method 29 or Method 0060 Stack Gas Samples for Multiple Metals Analyses (Including Mercury)

- 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.
- Sample Fraction 2B – This sample is prepared and digested following the procedures in Method 7470. The sample is diluted to 100 mL with water (if needed) and transferred to a 300 mL BOD bottle. To the sample are added 5 mL of sulfuric acid, 2.5 mL of concentrated nitric acid, and 15 mL of potassium permanganate. The sample is shaken and additional potassium permanganate is added as necessary until the purple color persists for at least 15 minutes. Eight mL of potassium persulfate is added to the sample, which is then heated for 2 hours at 95°C in a hot water bath. The sample is cooled and 6 mL of sodium chloride-hydroxylamine sulfate is added to reduce the excess permanganate. After at least 30 seconds, 5 mL of stannous sulfate is added to the sample, which is now labeled as Analytical Fraction 2B. This fraction is immediately analyzed for mercury by CVASS.

Acidified Permanganate Impinger Solutions (Containers 5A, 5B, and 5C) - These samples are analyzed separately for mercury. The volumes of Containers 5A and 5B are measured to the nearest 0.5 mL and labeled Sample Fractions 3A and 3B. The content of Container 5C is measured to the nearest 0.5 mL, diluted to 500 mL with water, and the container is labeled as Sample Fraction 3C. The Sample Fractions are prepared and digested according to Method 7470 as described above for Sample Fraction 2B. After digestion and addition of reagents, the Analytical Fractions 3A, 3B, and 3C are immediately analyzed for mercury by CVASS.

Quality
Control:

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.) A separate 100 mL portion of the nitric acid blank is processed in the same manner as Container 5A to produce Analytical Fraction Blank 3A, which is analyzed for mercury. This analytical result is combined with the analytical results from Analytical Blank Fractions 2B, 3B, and 3C as described below for Containers 9, 10, and 11.

Particulate filter blank (Container 12) - A blank filter is processed in the same manner as the Container 1 sample. The filter digestate and the acetone-nitric acid digestate are combined and filtered to form Analytical Fraction Blank 1A for total metals analyses. A portion of Analytical Fraction Blank 1A is digested to produce Analytical Fraction Blank 1B for mercury analysis. The analytical results are used as the front-half blank correction values for the target metals (except mercury).

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.

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

Acidified peroxide impinger blank (Container 9) – A 200 mL portion is combined with a 100 mL portion of deionized water blank (Container 8B) to form Sample Fraction Blank 1A. A 75-100 mL aliquot of this mixture is removed to form Sample Fraction Blank 2B.

Table B-12. Preparation of Method 29 or Method 0060 Stack Gas Samples for Multiple Metals Analyses (Including Mercury)

The remaining Sample Fraction Blank 2A is digested to produce Analytical Blank Fraction 2A for total metals analyses. These analytical results are used as back-half blank correction values for total metals (except mercury). Sample Fraction Blank 2B is digested and the digestate is labeled Analytical Fraction Blank 2B for mercury analysis by Method 7470A. The analytical result is added to the sum of the analytical results from Analytical Fraction Blanks 3A, 3B, and 3C to be used as the back-half blank correction value for mercury.

Acidified permanganate impinger blank (Container 10) – A 100 mL portion is combined with 33 mL of deionized water (Container 8B) and digested in the same manner as for Sample Fraction 3B. The digestate is labeled Analytical Fraction Blank 3B for mercury analysis by Method 7470A. The analytical result is combined with the results from Analytical Blank Fractions 2A, 3A, and 3C.

HCL reagent blank (Container 11) – A 225 mL portion of Container 11 is diluted with water to 500 mL and digested in the same manner as Sample Fraction 3C. The digestate is labeled Analytical Fraction Blank 3B for mercury analysis by Method 7470A. This analytical result is added to the results from Analytical Blank Fractions 2A, 3A, and 3C, the total of which is used as the back-half blank correction value for mercury

- 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 3051 – Microwave Assisted Acid digestion of Sediments, Sludges, Soils, and Oils”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.
 - “Method 7470A – Mercury in Liquid Wastes (Manual Cold-Vapor Technique)”, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.
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Table B-12. Preparation of Method 29 or Method 0060 Stack Gas Samples for Multiple Metals Analyses (including Mercury)

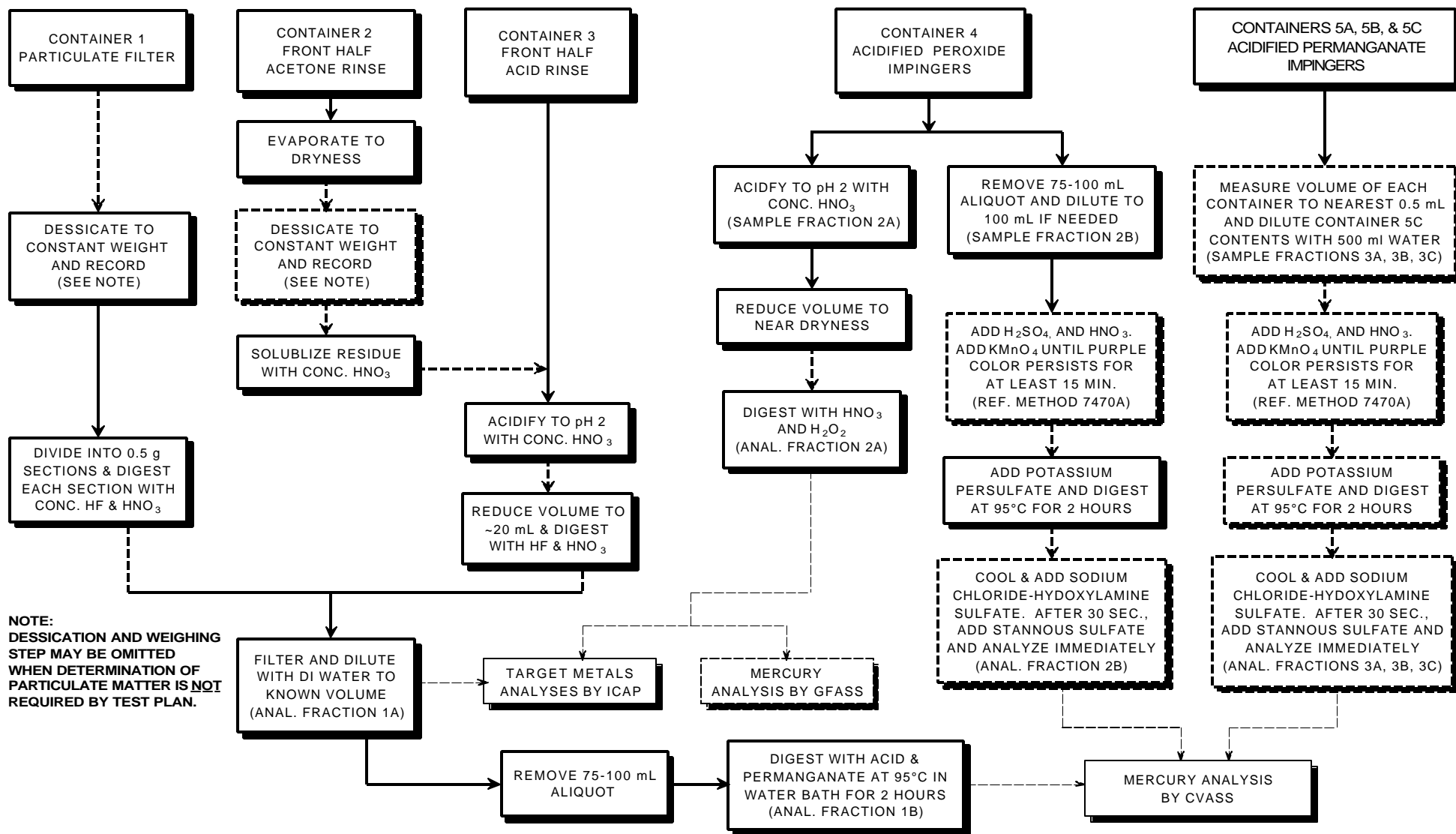


Figure 1. Preparation of Methods 29 and 0060 Stack Gas Samples for Multiple Metals Analyses (Including Hg)

Table B-12. Preparation of Method 29 or Method 0060 Stack Gas Samples for Multiple Metals Analyses (including Mercury)

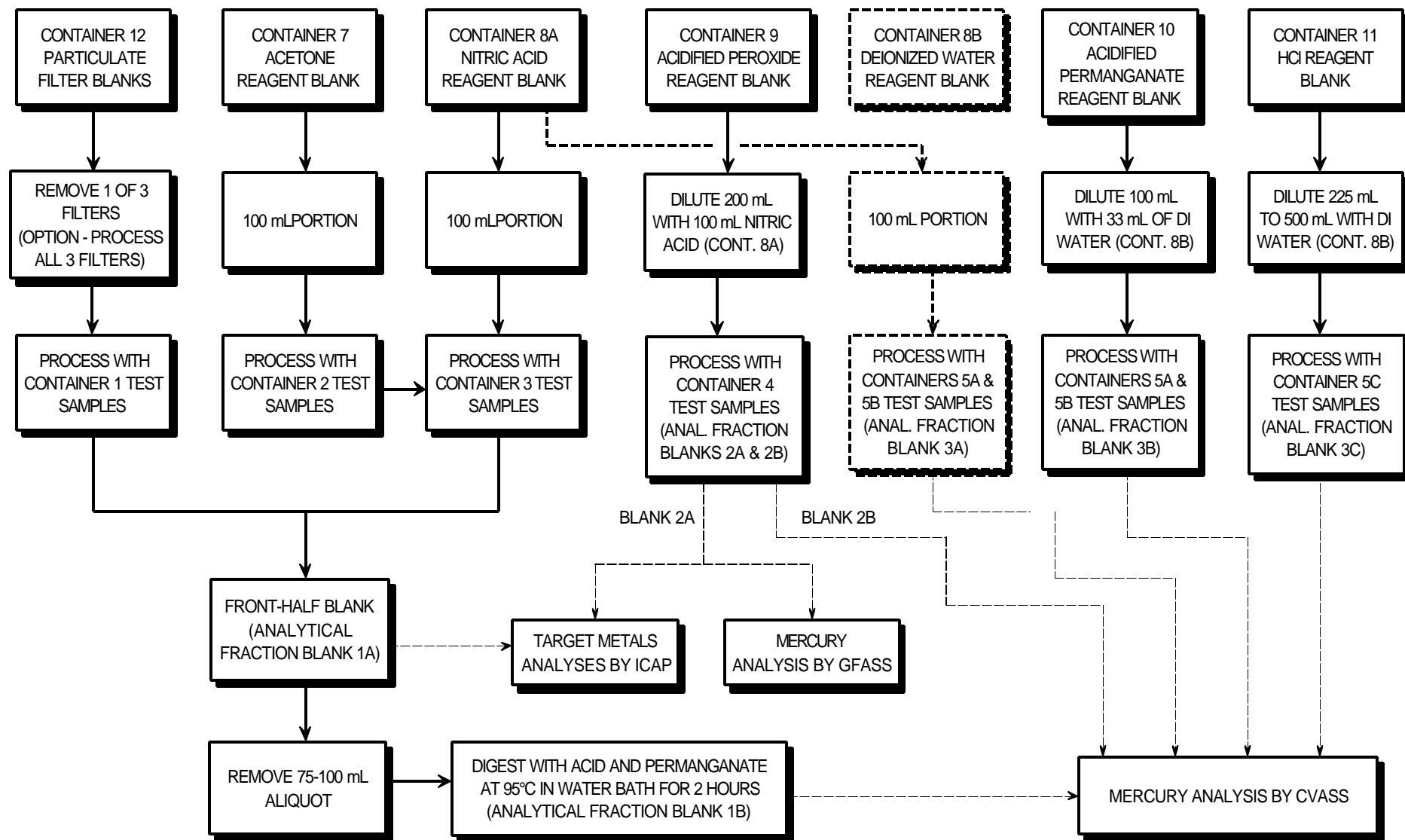


Figure 2. Preparation of Quality Control Samples for Multiple Metals Analyses (Including Hg)

Table B-13. Analysis of Multiple Metals Samples using SW-846 Method 6020 & 7470 (ICP-MS & CVAA)

Matrices:	Fractions 1A and 1B Fractions 2A and 2B Fractions 3A, 3B, and 3C Fraction Blanks 1A and 1B Fraction Blanks 2A and 2B Fraction Blanks 3A, 3B, and 3C
Procedure Summary:	<p>Fraction 1A, Fraction 2A, Fraction Blank 1A, and Fraction Blank 2A are analyzed using SW-846 Method 6020 – Inductively Coupled Plasma-Mass Spectrometry.</p> <p>Fraction 1B, Fraction 2B, Fraction 3A, Fraction 3B, Fraction 3C, Fraction Blank 1B, Fraction Blank 2B, Fraction Blank 3A, Fraction Blank 3B, and Fraction Blank 3C are analyzed using SW-846 Method 7470 – Mercury in Liquid Waste (Manual cold Vapor Technique).</p> <p>SW-846 Method 6020 Prior to analysis, the manufacturer's instructions should be followed to provide the appropriate operating configuration of the instrument's computer and the instrument's proper operating parameters. Allow the at least 30 minutes to equilibrate prior to analyzing and samples or standards. This must be verified by analyzing a tuning solution at least four times with relative standard deviations of $\leq 5\%$ for the analytes in the tuning solution. Mass calibration and resolution checks in the mass regions of interest are required with a requirement that the mass calibration differ no more than 0.1 amu from the true value.</p> <p>To begin analysis of samples, an initial calibration should be performed in accordance with the manufacturer's instructions. The calibration should consist of a minimum of a blank and a standard. The system should be rinsed between each standard. The calibration must be verified by analyzing a calibration verification solution (ICV or CCV) immediately following the calibration. This verification solution and calibration blank should be reanalyzed after every tenth sample. When the measurement exceeds $\pm 10\%$ of the accepted value, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the new calibration verified. Any samples analyzed under out-of-control calibration must be reanalyzed. An interference check sample should also be run at the beginning of each analytical run or once every twelve hours to verify the magnitude of interferences and the adequacy of corrections.</p> <p>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.</p> <p>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.</p>

Table B-13. Analysis of Multiple Metals Samples using SW-846 Method 6020 & 7470 (ICP-MS & CVAA)
(Continued)

Quality
Control:

SW-846 Method 6020

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 (post digestion 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 at least 100 times the concentration in the method blank) 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. One dilution test must be included for each matrix in a batch.

SW-846 Method 7470

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 Methods 6020 and 7470, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

Table B-14. Analysis of Hexavalent Chromium in M0061 Samples

Matrices: M0061 Sample train filtered potassium hydroxide impinger solution

Procedure Summary: The filtered potassium hydroxide absorbing solution from the sampling train impingers is analyzed for hexavalent chromium directly by ion chromatography coupled with a post-column reactor.

If increased sensitivity is required for trace levels of hexavalent chromium, a preconcentration system, utilizing a solid sorbent as described in the method referenced below, can be combined with the ion chromatograph and post-column reactor.

References: Method 7199, "Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography," SW-846, Test Methods for Evaluating Solid Waste, Third Edition, November 1986 and Updates.

Table B-15. Preparation of Semivolatile Organics and Organochlorine Pesticides in M0010 Samples using SW-846 Method 3542

Matrices:	Filter – Container No. 1 Front half rinse (acetone and methylene chloride) – Container No. 2 Adsorbent resin (XAD-2 resin)/glass wool – Container No. 3 Back half rinse (acetone and methylene chloride) – Container No. 5 Condensate and condensate rinse – Container No. 4
Procedure Summary:	<p>Extraction of the filter (Container No. 1) begins with the addition of the appropriate alternate spiking solution, and/or labeled standards uniformly to the filter while still in the petri dish. If isotopically labeled analogs are being used or a method spike is being prepared, these may also be added at this time. Using clean forceps, place the filter into a glass thimble and position the thimble in the Soxhlet extractor for extraction (Method 3540, with exceptions noted). The front half rinse is then filtered and any filtrate is added to the particulate matter filter and a piece of clean glass wool is placed over the filter. Slowly add methylene chloride to the Soxhlet extractor containing the two filters through the Soxhlet (with condenser removed), allowing the Soxhlet to cycle. Add sufficient solvent to half fill the round bottom flask and to submerge the thimble containing the filters. Extract sample for 18 hours, adjusting the mantle temperature for cycling approximately once every 30 minutes. After cooling, tilt the Soxhlet until the remaining solvent has drained to the round bottom flask. Transfer the solvent to an amber glass jar and rinse the flask three times each with approximately 10 ml of methanol and methylene chloride adding the rinses to the same amber glass jar. Store this portion at 4°C until the front half rinse extraction is completed.</p> <p>Transfer the liquid contents of the filtered front half rinse (Container No. 2) to a separatory funnel for extraction (Method 3510 with exceptions as noted). Rinse the sample container three times with methylene chloride transferring the rinse to the funnel. Add sufficient organic free water to the funnel to cause the organic and aqueous/methanol phases to separate into two distinct layers. The methylene chloride layer will be at the bottom. Continue to add water until the methylene chloride layer does not increase in volume. Add additional methylene chloride, if necessary, so that the ratio of water/methanol to methylene chloride is approximately 3:1. Add sodium hydroxide until the pH of the water layer is > 11 but <14. Shake vigorously for 2 minutes with periodic venting to release excess pressure. Allow the organic layer to separate for at least 10 minutes. Collect the methylene chloride in an amber glass bottle. Add a volume of methylene chloride (approximately equal to the volume of the first extraction) to the separatory funnel and repeat the extraction collecting the methylene chloride in the same amber glass bottle. Repeat the extraction a third time collecting the methylene chloride. Acidify the water remaining after the third extraction with sulfuric acid to a pH of <2 but > 0. Repeat the methylene chloride extraction on the acidified sample three times collecting the methylene chloride in an amber glass bottle.</p> <p>The combined filter extract, front half rinse extract, and rinses are then concentrated using a Kuderna-Danish concentrator. Pour the extracts to be condensed through a glass funnel with glass wool and methylene chloride rinsed sodium sulfate into the evaporative flask. Attach the three-ball macro Snyder column to the evaporative flask. Prewet the Snyder column by adding methylene chloride to the top. Attach the solvent vapor recovery glassware to the Snyder column of the K-D apparatus. Place the K-D apparatus in a hot water bath (80 – 85°C) so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 20 to 30 minutes. Rinse the sides of the K-D during concentration with a small volume of methylene chloride. When the volume of the liquid reaches 6-8 ml, remove the K_D apparatus from the water bath and allow the apparatus to cool and drain for at least 10 minutes. Repeat the concentration as many times as required using the same evaporative flask and systematically adding the</p>

Table B-15. Preparation of Semivolatile Organics and Organochlorine Pesticides in M0010 Samples using SW-846 Method 3542 (Continued)

remaining extract (allow sample to cool slightly before the addition of more extract) and adding new boiling chips each time.

To perform the final concentration, remove the Snyder column and evaporative flask. Add new boiling chips to the concentrator tube. Attach a two-ball micro Snyder column to the concentrator tube. Attach the solvent vapor recovery glassware to the Snyder column. Prewet the Snyder column with about 0.5 ml of methylene chloride. Place the K-D apparatus in a hot water bath. When the apparent volume reaches 4 –5 ml remove the K_D apparatus and allow to cool and drain for at least 10 minutes. If the volume is greater than 5 ml, add a new boiling chip to the concentrator tube, prewet the Snyder column, and concentrate again on the hot water bath. Wipe moisture from the outside of the concentrator tube. Transfer the extract to a calibrated vial or centrifuge tube, rinse concentrator tube with a minimum of methylene chloride, add rinses to the vial, and add methylene chloride, if necessary, to attain a final volume of 5 ml. Alternately, the final concentration may be performed by blowing the surface of the solvent with a gentle stream of nitrogen. When the nitrogen blowdown technique is used, the sides of the vessel must be carefully rinsed with a minimal amount of methylene chloride to ensure that analytes are in the methylene chloride solution and not deposited on the side of the glass container. The blowdown procedure should be performed in a calibrated vial or centrifuge tube that does not contain boiling chips. The final extract must be 5 ml. Transfer the extract to a 10-ml glass storage vial with a PTFE-lined screw cap and store at 4°C until analysis.

Condensate and condensate rinse extraction (Container No. 4) is accomplished by transferring the sample to a clean separatory funnel and rinsing the containers with three aliquots of methylene chloride. These rinses are also transferred to the separatory funnel. Appropriate alternate standard spiking solution, and/or labeled standards are then added to the liquid in the separatory funnel. If isotopically-labeled analogs are used, they may be added at this time. Add organic-free water to ensure separation of phases and perform three methylene chloride extractions as described above. After the third extraction, check the pH of the condensate/condensate rinse solution. If the pH is < 7, add acid until the pH is < 2 but > 0 and perform another methylene chloride extraction. Then make the solution basic (pH > 11 but < 14) and perform another methylene chloride extraction. Combine the methylene chloride from all pH levels, remove moisture, and concentrate for analysis (see description above). If, after the initial three methylene chloride extractions, the solution is basic, reverse the order of the acid and alkaline extractions by first adjusting the pH to >11 but < 14, performing the extraction, and then adjusting the pH acidic (pH < 2 but > 0) and performing the last extraction. Concentration is performed in the same manner as the front half extraction concentration.

To extract the XAD-2® sorbent trap (Container No. 3), the resin is transferred to the Soxhlet extractor thimble. Dry (free flowing) XAD-2® resin may be poured directly into the thimble. The trap is then rinsed with methylene chloride and the rinse added to the round bottom flask. If a ground glass stoppers are used to seal the sorbent trap during shipment, these stoppers should be rinsed with methylene chloride and the rinse added to the round bottom flask. If the XAD-2® resin is wet, removal may be difficult. Several alternatives for transfer are provided in Section 7.4.2 of this method.

With the XAD-2® in the Soxhlet extractor and the glass wool on top of the XAD-2®, add the appropriate alternate standard spiking solution, and/or labeled standards to the XAD-2®. If isotopically labeled standards are to be used or a method spike is being prepared, they should be added at this time.

The back-half rinse is then extracted in a separatory funnel (Method 3510 with exceptions as noted) in the same manner as the front half rinses. Once the separatory funnel

Table B-15. Preparation of Semivolatile Organics and Organochlorine Pesticides in M0010 Samples using SW-846 Method 3542 (Continued)

extraction is complete, the methylene chloride extract is poured through the XAD-2® in the Soxhlet extractor. The container is then rinsed three times with approximately 10 ml of methylene chloride and the rinses added to the Soxhlet. Additional methylene chloride is added to the Soxhlet, if needed, and the sample is allowed to extract for at least 18 hours but not more than 24 hours. After extraction, the sample is cooled and concentration is performed in the same manner as for the front half sample.

Prior to analysis, each of the three samples produced during this extraction procedure should be split into three equal portions. One portion will be sent to the freezer for archive, one to be used for semivolatile analysis, and one to be used for the organochlorine pesticides analysis.

Quality Control:

A method blank consist of a clean filter, clean dry XAD-2®, or organic-free reagent water, which is spiked with surrogates prior to extraction. The method blank is extracted and concentrated using the same procedures as the corresponding sample matrix. One method blank is extracted and analyzed for every ten samples.

A method spike consist of a clean filter, XAD-2®, or organic free reagent water, which is spiked with surrogates, isotopically-labeled standards, if used, and the method spike solution, if used, prior to extraction. The method spike is extracted and concentrated using the same procedure as the corresponding sample matrix. At least one method spike is extracted and analyzed for every matrix, with a frequency of one method spike for every twenty samples.

Field blanks must be submitted with the samples collected onsite. Field blanks include the sample bottles containing aliquots of sample recovery solvents, unused filters and resin cartridges.

References: Method 0010, Modified Method 5 Sampling train, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1986, and Updates

Method 3542, Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train), Test Methods for Evaluating Solid Waste, SW-846, Third Edition, December 1996, and Updates

Table B-16. Analysis of and SVOCs and OCPs in M0010 Samples
SW-846 Method 3542

Matrices: Front half rinse extract
Back half rinse extract
Condensate extract

Semivolatile Organic Analysis- Method 8270

Prior to analysis, the sample extracts should be allowed to warm to room temperature and internal quantitation standards should be added. Extracts may then be cleaned up using SW-846 Method 3640 or other appropriate cleanup method in necessary. An aliquot of extract is then injected into the GC/MS system using the same operating conditions as used for the calibration. Target analytes are identified and analyzed by gas chromatography/mass spectrometry according to the procedures in SW-846 Method 8270. The quantitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed. The analysis will be extended to identify and quantify as many non-target analytes as possible. These tentatively identified compounds (TICs) will include all non-target peaks that are at least 10% of the nearest internal standard, and the identification and quantitation will be made using EPA procedures for TICs. This includes forward and reverse library searches using a response factor of 1 for the nearest internal standard, unless a specific response factor can be determined and used for quantitation.

Method Blank

One laboratory method blank for semivolatile organics 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 traps.

Organochlorine Pesticides Method 8081

Extract Cleanup: Cleanup procedures may or may not be necessary however, specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements.

SW846 Method 3640 (Gel-permeation chromatography (GPC) cleanup) is recommended for samples of biological origin or containing high molecular weight materials. Additionally, one of the following adsorption chromatographic cleanups below may be required following the GPC cleanup. GPC is performed by packing the column with the required amount or preswelled absorbent flushed by with solvent for an extended period of time. The column is calibrated and then loaded with the sample to be cleaned up.

Table B-16. Analysis of and SVOCs and OCPs in M0010 Samples
SW-846 Method 3542

SW846 Method 3610 (alumina) may be used to remove phthalate esters. Alumina cleanup may be performed using one of three pH ranges of alumina to accomplish for different cleanup purposes. The alumina is heated until no more water is lost (typically overnight at 400 – 450°C). The alumina cleanup is accomplished using a glass chromatographic column packed with alumina or using solid-phase extraction cartridges containing alumina.

SW846 Method 3620 (florisil) may be used to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds. Florisil is a magnesium silicate with basic properties. Florosil cleanup is accomplished using a glass chromatographic column packed with florisil or using solid-phase extraction cartridges containing florisil.

SW846 Method 3630 (silica gel) may be used to separate single component organochlorine pesticides from some interferants. Silica gel cleanup may be accomplished using activated silica gel (after heating to 150 – 160°C) or deactivated with up to 10% water. This method provides the option of using either standard column chromatography techniques or solid-phase extraction cartridges for cleanup.

SW846 Method 3660 should be used to remove elemental sulfur, which interferes with the electron capture gas chromatography of certain pesticides. Sulfur cleanup may be accomplished by one of two techniques. One technique uses copper powder and the other uses tetrabutylammonium sulfite. The sample undergoing cleanup is mixed with either copper or tetrabutylammonium sulfite. The mixture is shaken and the extract is removed from the sulfur cleanup reagent.

Sample analysis Analyze the sample according to the procedures of SW-846 Method 8081. Concentrate the sample extract and inject a 2- μ L into either a single or dual column configuration. Record the volume injected to the nearest 0.05 μ L and the resulting peak size in area units. Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. Each tentative identification must be confirmed using either a second GC column of dissimilar stationary phase or using another technique such as GC/MS.

Prior to analysis, the laboratory must demonstrate acceptable performance through initial and continuing calibrations. Because several of the pesticides co-elute on any single column, the analyst should use two calibration mixtures. The specific mixture should be selected to minimize peak over lap

Method Blank

One laboratory method blank for organochlorine pesticides 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 traps.

Table B-16. Analysis of and SVOCs and OCPs in M0010 Samples
SW-846 Method 3542

- References: Method 8270, Semivolatile Organics by Gas Chromatography, Mass Spectrometry (GC/MS), Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.
- Method 3640, Gel-Permeation Cleanup, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1994, and Updates.
- Method 3610, Alumina Cleanup, Test Methods for Evaluating Solid Waste, SW846, Third Edition, December 1996, and Updates
- Method 3620, Florisil Cleanup, Test Method for Evaluating Solid Waste, SW846, Third Edition, December 1996, and Updates
- Method 3630, Silica Gel Cleanup, Test Methods for Evaluating Solid Waste, SW846, December 1996, and Updates
- Method 3660, Sulfur Cleanup, Test Methods for Evaluating Solid Waste, SW846, December 1996, and Updates
- Method 8081, Organochlorine Pesticides by Gas Chromatography, Test Methods for Evaluating Solid Waste, SW846, December 1996, and Updates

Table B-17. Preparation of Polyaromatic Hydrocarbons and Polychlorinated Biphenyls in M0010 Samples using SW-846 Method 3542

Matrices:	Filter – Container No. 1 Front half rinse (acetone and methylene chloride) – Container No. 2 Adsorbent resin (XAD-2 resin)/glass wool – Container No. 3 Back half rinse (acetone and methylene chloride) – Container No. 5 Condensate and condensate rinse – Container No. 4
Procedure Summary:	<p>Extraction of the filter (Container No. 1) begins with the addition of the appropriate alternate spiking solution, and/or labeled standards uniformly to the filter while still in the petri dish. If isotopically labeled analogs are being used or a method spike is being prepared, these may also be added at this time. Using clean forceps, place the filter into a glass thimble and position the thimble in the Soxhlet extractor for extraction (Method 3540, with exceptions noted). The front half rinse is then filtered and any filtrate is added to the particulate matter filter and a piece of clean glass wool is placed over the filter. Slowly add methylene chloride to the Soxhlet extractor containing the two filters through the Soxhlet (with condenser removed), allowing the Soxhlet to cycle. Add sufficient solvent to half fill the round bottom flask and to submerge the thimble containing the filters. Extract sample for 18 hours, adjusting the mantle temperature for cycling approximately once every 30 minutes. After cooling, tilt the Soxhlet until the remaining solvent has drained to the round bottom flask. Transfer the solvent to an amber glass jar and rinse the flask three times each with approximately 10 ml of methanol and methylene chloride adding the rinses to the same amber glass jar. Store this portion at 4°C until the front half rinse extraction is completed.</p> <p>Transfer the liquid contents of the filtered front half rinse (Container No. 2) to a separatory funnel for extraction (Method 3510 with exceptions as noted). Rinse the sample container three times with methylene chloride transferring the rinse to the funnel. Add sufficient organic free water to the funnel to cause the organic and aqueous/methanol phases to separate into two distinct layers. The methylene chloride layer will be at the bottom. Continue to add water until the methylene chloride layer does not increase in volume. Add additional methylene chloride, if necessary, so that the ratio of water/methanol to methylene chloride is approximately 3:1. Add sodium hydroxide until the pH of the water layer is > 11 but <14. Shake vigorously for 2 minutes with periodic venting to release excess pressure. Allow the organic layer to separate for at least 10 minutes. Collect the methylene chloride in an amber glass bottle. Add a volume of methylene chloride (approximately equal to the volume of the first extraction) to the separatory funnel and repeat the extraction collecting the methylene chloride in the same amber glass bottle. Repeat the extraction a third time collecting the methylene chloride. Acidify the water remaining after the third extraction with sulfuric acid to a pH of <2 but > 0. Repeat the methylene chloride extraction on the acidified sample three times collecting the methylene chloride in an amber glass bottle.</p> <p>The combined filter extract, front half rinse extract, and rinses are then concentrated using a Kuderna-Danish concentrator. Pour the extracts to be condensed through a glass funnel with glass wool and methylene chloride rinsed sodium sulfate into the evaporative flask. Attach the three-ball macro Snyder column to the evaporative flask. Prewet the Snyder column by adding methylene chloride to the top. Attach the solvent vapor recovery glassware to the Snyder column of the K-D apparatus. Place the K-D apparatus in a hot water bath (80 – 85°C) so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 20 to 30 minutes. Rinse the sides of the K-D during concentration with a small volume of methylene chloride. When the volume of the liquid reaches 6-8 ml, remove the K_D apparatus from the water bath and allow the apparatus to cool and drain for at least 10 minutes. Repeat the concentration as many times as required using the same evaporative flask and systematically adding the</p>

Table B-17. Preparation of Polyaromatic Hydrocarbons and Polychlorinated Biphenyls in M0010 Samples using SW-846 Method 3542 (Continued)

remaining extract (allow sample to cool slightly before the addition of more extract) and adding new boiling chips each time.

To perform the final concentration, remove the Snyder column and evaporative flask. Add new boiling chips to the concentrator tube. Attach a two-ball micro Snyder column to the concentrator tube. Attach the solvent vapor recovery glassware to the Snyder column. Prewet the Snyder column with about 0.5 ml of methylene chloride. Place the K-D apparatus in a hot water bath. When the apparent volume reaches 4 –5 ml remove the K_D apparatus and allow to cool and drain for at least 10 minutes. If the volume is greater than 5 ml, add a new boiling chip to the concentrator tube, prewet the Snyder column, and concentrate again on the hot water bath. Wipe moisture from the outside of the concentrator tube. Transfer the extract to a calibrated vial or centrifuge tube, rinse concentrator tube with a minimum of methylene chloride, add rinses to the vial, and add methylene chloride, if necessary, to attain a final volume of 5 ml. Alternately, the final concentration may be performed by blowing the surface of the solvent with a gentle stream of nitrogen. When the nitrogen blowdown technique is used, the sides of the vessel must be carefully rinsed with a minimal amount of methylene chloride to ensure that analytes are in the methylene chloride solution and not deposited on the side of the glass container. The blowdown procedure should be performed in a calibrated vial or centrifuge tube that does not contain boiling chips. The final extract must be 5 ml. Transfer the extract to a 10-ml glass storage vial with a PTFE-lined screw cap and store at 4°C until analysis.

Condensate and condensate rinse extraction (Container No. 4) is accomplished by transferring the sample to a clean separatory funnel and rinsing the containers with three aliquots of methylene chloride. These rinses are also transferred to the separatory funnel. Appropriate alternate standard spiking solution, and/or labeled standards are then added to the liquid in the separatory funnel. If isotopically-labeled analogs are used, they may be added at this time. Add organic-free water to ensure separation of phases and perform three methylene chloride extractions as described above. After the third extraction, check the pH of the condensate/condensate rinse solution. If the pH is < 7, add acid until the pH is < 2 but > 0 and perform another methylene chloride extraction. Then make the solution basic (pH > 11 but < 14) and perform another methylene chloride extraction. Combine the methylene chloride from all pH levels, remove moisture, and concentrate for analysis (see description above). If, after the initial three methylene chloride extractions, the solution is basic, reverse the order of the acid and alkaline extractions by first adjusting the pH to >11 but < 14, performing the extraction, and then adjusting the pH acidic (pH < 2 but > 0) and performing the last extraction. Concentration is performed in the same manner as the front half extraction concentration.

To extract the XAD-2® sorbent trap (Container No. 3), the resin is transferred to the Soxhlet extractor thimble. Dry (free flowing) XAD-2® resin may be poured directly into the thimble. The trap is then rinsed with methylene chloride and the rinse added to the round bottom flask. If a ground glass stoppers are used to seal the sorbent trap during shipment, these stoppers should be rinsed with methylene chloride and the rinse added to the round bottom flask. If the XAD-2® resin is wet, removal may be difficult. Several alternatives for transfer are provided in Section 7.4.2 of this method.

With the XAD-2® in the Soxhlet extractor and the glass wool on top of the XAD-2®, add the appropriate alternate standard spiking solution, and/or labeled standards to the XAD-2®. If isotopically labeled standards are to be used or a method spike is being prepared, they should be added at this time.

The back-half rinse is then extracted in a separatory funnel (Method 3510 with exceptions as noted) in the same manner as the front half rinses. Once the separatory funnel

Table B-17. Preparation of Polyaromatic Hydrocarbons and Polychlorinated Biphenyls in M0010 Samples using SW-846 Method 3542 (Continued)

extraction is complete, the methylene chloride extract is poured through the XAD-2® in the Soxhlet extractor. The container is then rinsed three times with approximately 10 ml of methylene chloride and the rinses added to the Soxhlet. Additional methylene chloride is added to the Soxhlet, if needed, and the sample is allowed to extract for at least 18 hours but not more than 24 hours. After extraction, the sample is cooled and concentration is performed in the same manner as for the front half sample.

Prior to analysis, each of the three samples produced during this extraction procedure should be split into three equal portions. One portion will be sent to the freezer for archive, one to be used for polyaromatic hydrocarbon analysis the polychlorinated biphenyl analysis

Quality Control:

A method blank consist of a clean filter, clean dry XAD-2®, or organic-free reagent water, which is spiked with surrogates prior to extraction. The method blank is extracted and concentrated using the same procedures as the corresponding sample matrix. One method blank is extracted and analyzed for every ten samples.

A method spike consist of a clean filter, XAD-2®, or organic free reagent water, which is spiked with surrogates, isotopically-labeled standards, if used, and the method spike solution, if used, prior to extraction. The method spike is extracted and concentrated using the same procedure as the corresponding sample matrix. At least one method spike is extracted and analyzed for every matrix, with a frequency of one method spike for every twenty samples.

Field blanks must be submitted with the samples collected onsite. Field blanks include the sample bottles containing aliquots of sample recovery solvents, unused filters and resin cartridges.

References: Method 0010, Modified Method 5 Sampling train, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1986, and Updates

Method 3542, Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train), Test Methods for Evaluating Solid Waste, SW-846, Third Edition, December 1996, and Updates

Table B-18. Analysis of PAHS and PCBS in M0010 Samples
SW-846 Method 3542

Matrices: Front half rinse extract
Back half rinse extract
Condensate extract

Polychlorinated Biphenyls Analysis- Method 1668

Sample cleanup After extraction, samples are cleaned up using back-extraction with sulfuric acid and/or base, and gel permeation, silica gel, Florisil and activated carbon chromatography.

Sample analysis After cleanup, the extract is concentrated to near dryness. Immediately prior to injection, internal standards are added to each extract, and an aliquot of the extract is injected into the gas chromatograph. The analytes are separated by the GC and detected by a high-resolution ($\leq 10,000$) mass spectrometer. Two exact m/z's are monitored for each analyte.

An individual PCB congener is identified by comparing the GC retention time and ion-abundance ratio of two exact m/z's with the corresponding retention time of an authentic standard. For PCBs with labeled analogs, the GC/MS system is calibrated, and the concentration of each compound is determined using the isotope dilution technique. For PCBs without labeled compounds, the GC/MS system is calibrated and the concentration of each compound is determined using the internal standard technique.

Method Blank

One laboratory method blank for polychlorinated biphenyls 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 traps.

Polycyclic Aromatic Hydrocarbons

Column Cleanup: Before using a procedure for the cleanup of sample extracts, the analyst must demonstrate that the requirements of Section 8.1.3.1 and 8.2.6 can be met using the cleanup procedure. Acceptable alternative cleanup procedures may also be used provided that the analyst can demonstrate that the performance requirements can be met.

Concentrate the sample extract to about 1 ml using the nitrogen blowdown apparatus, and is transferred quantitatively with hexane rinsing to at least one of the columns described below.

Silica Gel Column – Elute the column with 40 ml of hexane. The rate for all elution should be about 2 ml/min. Discard the elute and just prior to exposure of the sodium sulfate layer to the air, transfer the 1 ml sample extract onto the column using two additional 2 ml rinses of hexane to complete transfer. Just prior to exposure of the sodium sulfate layer to the air, begin elution of the column with 25 ml of hexane followed by 25 ml of methylene chloride/hexane (2:3)(v/v). Collect the entire elute. Concentrate the collected fraction to about 5 ml using the K-D apparatus or a rotary evaporator. Do not allow the extract to go to dryness.

Table B-18. Analysis of PAHS and PCBS in M0010 Samples

SW-846 Method 3542

Transfer to a minivial using a hexane rinse and concentrate to 450 μ l using a gentle stream of nitrogen. Store the extract at 4°C or lower until analysis.

Alumina Column - Elute the column with 50 ml of hexane. Let the solvent flow through the column until the head of the liquid in the column is just above the sodium sulfate layer. Close the stopcock to stop solvent flow.

Transfer 1 ml of the sample extract onto the column. Rinse out extract vial with two 1 ml rinses of hexane and add it to the top of the column immediately. Just prior to exposure of the sodium sulfate to the air, elute the column with a total of 15 ml of hexane. Collect the effluent and concentrate to about 2 ml using the K-D apparatus or a rotary evaporator.

Transfer to a minivial using a hexane rinse and concentrate to 450 μ l using a gentle stream of nitrogen. Store the extracts at 4°C or lower until analysis.

Sample analysis Approximately one hour before analysis, adjust the sample volume to approximately 500 μ l by adding 50 μ l of the recovery standard solution. If the sample volume is adjusted to achieve a desired detection limit, the recovery standard volume must also be adjusted.

Prior to analysis, the laboratory must demonstrate acceptable performance through initial and continuing calibrations. An aliquot of extract is then injected into the GC/MS system using the same operating conditions as used for the calibration. Analytes are identified by the presence of quantitation ions and the relative retention time. The relative retention time of the analyte must be within \pm 0.008 RRT units of the standards. The signal to noise ratio for the unlabeled compounds must be greater than 2.5 to 1 for the quantitation ions for HRMS and for both the quantitation and confirmation ions for LRMS.

Method Blank

One laboratory method blank for polycyclic aromatic hydrocarbons will be analyzed for every batch of 15 or fewer 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 traps.

References: Method 8270, Semivolatile Organics by Gas Chromatography, Mass Spectrometry (GC/MS), Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.

Method 3640, Gel-Permeation Cleanup, test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1994, and Updates.

Method 429, Determination of Polycyclic Aromatic Hydrocarbon (PAH) Emissions From Stationary Sources, California Environmental Protection Agency, air Resources Board (CARB), Stationary source Test Methods, Volume 3, Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources, July 28, 1997.

Table B-19. Analysis of M0023 Samples for PCDD/PCDF
(Separate Front Half and Back Half Samples)

Matrices:	Particulate Filter (Container 1) Front half rinse - acetone, methylene chloride, and toluene (Container 2) Adsorbent module (Container 3) Back half rinse -acetone, methylene chloride, and toluene (Container 4) Reagent blanks -acetone, methylene chloride, and toluene reagent blanks (archive) Adsorbent resin module trip blank (archive) Particulate filter trip blank (archive)
Procedure Summary:	<p>Exactly 1.0 mL of the surrogate spiking solution is added uniformly onto the surface of the particulate filter (Container 1). The filter is then transferred to the extraction thimble of a Soxhlet extraction system.</p> <p>The front half rinse (Container 2) is concentrated to 1-2 mL using a Kuderna-Danish (K-D) concentrator apparatus followed by nitrogen blow down at a temperature of less than 37°C. The front half rinse sample container is rinsed three times with small portions of methylene chloride and these rinses are added to the K-D concentrate and the mixture is further concentrated to near dryness. This concentrate is added to the particulate filter in the extraction thimble. Internal standards are added to the thimble and the thimble contents are extracted for 16 hours. The Soxhlet extract is concentrated to about 10 mL in a rotary evaporator and to 10 µL in a nitrogen evaporative concentrator. The concentrated extract is dissolved in 5 mL of hexane to form the front half sample (Sample 1).</p> <p>The XAD resin is transferred from the adsorbent module (Container 3) to a separate extraction thimble using toluene to rinse the glass module and the rinse catch beaker. The glass wool plug is added to the thimble.</p> <p>The back half rinse (Container 4) is concentrated to 1-2 mL using a K-D apparatus followed by nitrogen blow down at a temperature of less than 37°C. The back half rinse sample container is rinsed three times with small portions of methylene chloride and the rinses are added to the K-D concentrate and the solution is further concentrated to near dryness. The K-D concentrate is added to the thimble (which contains the resin, container rinses, and glass wool plug). Internal standards are added to the thimble and the thimble contents are extracted with toluene for 16 hours. The Soxhlet extract is then concentrated to about 10 mL in a rotary evaporator and to 100 µL in a nitrogen evaporative concentrator. The concentrated extract is then dissolved in 5 ml of hexane to form the back half sample (Sample 2).</p> <p>Sample 1 (front half) and Sample 2 (back half) are each spiked with 40 µL of alternate standards and divided into two equal portions. One portion of each sample is archived for possible repeat analysis if needed. The other portions are subjected to the chromatographic cleanup steps of Method 8290.</p> <p>Each prepared sample is spiked with the recovery standard solution and immediately analyzed by HRGC/HRMS following 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.</p>
Quality Control:	One laboratory method blank is 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 is obtained from the same batch used for preparation of the field traps.

Table B-19. Analysis of M0023 Samples for PCDD/PCDF
(Separate Front Half and Back Half Samples)

- References: "Method 0023 - Sampling Method for Polychlorinated Dibenzo-p-Dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Emissions from Stationary Sources", Test Methods for Evaluating Solid Wastes, SW-846, Third Edition, November 1986, and Updates.
- "Method 8290 - Polychlorinated Dibenzodioxin (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography, High-Resolution Mass Spectrometry (HRGC/HRMS)", Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.
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Table B-19. Analysis of M0023 Samples for PCDD/PCDF
(Separate Front Half and Back Half Samples)

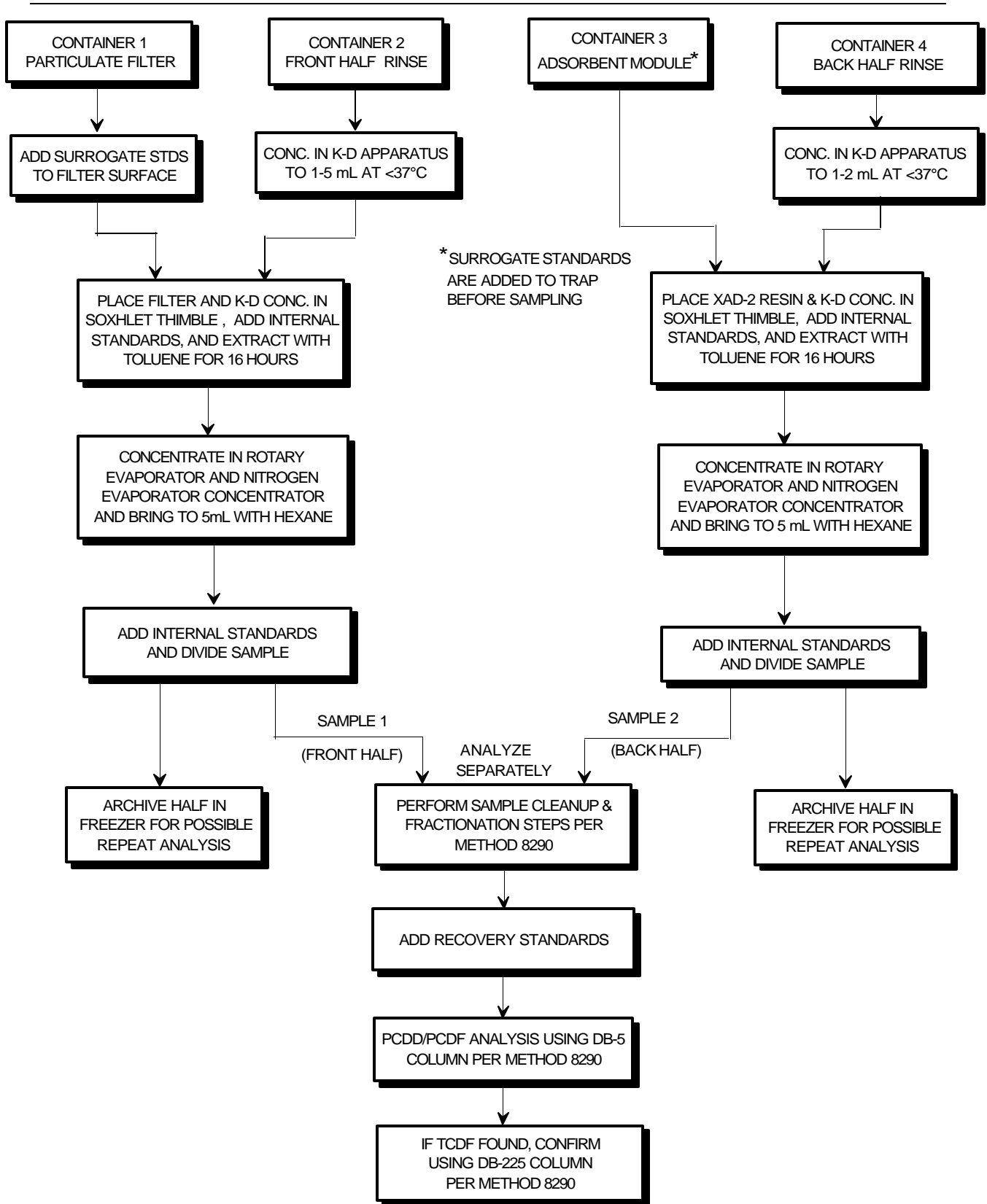


Figure 1. Recovery, Preparation, and Analysis of M0023A Samples for PCDD/PCDF

Table B-20. Preparation of SVOC and NVOC in M0010 Samples using SW-846 Method 3542

Matrices:	Filter – Container No. 1 Front half rinse (acetone and methylene chloride) – Container No. 2 Adsorbent resin (XAD-2 resin)/glass wool – Container No. 3 Back half rinse (acetone and methylene chloride) – Container No. 5 Condensate and condensate rinse – Container No. 4
Procedure Summary:	<p>Extraction of the filter (Container No. 1) begins by using clean forceps, place the filter into a glass thimble and position the thimble in the Soxhlet extractor for extraction (Method 3540, with exceptions noted). The front half rinse is then filtered and any filtrate is added to the particulate matter filter and a piece of clean glass wool is placed over the filter. Slowly add methylene chloride to the Soxhlet extractor containing the two filters through the Soxhlet (with condenser removed), allowing the Soxhlet to cycle. Add sufficient solvent to half fill the round bottom flask and to submerge the thimble containing the filters. Extract sample for 18 hours, adjusting the mantle temperature for cycling approximately once every 30 minutes. After cooling, tilt the Soxhlet until the remaining solvent has drained to the round bottom flask. Transfer the solvent to an amber glass jar and rinse the flask three times each with approximately 10 ml of methanol and methylene chloride adding the rinses to the same amber glass jar. Store this portion at 4°C until the front half rinse extraction is completed.</p> <p>Transfer the liquid contents of the filtered front half rinse (Container No. 2) to a separatory funnel for extraction (Method 3510 with exceptions as noted). Rinse the sample container three times with methylene chloride transferring the rinse to the funnel. Add sufficient organic free water to the funnel to cause the organic and aqueous/methanol phases to separate into two distinct layers. The methylene chloride layer will be at the bottom. Continue to add water until the methylene chloride layer does not increase in volume. Add additional methylene chloride, if necessary, so that the ratio of water/methanol to methylene chloride is approximately 3:1. Add sodium hydroxide until the pH of the water layer is > 11 but <14. Shake vigorously for 2 minutes with periodic venting to release excess pressure. Allow the organic layer to separate for at least 10 minutes. Collect the methylene chloride in an amber glass bottle. Add a volume of methylene chloride (approximately equal to the volume of the first extraction) to the separatory funnel and repeat the extraction collecting the methylene chloride in the same amber glass bottle. Repeat the extraction a third time collecting the methylene chloride. Acidify the water remaining after the third extraction with sulfuric acid to a pH of <2 but > 0. Repeat the methylene chloride extraction on the acidified sample three times collecting the methylene chloride in an amber glass bottle.</p> <p>The combined filter extract, front half rinse extract, and rinses are then concentrated using a Kuderna-Danish concentrator. Pour the extracts to be condensed through a glass funnel with glass wool and methylene chloride rinsed sodium sulfate into the evaporative flask. Attach the three-ball macro Snyder column to the evaporative flask. Prewet the Snyder column by adding methylene chloride to the top. Attach the solvent vapor recovery glassware to the Snyder column of the K-D apparatus. Place the K-D apparatus in a hot water bath (80 – 85°C) so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 20 to 30 minutes. Rinse the sides of the K-D during concentration with a small volume of methylene chloride. When the volume of the liquid reaches 6-8 ml, remove the K_D apparatus from the water bath and allow the apparatus to cool and drain for at least 10 minutes. Repeat the concentration as many times as required using the same evaporative flask and systematically adding the remaining extract (allow sample to cool slightly before the addition of more extract) and adding new boiling chips each time. The particulate matter filter and front half rinse extract in then combined with the other two fraction extracts for concentration.</p>

Table B-20. Preparation of SVOC and NVOC in M0010 Samples using SW-846 Method 3542
(Continued)

Condensate and condensate rinse extraction (Container No. 4) is accomplished by transferring the sample to a clean separatory funnel and rinsing the containers with three aliquots of methylene chloride. These rinses are also transferred to the separatory funnel. Add organic-free water to ensure separation of phases and perform three methylene chloride extractions as described above. After the third extraction, check the pH of the condensate/condensate rinse solution. If the pH is < 7 , add acid until the pH is < 2 but > 0 and perform another methylene chloride extraction. Then make the solution basic (pH > 11 but < 14) and perform another methylene chloride extraction. Combine the methylene chloride from all pH levels, remove moisture, and concentrate for analysis (see description above). If, after the initial three methylene chloride extractions, the solution is basic, reverse the order of the acid and alkaline extractions by first adjusting the pH to > 11 but < 14 , performing the extraction, and then adjusting the pH acidic (pH < 2 but > 0) and performing the last extraction. The condensate and condensate rinse extract is then combined with the other two fraction extracts for concentration.

To extract the XAD-2® sorbent trap (Container No. 3), the resin is transferred to the Soxhlet extractor thimble. Dry (free flowing) XAD-2® resin may be poured directly into the thimble. The trap is then rinsed with methylene chloride and the rinse added to the round bottom flask. If a ground glass stoppers are used to seal the sorbent trap during shipment, these stoppers should be rinsed with methylene chloride and the rinse added to the round bottom flask. If the XAD-2® resin is wet, removal may be difficult. Several alternatives for transfer are provided in Section 7.4.2 of this method.

The back-half rinse is then extracted in a separatory funnel (Method 3510 with exceptions as noted) in the same manner as the front half rinses. Once the separatory funnel extraction is complete, the methylene chloride extract is poured through the XAD-2® in the Soxhlet extractor. The container is then rinsed three times with approximately 10 ml of methylene chloride and the rinses added to the Soxhlet. Additional methylene chloride is added to the Soxhlet, if needed, and the sample is allowed to extract for at least 18 hours but not more than 24 hours. The XAD-2® sorbent trap and back-half rinse extract is then combined with the other two fraction extracts for concentration.

Once all three extraction fractions have been combined, a final concentration is performed. To perform the final concentration, remove the Snyder column and evaporative flask. Add new boiling chips to the concentrator tube. Attach a two-ball micro Snyder column to the concentrator tube. Attach the solvent vapor recovery glassware to the Snyder column. Prewet the Snyder column with about 0.5 ml of methylene chloride. Place the K-D apparatus in a hot water bath. When the apparent volume reaches 4 –5 ml remove the K_D apparatus and allow to cool and drain for at least 10 minutes. If the volume is greater than 5 ml, add a new boiling chip to the concentrator tube, prewet the Snyder column, and concentrate again on the hot water bath. Wipe moisture from the outside of the concentrator tube. Transfer the extract to a calibrated vial or centrifuge tube, rinse concentrator tube with a minimum of methylene chloride, add rinses to the vial, and add methylene chloride, if necessary, to attain a final volume of 5 ml. Alternately, the final concentration may be performed by blowing the surface of the solvent with a gentle stream of nitrogen. When the nitrogen blowdown technique is used, the sides of the vessel must be carefully rinsed with a minimal amount of methylene chloride to ensure that analytes are in the methylene chloride solution and not deposited on the side of the glass container. The blowdown procedure should be performed in a calibrated vial or centrifuge tube that does not contain boiling chips. The final extract must be 5 ml. Transfer the extract to a 10-ml glass storage vial with a PTFE-lined screw cap and store at 4°C until analysis.

Prior to analysis, the final extract produced during this procedure should be split into four equal portions. One portion will be sent to the freezer for archive, one portion will be

Table B-20. Preparation of SVOC and NVOC in M0010 Samples using SW-846 Method 3542
(Continued)

used for TCO GC/FID analysis and the remaining two portions will be used for duplicate GRAV analysis.

Quality
Control:

A method blank consists of a clean filter, clean dry XAD-2®, or organic-free reagent water, which is spiked with surrogates prior to extraction. The method blank is extracted and concentrated using the same procedures as the corresponding sample matrix. One method blank is extracted and analyzed for every twenty samples.

A method spike consists of a clean filter, XAD-2®, or organic free reagent water, which is spiked with surrogates, isotopically-labeled standards, if used, and the method spike solution, if used, prior to extraction. The method spike is extracted and concentrated using the same procedure as the corresponding sample matrix. At least one method spike is extracted and analyzed for every matrix, with a frequency of one method spike for every twenty samples.

Field blanks must be submitted with the samples collected onsite. Field blanks include the sample bottles containing aliquots of sample recovery solvents, unused filters and resin cartridges.

References: Method 0010, Modified Method 5 Sampling train, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1986, and Updates

Method 3542, Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train), Test Methods for Evaluating Solid Waste, SW-846, Third Edition, December 1996, and Updates

Guidance for Total Organics, EPA-68-D4-0022; EPA/600/R-96/036, November 1996

Table B-21. Analysis of Total Chromatographic Organics (TCO by GC/FID) in M0010 Samples

Matrices:	Final Extract from the M0010 train
Procedure Summary:	<p>Total Chromatographic Organic (TCO) identifies organic compounds in the M0010 extract with boiling points between 100°C and 300°C. The analysis window is established by injecting <i>n</i>-heptane (C₇) and <i>n</i>-heptadecane (C₁₇) as reference peaks between which the integration will occur. The TCO concentration is determined from the calibration curve. The calibration curve is created using hydrocarbon standards which fall within the TCO range, specifically decane (C₁₀), dodecane (C₁₂), and tetradecane (C₁₄). An integrator or GC data system is used to record the data points as they are obtained from the injection of calibration standard and the identified organics within the boiling point range are quantified and totaled to obtain the TCO portion of the total organics number. A multipoint calibration of at least three standards analyzed in duplicate are required for this analysis. The calibration must have a correlation coefficient greater than 0.97 to be acceptable.</p> <p>After the calibration, a daily quality control check is run using a standard with a concentration in the middle of the working range of the calibration curve. If control samples run in duplicate differ by more than 15% of the actual value, prepare new QC sample and repeat the test. If the new sample fails the test, determine if there is a loose column connection, septum, or altered split flow. After correction, run a new QC sample. If the new sample fails, recalibrate and or perform column change if needed.</p> <p>For sample analysis, an aliquot of this extract is injected into a capillary gas chromatograph (GC) column with a flame ionization detector (FID). The peak areas are summed over the retention time window that encompasses the boiling point range with the value determined using the established calibration curve. The reporting unit is generally in terms of μg per sample.</p>
Quality Control:	<p>A reagent blank should be run with each new lot of solvent used. If the analysis shows contamination above the detection limit, then the solvent should be distilled in glass and retested, or the reagent batch will be unacceptable for TCO analysis.</p> <p>Method blanks consist of a clean filter, clean dry XAD-2®, or organic-free reagent water. The method blank is extracted and concentrated using the same procedures as the corresponding sample matrix. One method blank is extracted and analyzed for every twenty samples.</p> <p>Field blanks must be submitted with the samples collected onsite. Field blanks include the sample bottles containing aliquots of sample recovery solvents, unused filters and resin cartridges.</p>
References:	<p>Method 0010, Modified Method 5 Sampling train, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1986, and Updates</p> <p>Guidance for Total Organics, EPA-68-D4-0022; EPA/600/R-96/036, November 1996</p>

Table B-22. Analysis of Gravimetric Organics (GRAV) in M0010 Samples

Matrices: Final Extract from the M0010 train

Procedure
Summary:

The gravimetric mass analysis is a measurement of the nonvolatile organics found in the extract of the sampling train. The range of these compounds are defined as compounds with boiling points greater than 300°C.

Prior to analysis, the aluminum weighing pans should be cleaned with methyl alcohol, methylene chloride, and finally with methyl alcohol. The pans should then be dried to a constant weight in a 105°C oven and then desiccated for a minimum of 4 hours. Samples should be weighed to a constant weight to an accuracy of ± 0.01 mg.

To perform this analysis, a 1 ml aliquot of the M0010 extract is placed in a the prepared aluminum weighing pan and allowed to dry with a cover, which allows for air circulation, on a clean piece of aluminum in a fume hood. Evaporate the sample to visual dryness and move to a desiccator for a minimum of 8 hours. Weigh the sample at approximately 4-hour intervals until three successive values differ by no more than ± 0.03 mg. If the residue weight is less than 0.1 mg, concentrate more sample in the same sample pan if available.

Quality
Control:

A method blank shall be run in duplicate for each lot of solvent or set of samples to provide a control check on the purity of the solvent and the glassware cleaning procedure. The method blank shall be prepare and concentrated in an identical manner as the samples.

An audit sample must be analyzed in duplicate with each batch of samples. The audit sample may be a solution of 100 mg of eicosane in 250 ml methylene chloride or may be any audit sample prepared in house or received from an independent laboratory containing organic compounds with chain lengths of more than 18 carbons (and boiling point above 300°C) in sufficient concentration to be determined.

Two reagent blanks must be submitted each day samples are run. Reagent blanks shall be solvent samples from the same lot used to prepare the samples and shall not be concentrated prior to analysis.

All samples shall be analyzed in duplicate. Duplicate results must agree to within 20% of the average. If this condition is not met, reanalyze the sample.

References: Method 0010, Modified Method 5 Sampling train, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1986, and Updates
Guidance for Total Organics, EPA-68-D4-0022; EPA/600/R-96/036, November 1996

Table B-23. Analysis of Total VOC in M0040 Samples

Matrices: M0040 gas sample bag
M0040 condensate

Procedure

Summary: The gas bag will be connected directly to the analytical instrument sample loop and analyzed for C₁ - C₇ hydrocarbons, organic compounds boiling in the range -160°C to 100°C by GC/FID according to the procedures in EPA Guidance for Total Organics. Details of the procedure are described in Appendices A and E.

Condensate samples will be analyzed by GC/FID directly using a purge-and-trap device. Details of the procedure are describe in Appendix B of the EPA Guidance for Total Organics.

References: Guidance for Total Organics, EPA/600/R-96/036.

ATTACHMENT C
QUALITY ASSURANCE PROJECT PLAN

**QUALITY ASSURANCE PROJECT PLAN
FOR THE
CARBON REACTIVATION UNIT
PERFORMANCE DEMONSTRATION TEST**

**Westates Carbon – Arizona, Inc.
2523 Mutahar Street
Parker, Arizona 85344**

**Revision: DRAFT
July 2002
Focus Project No. 010111**

PREPARED BY:

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1.0 QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM AND DISTRIBUTION LIST

Project: Westates Carbon – Arizona, Inc. (WCAI)

RCRA Subpart X Performance Demonstration Test

Parker, Arizona

Approved Plan Submittal Date: _____

Scheduled Test Start Date: _____

Key test Personnel Approvals and Distribution

Name/Function/Organization	Signature	Date
Drew Bolyard/Plant Manager Westates Carbon – Arizona, Inc.		
Douglas Eisner/Director of Environmental Health & Safety Westates Carbon – Arizona, Inc.		
Monte McCue/Director of Plant Operations Westates Carbon – Arizona, Inc.		
Test Coordinator Test Management Contractor		
Quality Assurance Officer Test Management Contractor		

QAPP Distribution List

Project Organization Title	Organization/Name	No. of Copies
Plant Manager	Westates Carbon – Arizona, Inc.	1
Director of Environmental Health & Safety	Westates Carbon – Arizona, Inc.	1
Director of Plant Operations	Westates Carbon – Arizona, Inc.	1
Test Coordinator	Test Management Contractor	1
Quality Assurance Officer	Test Management Contractor	1
Stack Sampling Coordinator	Sampling Contractor	1
Sample Custodian	Sampling Contractor	1
Laboratory Analysis Coordinator	Contract Laboratory	1
Tribal Representative	Colorado River Indian Tribes (CRIT)	2
U.S. Environmental Protection Agency	EPA Region IX	2

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APPENDICES

- A RESUMES OF KEY INDIVIDUALS

ACRONYMS AND ABBREVIATIONS

ASTM	American Society for Testing Materials
B.P.	Boiling Point
CAR	Corrective action request
CCV	Continuing calibration verification
CEM	Continuous emissions monitor
CEMS	Continuous emissions monitoring system
CF	Calibration factor
CLP	Contract Laboratory Program
CMS	Continuous monitoring system
COC	Chain of Custody
CRIT	Colorado River Indian Tribes
CVAA or CVAAS	Cold vapor atomic adsorption spectroscopy
DI	Deionized (water)
DQO	Data quality objective
DRE	Destruction and removal efficiency
dscf	Dry standard cubic foot
dscfm	Dry standard cubic feet per minute
dscm	Dry standard cubic meter
dscmm	Dry standard cubic meters per minute
EDL	Estimated detection limit
EPA	U.S. Environmental Protection Agency
GC/MS	Gas chromatograph/mass spectrometry
g	grams
gr	Grains
HC	Hydrocarbons
HWC	Hazardous Waste Combustor
ICP or ICAP	Inductively coupled argon plasma spectroscopy
ICP-MS or ICAP-MS	Inductively coupled argon plasma spectroscopy/mass spectrometry
ICV	Initial calibration verification
kg	Kilograms
L	Liter
LAC	Laboratory Analysis Coordinator

lb or lbs	Pounds
LCS	Laboratory control standard
MACT	Maximum Achievable Control Technology
MDL	Method detection limit
mg	Milligrams
µg or ug	Micrograms
MS	Matrix spike
MSD	Matrix spike duplicate
ND	Not Detected
ng or ng	Nanograms
OCP	Organochlorine Pesticides
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PCDD	Polychlorinated dibenzo-p-dioxin
PCDF	Polychlorinated dibenzofuran
PDS	Post-digestion spike
PDT	Performance Demonstration Test
PDTP	Performance Demonstration Test Plan
PE	Performance evaluation
PIC	Product of incomplete combustion
POHC	Principal organic hazardous constituent
ppm	Parts per million
ppmv	
or ppmv	Parts per million dry volume
QA	Quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality control
RCRA	Resource Conservation and Recovery Act
RDL	Reliable detection limit
RFA	Request for analysis
RPD	Relative percent difference
RSD	Relative Standard Deviation
RF	Response factor of Reactivation Furnace (note difference by context)
RSD	Relative standard deviation
RT	Retention time
SOP	Standard operating procedure

TCL	Target Compound List
THC	Total hydrocarbons
WCAI	Westates Carbon – Arizona, Inc.
WM	Wide-mouth
VOA	Volatile organic analysis
VOC	Volatile organic compound
VOST	Volatile organic sampling train

3.0 PROJECT DESCRIPTION

3.1 GENERAL

Westates Carbon – Arizona, Inc. (WCAI) has prepared a RCRA Part B permit application for its Carbon Regeneration Furnace located in the Colorado River Indian Tribes (CRIT) Industrial Park near Parker, Arizona. WCAI has prepared the required RCRA Subpart X Performance Demonstration Test Plan (PDTP) which is designed to demonstrate the capability of RF unit to operate within the applicable emission limitations. WCAI has also been requested by EPA Region IX to perform a site-specific multiple pathway human health risk and ecological assessment as part of the permitting process. Accordingly, WCAI has prepared the PDTP and this Quality Assurance Project Plan (QAPP) to incorporate the gathering of emissions data to demonstrate compliance with the applicable regulatory requirements and for use in the risk assessments. Specific guidance issued by EPA (Risk Burn Guidance for Hazardous Waste Combustion Facilities, EPA530-R-01-001, July 2001) has been used, along with WCAI's Risk Assessment Protocol document, to identify compounds of potential concern for the risk assessment and to select appropriate sampling and analytical techniques.

3.2 TEST SCOPE

The WCAI Performance Demonstration Test Plan has been prepared to provide comprehensive performance testing of the RF unit to demonstrate compliance with the applicable HWC MACT emission standards and to gather data for use in a site-specific risk assessment. The objectives of the PDTP are to demonstrate regulatory compliance with standards such as Destruction and Removal Efficiency (DRE) and particulate matter emission concentration, while operating at "worst case" conditions processing normal feed materials, which have been augmented with metals, chloride, etc., to establish operating conditions that will be included in the permit. More specifically, the objectives of the Performance Demonstration Test (PDT) are as follows:

3.2.1 Test Objectives

1. Demonstrate Compliance with Applicable USEPA Regulatory Performance Standards (Based on HWC MACT Standards for Existing Hazardous Waste Incinerators):
 - Demonstrate a DRE of greater than or equal to 99.99% for the selected principal organic hazardous constituents (POHCs) chlorobenzene and tetrachloroethene.
 - Demonstrate stack gas carbon monoxide concentration less than or equal to 100 ppmv, dry basis, corrected to 7% oxygen.
 - Demonstrate stack gas hydrocarbon concentration of less than or equal to 10 ppmv, as propane, dry basis, corrected to 7% oxygen.
 - Demonstrate a stack gas particulate concentration less than or equal to 34 mg/dscm (0.015gr/dscf) corrected to 7% oxygen.

- Demonstrate that the stack gas concentration of hydrogen chloride (HCl) and chlorine (Cl₂) are no greater than 77 ppmv, dry basis, corrected to 7% oxygen, expressed as HCl equivalents.
- Demonstrate that the stack gas mercury concentration is less than or equal to 130 µg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of semivolatile metals (cadmium and lead, combined) is less than or equal to 240 µg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of low volatility metals (arsenic, beryllium, and chromium, combined) is less than or equal to 97 µg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of dioxins and furans does not exceed 0.40 ng/dscm, corrected to 7% oxygen, expressed as toxic equivalents of 2,3,7,8-TCDD (TEQ). This is the applicable standard since the gas temperature entering the first particulate matter control device is less than 400°F.

2. Establish Permit Operating Limits

- Demonstrate maximum feed rate for spent activate carbon.
- Demonstrate minimum afterburner gas temperature
- Demonstrate maximum combustion gas velocity (or a suitable surrogate indicator)
- Demonstrate maximum total chlorine/chloride feed rate
- Establish a Maximum Theoretical Emission Concentration (MTEC) limit for mercury
- Demonstrate system removal efficiency (SRE) for semivolatile and low volatility metals so feed rate limits can be developed by extrapolation from test results.
- Establish appropriate operating limits for the air pollution control system components.

3. Gather Information for Use in a Site-Specific Risk Assessment

- Measure emissions of metals, including hexavalent chromium
- Measure emissions of specific volatile and semivolatile products of incomplete combustion (PICs)
- Measure emissions of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDF)
- Measure emissions of polychlorinated biphenyls (PCBs)
- Measure emissions of specific organochlorine pesticides
- Measure emissions of total volatile, semivolatile, and nonvolatile organics
- Determine the stack gas particle size distribution.

3.2.2 Test Protocol Summary

In order to accomplish the PDT objectives, (i.e., demonstrating that the unit will meet all applicable environmental performance standards) a single test condition representing “worst case” operations of minimum temperature, maximum combustion gas velocity (minimum residence time), and maximum

spent activated carbon feed rate will be performed. The test will consist of at least three replicate sampling runs.

A summary description of the testing conditions, analytical parameters, and sampling methods follows:

3.2.3 Test Condition 1 ("Worst-Case" Operations)

Sampling and monitoring protocols that will be utilized while carrying out the performance test are summarized as follows:

- Spent Activated Carbon Feed - total chlorine/chloride, elemental (C, H, N, O, S, moisture), volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Makeup Water - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Caustic feed to APC - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ag, Tl, Se, Ni, V, Zn)
- Scrubber Blowdown - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Wastewater Discharge to POTW - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Stack gas particulate, HCl, and Cl₂ using EPA Method 26A
- Stack gas target volatile organics using VOST, SW-846 Method 0030
- Stack gas target semivolatile organics and organochlorine pesticides using SW-846 Method 0010
- Stack gas PAHs and PCBs using a separate SW-846 Method 0010 sampling train
- Stack gas PCDD/PCDF using SW-846 Method 0023A
- Stack gas total volatile organics using SW-846 Method 0040
- Stack gas total semivolatile and nonvolatile organics using SW-846 Method 0010
- Stack gas metals (Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn) using EPA Method 29
- Stack gas hexavalent chromium using SW-846 Method 0061
- Stack gas particle size distribution using a cascade impactor
- Stack gas CO and O₂ by permanently installed CEM according to the protocols in the Appendix to 40 CFR 63, Subpart EEE; Performance Specification 4B of 40 CFR 60, Appendix B.
- Stack gas total hydrocarbons (as propane) by temporary CEM according to EPA Method 25A and the protocols in the Appendix to 40 CFR 63, Subpart EEE.

3.3 QUALITY ASSURANCE PROJECT PLAN SCOPE

This QAPP presents the organization, objectives, functional activities and specific Quality Assurance (QA) and Quality Control (QC) activities for the Performance Demonstration Test to be performed at the WCAI Carbon Reactivation facility near Parker, Arizona. This QAPP also describes the specific QA/QC protocols that will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory analysis during the test program. The QAPP is an integral part of the PDT plan and must be used in conjunction with the PDT plan.

All QA/QC procedures will be in accordance with applicable professional technical standards, government regulations and guidelines, and specific project goals and requirements. This QAPP has been prepared in accordance with EPA QAPP guidance documents, in particular the following:

1. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80)
2. Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January 1990.

4.0 ORGANIZATION OF PERSONNEL, RESPONSIBILITIES, AND QUALIFICATIONS

The project organization for this test is summarized in Figure 4-1.

All test activities are conducted under the overall direction of the WCAI Plant Manager, and in accordance with WCAI policies administered by the Director of Environmental Health and Safety. During the test, the WCAI Plant Manager will be responsible for ensuring that the process runs properly and that the unit achieves the desired test conditions on each test day. As such he will assign responsibilities to the unit operators. This individual will also be responsible for ensuring that all of the applicable process data are collected during each of the test runs. The WCAI Director of Plant Operations will be responsible for supervising all of the contractors associated with the program and will serve as the official communication link between WCAI and the respective contractors and regulatory observers. The contractors include the Test Coordinator, Sample Custodian, Feed Spiking Operator, Process Sampling Coordinator, Stack Sampling Coordinator, and the Laboratory Analysis Coordinator.

The WCAI Director of Environmental Health and Safety is responsible for environmental compliance activities related to the plant site. During the testing, the WCAI Director of Environmental Health and Safety and other environmental staff members will be available to lend support to the testing program where needed.

The project team consisting of the WCAI Director of Plant Operations and representatives of the contractors will implement the test programs. The Test Coordinator will be a consulting contractor who is experienced in the technical coordination and QA/QC associated with the testing of hazardous waste thermal treatment systems. The stack sampling for this project will be conducted by a contractor who is experienced in conducting the stack sampling called for in the PDT plan. Analytical services will be provided by a contract laboratory(s) experienced in the analysis of stack emissions test samples and waste samples.

The Test Coordinator is responsible for the execution of the PDT plan, the QAPP, the preparation of the Test Reports, and the interpretation of the results of the tests. During the tests, the Test Coordinator is responsible for the overall implementation of the tests. The Test Coordinator will serve as the focal point between the WCAI Plant Manager, WCAI Director of Plant Operations, and the sampling contractors on testing related matters, and will coordinate activities among various project team members. Specific responsibilities will include:

- Ensuring compliance with the PDT plan and the QAPP by all project team members during the test

- Documenting testing activities in a field logbook
- Assisting WCAI in interfacing with the regulatory observers and/or oversight contractors during the test
- Providing coordination among WCAI personnel and the sampling team during the test, especially regarding decisions to start, stop, hold or repeat sampling runs
- Providing field review of process operating logs, sample collection sheets, stack sampling logs, chain of custody forms, and request for analysis forms
- Interfacing with the Laboratory Analysis Coordinator while samples are being analyzed
- Interfacing with the Stack and Process Sampling Coordinators while the sampling data is being reduced
- Supervising production of the Test Report
- Certifying the overall PDT Results and PDT Report
- Preparing operating specifications for the systems based on the results of the test.

A Quality Assurance Officer (QAO) will be appointed whose responsibilities will include the following:

- Reviewing QA/QC activities and communicating the results of those activities to the appropriate personnel (refer to Figure 4-1)
- Making recommendations to WCAI and the Test Coordinator regarding any problems that may be detected
- Ensuring that the sample preservation and shipments are being properly monitored by the Sample Custodian and that any samples with preservation or holding time exceedances are reported to the Test Coordinator and WCAI immediately
- Ensuring that appropriate corrective actions are taken if problems are detected
- Conducting or coordinating any required audits of field, office, or laboratory procedures to ensure compliance with the QAPP
- Verifying that test data are adequately recorded and maintained and that data are properly reduced, validated, and interpreted
- Assuring all individuals included in the QAPP Distribution List receive current copies of revisions as applicable
- Performing inspections of the process equipment, process controls, process operations, data acquisition and recording systems, and sampling activities for compliance with this QAPP and the PDT plan
- Performing audits of the laboratories for compliance with this QAPP and the PDT plan
- Reviewing the stack sampling and analytical reports for completeness and accuracy
- The results of the above mentioned inspections will be documented in a written report, included in the PDT report.

A Process Sampling Coordinator will be appointed who will have overall responsibility for the collection and handling of all process related samples other than stack gas related samples. The Process Sampling Coordinator may also be the Sample Custodian and has the following responsibilities:

- Coordinating the preparation and shipment of process sampling equipment, and shipping containers to the test site
- Directing and/or participating in sampling activities
- Recording field test data required by the PDT plan or sampling methods
- Reviewing and approving sample collection sheets and field data sheets
- Documenting process sampling activities in a field logbook
- Overseeing recovery of samples and preservation of samples in the field
- Performing all QA activities required by the sampling methods
- Preparing a draft and final report of process sampling activities.
- Documenting all samples taken.

A Stack Sampling Coordinator will be appointed who will have overall responsibility for the collection and handling of all stack samples. The Stack Sampling Coordinator has the following responsibilities:

- Preparing and shipping stack sampling equipment, and shipping containers to the test site
- Preparing and calibrating stack sampling equipment
- Directing and/or participating in stack sampling activities
- Recording field test data required by the PDT plan and stack sampling methods
- Reviewing and approving stack sample collection sheets and stack sampling field data sheets
- Documenting stack sampling activities in a field logbook
- Overseeing recovery of stack samples and preservation of those samples
- Reducing stack sampling data and performing all calculations and QA activities required by the stack sampling methods
- Preparing a draft and final report of stack sampling activities.
- Notify the Sample Custodian of all samples taken.

The WCAI Process Operators will be responsible for the operation of the RF. Their duties will include:

- Maintaining the RF systems within specified target limits
- Maintaining logs of process data as required
- Downloading and providing on 3.5-inch or compact disc in Microsoft Excel or ASCII format the RF one-minute operating data to the Test Coordinator
- Collecting selected process samples
- Notifying the Sample Custodian of all samples taken.

A Sample Custodian will be responsible for handling all samples collected during the test. His/her duties will include:

- Assigning and recording sample numbers

- Preparing samples and packaging them for shipment to the laboratory
- Preparing chain of custody (COC) and request for analysis (RFA) forms for all samples
- Coordinate shipping of all samples to the laboratory.
- Monitoring the shipment of samples to the laboratory to ensure that all samples are received on schedule and with all preservation requirements being met (Any discrepancies should be immediately reported to the QAO, Test Coordinator and WCAI)

A Laboratory Analysis Coordinator (LAC) will be appointed for the laboratory that provides analytical services for the project. His/her responsibilities will include:

- Receiving, verifying, and documenting that incoming field samples correspond to the sample chain of custody information
- Notifying the Sample Custodian, QAO, Test Coordinator and WCAI of any discrepancies or problems in the chain of custody information, preservation, or sample condition
- Maintaining records of incoming samples
- Tracking samples through processing, analysis, and disposal
- Preparing QC samples for analysis during the project
- Verifying that personnel are trained and qualified in specified laboratory QC and analytical procedures
- Verifying that laboratory QC and analytical procedures are being followed as specified in the this QAPP and the laboratory specific QA/QC Plan and analytical standard operating procedures (SOPs)
- Reviewing QC and sample data during analysis and determining if repeat samples or analyses are needed
- Submitting certified QC and sample analysis results and data packages to the Test Coordinator
- Archiving analytical data.

Resumes of key individuals who will be implementing the test are presented in Appendix A.

5.0 QUALITY ASSURANCE OBJECTIVES AND QUALITY CONTROL OBJECTIVES

5.1 GENERAL

The overall quality assurance objective of this test project is identifying the complete set of data necessary to provide a complete quality assessment of the test results. These data include all the quality indicators generated during the project, and the adherence of the test data to the acceptance criteria for precision and accuracy that are used to assess the data quality. The specific quality objective is to produce a complete data set that can be used to fully assess and validate the RF's operation relative to the applicable emissions and performance standards.

The field and laboratory data obtained during this test will be reviewed by the Quality Assurance Officer, and a complete assessment of the data quality indicators will be included in the final test report. The data quality will be discussed with respect to meeting the respective data quality objectives (DQOs) and the overall project objective. Data that is outside of the target DQO limits will be evaluated relative to the impact(s) on the overall project objective of assessing the RF system's performance. This data evaluation and validation will be included in the test report.

Table 5-1 presents target DQOs for precision and accuracy for each type of analysis that will be performed during the test program. QA/QC objectives for precision, accuracy, representativeness, completeness, comparability, and sensitivity are defined in this section. Procedures and formulas for determining accuracy and precision are presented in Section 13.0 of this document. The following definitions briefly describe the meaning of each QA/QC objective:

Precision: A measure of mutual agreement among individual measurements of the same property, usually under "prescribed similar conditions." Various measures of precision exist depending on the prescribed similar conditions. If the number of samples is less than three, the precision is described as range percent or relative percent difference (RPD) from the average of replicate measured values for analysis of the same parameter. If the number of samples is three or greater, precision is best described in terms of relative standard deviation (RSD).

Accuracy: The degree of agreement of a measurement (or an average of measurements of the same parameter) X , with an accepted reference or true value, T . Accuracy is usually expressed as the difference between the two values, $X - T$, or the difference as a percentage of the reference or true value, $100(X - T)/T$, and sometimes expressed as a ratio, X/T . In some cases, accuracy is described as the percentage recovery of a known quantity of material added to a sample prior to analysis. Accuracy is a measure of the bias in a system.

Completeness: A measure of the amount of valid data obtained compared to the amount expected to be collected under normal conditions. Completeness is usually expressed as a percentage.

Representativeness: The degree to which data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, process condition, or an environmental condition.

Comparability: The confidence with which one set of data can be compared to another.

Sensitivity: The ability of a measurement system to accurately and precisely determine a desired property within the limits needed to assess the measurement result against established criteria. For this type of program the required sensitivity is a function of assessment criteria, sample size, and analytical detection limit.

5.2 PRECISION AND ACCURACY

A number of procedures will be used to meet the precision and accuracy objectives of the analytical program. All sampling and analytical activities will be conducted following referenced procedures. All reference materials used as calibration standards, surrogate compounds, or laboratory control samples will be of the highest purity commercially available. Contract Laboratory Program (CLP) compounds for matrix spikes and surrogates will be used for organic analyses by gas chromatograph/mass spectrometry (GC/MS). All spiking levels will be in accordance with the referenced methods. Table 5-2 lists the organic compounds and the applicable control limits of laboratory surrogates and field spikes to be used to spike samples. The calibration of instruments used during analysis will be verified each day that samples are analyzed as described in later sections of this QAPP. Assessment of data precision and accuracy will be accomplished by evaluating the results from multiple analyses of the same parameter, and analysis of standards, duplicates and spiked samples. Field and laboratory contamination will be assessed through the analysis of reagent, instrument, method, field and trip blanks.

Precision estimates presented in Table 5-1 represent variability for replicate measurements of the same parameters, expressed in terms of relative percent difference (RPD) for duplicate samples or relative standard deviation (RSD) for three or more measurements, as appropriate. For analyses of samples with detectable concentrations of the target analytes, precision is evaluated by conducting duplicate analyses of unspiked samples and assessing the RPD. In the evaluation of larger data sets (three or more data points), the RSD is assessed. When duplicate analyses are performed, the average of the original and duplicate results are used in test calculations. If the variance in the duplicate analyses bring into question the analytical precision, additional analyses, if allowed by the method, will be performed to better determine the actual value or to evaluate the potential reason(s) for the measurement variability.

For analytical results near the detection limit, precision can be impacted. For the cases where the original and duplicate results are a combination of detect and non-detect results at the method detection limit (MDL) where precision can not be calculated, the data will be flagged as estimated.

Accuracy values in Table 5-1 include components of both random error and bias, expressed as a percentage of the “true” or “known” value (for reference materials) or percent analyte recovery (for spiked samples). The QA/QC program will focus upon controlling measurement error within the estimated limits of measurement uncertainty, as specified in Table 5-1. It should be noted that these limits are estimates that are, in most cases, described in the referenced analytical methods or in QA/QC guidance for hazardous waste incineration. They represent the range of results that can be expected from these methods based on actual field sampling results and laboratory-based QA/QC studies. Therefore, it is reasonable to expect that the measurement errors associated with this project will be within the objectives shown in Table 5-1. QA/QC determinations which fall outside of the target range will be flagged and an assessment of the impact, if any, on the usefulness of the data and the overall results and conclusions of the test program will be provided in the Test Report. Specifically, if Matrix Spike/Matrix Spike Duplicate (MS/MSD) percent recoveries fall outside the control limits the Laboratory Control Samples (LCSs) and field blanks will be reviewed to determine the effect of the matrix on spike recovery.

If ongoing QA/QC procedures reveal that a measurement's error has exceeded the estimated data quality limits, the source of the excessive error will be identified and corrective action will be taken, as described in Section 14.0. If data fall outside the acceptable range of precision and accuracy, even after corrective action has been taken, those data points will be flagged in the final report. The precision and accuracy for those measurements will be reported as determined using the actual data. Also, alternative procedures (either sampling or analytical) may be considered and recommended if possible.

The analytical laboratory conducting the analysis of the samples will be required to have standard operating procedures (SOPs) for each analysis to be performed. The laboratory will also be required to have procedures for preparing, reviewing, modifying, and controlling distribution of analytical procedures.

5.2.1 CEMS Precision and Accuracy

The precision of the installed carbon monoxide and oxygen continuous emissions monitoring system (CEMS) analyzers will be assessed during the test using the recommended calibration gases in accordance with 40 CFR 60, Appendix B, Specification 4B. The precision of the temporary hydrocarbon analyzer to demonstrate compliance with 40 CFR 63.1203(b)(5)(ii), as allowed by 40 CFR 63.1206(b)(6) will be assessed during the test using the recommended calibration gases in accordance with 40 CFR 60, Appendix A, Method 25A. Precision will be assessed using the following equation:

$$\text{Precision (\% drift)} = \left(\frac{R_f - R_i}{\text{Span}} \right) \times 100$$

where:

R_f = Final monitor response at end of the test run

R_i = Initial monitor response at start of the test run

Span = Maximum range of the analyzer.

The accuracy of all CEMS analyzers will be evaluated during the test by the measurement of percent accuracy as defined by the equation below:

$$\text{Accuracy (\%)} = \left(\frac{R_a - R_c}{\text{Span}} \right) \times 100$$

where:

R_a = Analyzer indicated concentration of the calibration gas

R_c = Certified concentration of the calibration gas

Span = Maximum range of the analyzer.

The accuracy of oxygen and carbon monoxide CEMS analyzers will be evaluated in accordance with the CMS/CEMS Performance Evaluation Test Plan conducted prior to the test. This test will include calibration drift tests, response time tests, calibration error tests, and relative accuracy tests per the 40 CFR 60, Appendix B, Specification 4B.

5.2.2 VOST Precision and Accuracy

The Volatile Organic Sampling Train (SW-846, Method 0030) will be used to sample stack gases for the volatile POHCs and volatile PICs during the PDT. Prior to their use in the field, two pairs of the Tenax and Tenax/charcoal tubes from the batch of tubes prepared for specifically this project will be spiked with the project-specific volatile surrogates and matrix spike compounds and analyzed prior to field sample analysis. The precision assessment for VOST requires that the RPD associated with each analyte be $\leq 25\%$ for these spiked resin blanks. Additional precision data for the actual samples are obtained by calculating the RSD associated with surrogate spikes applied to each VOST sample. The variation of surrogate recoveries should be $\leq 35\%$ RSD for actual VOST analyses.

VOST accuracy is best assessed via a blind audit. The VOST audit includes VOST sampling of a compressed gas cylinder containing a mixture of volatile organic compounds in nitrogen. During the VOST audit, the VOST is operated by the same technician that will perform the stack sampling. The VOST audit kit, which includes the audit cylinder, cylinder heater and connecting adapters, is provided by the regulatory observers. Using the same VOST apparatus and VOST tubes prepared for the test, the technician will setup the audit cylinder per the accompanying directions and sample its contents. A minimum of four tube set pairs will be collected. The VOST audit samples are then submitted for analysis along with the other test VOST samples. The concentrations of the volatile organics in the cylinder determined via the VOST audit sample analyses are included in the QA/QC section of the test report. Only the concentrations of volatile compounds from the target list of volatile compounds for this test program will be included in the reported audit results.

The VOST Audit Cylinder Gas Coordinator is an independent government office located in Research Triangle Park, North Carolina. The identity of the compounds and concentrations of the compounds in the VOST audit cylinder are not known to the regulatory observers. The VOST audit kit may only be requested by the observing regulatory agency, and must be provided to the test site by the regulatory observers for sampling by the auditee. The VOST kit must remain in the custody of the regulatory observers at all times. The VOST audit kit and cylinder seals must remain intact until they are broken by the regulatory observers at the time of the on-site audit. The VOST audit procedure must be continually observed by the regulatory observers. The auditee may only sample the audit cylinder using the VOST; NO OTHER SAMPLING OF THE CYLINDER SUCH AS BAG SAMPLES IS ALLOWED. At the end of the audit, the VOST kit must be secured and retained by the regulatory observers for return to the Audit Cylinder Gas Coordinator.

The regulatory observers must forward the VOST audit results from the test report to the Audit Cylinder Gas Coordinator for review. The VOST audit is a simply pass/fail audit. The successful VOST audit will determine the concentrations of specific volatile organic compounds within 50-150% of the known value, known only by the Audit Cylinder Gas Coordinator. The Audit Cylinder Gas Coordinator will issue a pass/fail determination to the regulatory observers.

Recent VOST audit experience has shown that the VOST audit procedures must be modified slightly from the directions provided with the VOST audit kit and the way stack gas is actually sampled. Specifically, the standard sample volume per tube set is ~20 liters of stack gas. However, if similar size samples were collected from the VOST audit cylinder, the concentrations of organic analytes can saturate the tubes,

and may exceed the range of the Method 5041/6260 analysis (typically >1000 ng) and may not meet the criterion noted in the preceding paragraph. VOST audit experience has shown that this situation can usually be precluded if the VOST audit sample volumes are limited to 2.5 to 5 liters per tube set.

A more qualitative evaluation of accuracy for the VOST is prescribed in Section 7.3.7 of the Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January 1990. This reference requires that the Tenax and Tenax/charcoal VOST tubes from each set of test run tube pairs be analyzed separately to determine possible POHC breakthrough to the Tenax/charcoal tube. The analysis of a Tenax/charcoal tube should indicate less than 30% of the POHC concentration that is collected on the Tenax tube. Breakthrough of the POHC to the Tenax/charcoal tube above this level may cause loss of the desorption efficiency and result in a low bias in the analytical result. This criterion does not apply when less than 75 ng of POHC is detected on the Tenax/charcoal tube.

5.2.3 Method 0010 Semivolatile Organic Sampling Precision and Accuracy

A SW-846 Method 0010 sampling train, sometimes referred to as the Modified Method 5 (MM5) sampling train, will be used to sample the stack gas for semivolatile organic compound (SVOC) PICs including PAHs, OC Pesticides, and PCBs. Prior to use in the field, the XAD-2 resin traps for use in the Method 0010 sampling train are spiked with isotopically labeled versions of semivolatile organics noted in Table 5-2. Prior to their use in the field, two of the prepared XAD-2 resin traps from the batch of traps prepared specifically for this project will be analyzed prior to field sample analysis. The precision assessment for Method 0010 sampling train requires that the RPD associated with each semivolatile analyte be $\leq 50\%$ for the spiked semivolatile, PAH, OC Pesticides, and PCB compounds. The variation of surrogate recoveries should be $\leq 40\%$ RSD for actual Method 0010 analyses.

5.2.4 Method 0023A Dioxin/Furan Sampling Precision and Accuracy

A SW-846 Method 0023A sampling train will be used to sample the stack gas for polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/PCDFs). Prior to use in the field, the XAD-2 resin traps for use in the Method 0023A sampling train are spiked with isotopically labeled versions of the PCDD/PCDFs noted in Table 5-2. Prior to their use in the field, two of the prepared XAD-2 resin traps from the batch of traps prepared specifically for this project will be analyzed prior to field sample analysis. The precision assessment for Method 0023A sampling train requires that the RPD associated with each analyte be $\leq 30\%$ for the spiked compounds. The variation of surrogate recoveries should be $\leq 30\%$ RSD for actual Method 0023A analyses.

5.3 DETECTION LIMITS AND REPORTING

How the detection limits will be used in data reduction and reporting is also described in Section 11.3.3. For inorganic analyses and non-isotope dilution method organic analyses, the laboratory report will provide both the sample specific Method Detection Limit (MDL) and the Reliable Detection Limit (RDL). The laboratory will also provide a description detailing how each detection limit and reporting limit was derived. All non-detects for target analytes will be reported at the laboratory-determined MDL. If the analyte is detectable at some value between the MDL and RDL, the detected value will be reported and flagged as estimated. For isotope dilution organic analysis methods, the non-detects for the isotope dilution methods will be determined using the method specific SW-846 definition of an estimated detection limit (EDL) without the use of empirical factors or other mathematical manipulations specific to the laboratory. The verification of the analytical methods and MDLs for non-detect values relative the project specific matrices is discussed in the following sections. Estimates stack gas detection limits for this project are presented in Table 5-3.

5.3.1 Process Sample Properties

To demonstrate the reproducibility of the analyses of physical/chemical properties (e.g., heating value, ash content, elemental analysis, and total chlorine) in waste feed samples, analyses will include a prescribed number of duplicate analyses. Laboratory standards will be used to demonstrate the accuracy of the analytical methods applied to the project samples.

5.3.2 Process Sample Metals

A system of post-digestion spikes and matrix spikes are performed to provide appropriate and defensible reporting limits for metals in waste feed samples. Spiked samples will be spiked with metal analytes at 3 to 5 times the MDL used for the inductively coupled argon plasma spectroscopy (ICP) for non-mercury (non-Hg) metals and cold vapor atomic adsorption spectroscopy (CVAA) for mercury (Hg) to demonstrate the recovery and reproducibility of the methods.

5.3.3 Process Sample Organics

Matrix spikes are performed to provide appropriate and defensible reporting limits for organics in waste feed, caustic, makeup water, and blowdown samples. Spiked samples will be spiked with POHCs at 3 to 5 times the MDL used for the GCMS to demonstrate the recovery and reproducibility of the methods

5.3.4 Stack Gas VOST

Non-detect results for the volatile POHC are not expected for VOST samples due to the relative rates that the volatile POHC will be present in or will be spiked into the waste feeds. Therefore, performance is not based on the POHC detection limits. Should it be necessary to demonstrate that non-detect values are

appropriate, blank spike samples will be performed by the laboratory to demonstrate the recovery and reproducibility of spikes of the POHC applied to the Tenax material of each tube of a VOST tube pair.

Volatile PICs will also be assessed using the VOST. A target list of volatile organic compounds is included in this test program. Compounds on the target compound list will be reported per the standard SW-846 method using laboratory-determined MDLs. In addition to the target list of compounds, all non-target compound list peaks exhibiting a relative response greater than 10% of the nearest internal standard will be tentatively identified using a library search for all SW-846 Method 8260 analytes. TICs will be quantified based on the nearest internal standard and reported.

5.3.5 Stack Gas Dioxin/Furans

Each dioxin and furan 2,3,7,8-congener and tetra- through octa- congener group have sample-specific detection limits (EDLs). When Method 0023A stack gas samples for dioxins and furans are non-detect for a target dioxin or furan analyte, a sample-specific detection limit is calculated for that analyte. This is done by determining the HRGC/HRMS peak height of the noise or interferent in the expected region of the analyte signal. This value is typically multiplied by a factor of 2.5. The resulting EDL signal response value is then used in the sample calculation as if it were a detected value. The result is the estimated sample detection limit.

5.3.6 Stack Gas SVOCs, PAHs, OCP, and PCBs

Semivolatile organic compound (SVOC) PICs, including polynuclear aromatic hydrocarbons (PAHs), organochlorine pesticides (OCP), and polychlorinated biphenyls (PCBs), will be assessed using the Method 0010 sampling train. The sampling train XAD-2 resin traps will be pre-spiked with isotopically labeled surrogate compounds of SVOCs, PAHs, OCP, and PCBs. The prepared extracts from the sampling train will be split four ways for the separate analyses and an archive. The relative accuracy of the recovery of the surrogate spikes will demonstrate the required accuracy performance.

A target list of semivolatile organic compounds (SVOCs) are included in this test program. Compounds on the target compound list will be reported per the standard SW-846 method using laboratory-determined MDLs. In addition to the target list of compounds, all non-target compound list peaks exhibiting a relative response greater than 10% of the nearest internal standard will be tentatively identified using a library search for all SW-846 Method 8270 analytes. TICs will be quantified based on nearest internal standard and reported.

The target list of the PAH compounds includes the 20 PAH compounds included in California Air Resources Board (CARB) Method 429. The target list of the OCP compounds includes 26 of the 28 compounds listed in SW846 Method 8081. The two compounds not included for analysis as pesticides

are included in the SVOC list. The target list of the PCB compounds includes the 13 dioxin-like (coplanar) PCB compounds and the ten homologue groups (mono- through deca-CB). PAHs and PCBs will be analyzed via high resolution GC/MS (HRGC/HRMS). Like dioxins and furans, when stack gas samples are non-detect for a target PAH analyte, a sample-specific detection limit is calculated for that analyte. This is done by determining the HRGC/HRMS peak height of the noise or interferant in the expected region of the analyte signal. This value is typically multiplied by a factor of 2.5. The resulting EDL signal response value is then used in the sample calculation as if it were a detected value. The result is the estimated sample detection limit.

5.3.7 Stack Gas Metals Samples

Non-detect results may be reported for Method 29 stack gas samples for some metals. The test program includes collection of two blank Method 29 trains, one for a matrix spike sample and one for a matrix spike duplicate sample. The analysis program also includes post-digestion spikes of actual Method 29 samples. The recovery and reproducibility of these spikes and analyses will serve to prove that any non-detect values for Method 29 stack gas samples are valid. The spiked samples will be spiked with metal analytes at 3 to 5 times the MDL used for the ICP and CVAA to demonstrate the recovery and reproducibility of the preparation and analysis methods.

5.3.8 Stack Gas Hexavalent Chromium

Non-detect results may be reported for Method 0061 stack gas samples for hexavalent chromium. The test program includes the analysis of a matrix spike analyzed in duplicate. Additionally all samples are analyzed in duplicate. The recovery and reproducibility of these spikes and sample analyses will serve to prove that any non-detect values for Method 0061 stack gas samples are valid. The spiked samples will be spiked with metal analytes at 1 to 2 times the apparent concentration of the unspiked sample or at 10 times the MDL.

5.3.9 Stack Gas Chloride and Particulate

Non-detect results are expected for Method 26A stack gas chloride. The matrix spike samples of the Method 26A impinger solutions will show that any non-detect values are appropriate. Matrix spikes will be performed at levels 3 to 5 times the MDL used for the ion chromatography method to demonstrate the recovery and reproducibility of the method. Each field sample, calibration standard and QC sample will be analyzed in duplicate. For particulate analyses, duplicate measurements will entail replicate weight determinations to demonstrate reproducibility and consistency, and balance calibration standards will be used to assess analytical accuracy.

5.3.10 Stack Gas Method 0040 VOC Samples

SW-846 Method 0040 includes collection and analysis of Tedlar bag samples of the stack gas for C₁ through C₇ hydrocarbons via gas chromatograph/flame ionization detector (GC/FID) analysis. The GC/FID instrument will be setup and calibrated in the field. Bag samples will be analyzed within seventy-two hours of collection for C₁ through C₇ hydrocarbons. The GC/FID will be calibrated in accordance with Method 0040 procedures before and after analysis of the bag samples. Method blank analysis includes nitrogen Tedlar bag samples. The calibration and blank analysis results will demonstrate the required accuracy performance. Condensate samples recovered from the Method 0040 sampling will be collected in 40 ml VOA vials, topped off with deionized water (for zero head space) and submitted to the laboratory for analysis with 14 days of collection.

5.3.11 Stack Gas Method 0010 TCO/Grav Samples

A variation of the SW-846 Method 0010 sampling train [Modified Method 5 (MM5)] sampling train will be used to sample the stack gas for total semivolatile organic compounds (Boiling Points from 100°C to 300°C) and non-volatile organic compounds (Boiling Points greater than 300°C). The XAD-2 resin traps used in this sampling train do not have isotopically labeled surrogates added before their use in the field. The sample extraction process also excludes the use of isotopically labeled surrogates. The extracts of the pooled components of the sampling train are used to determine the Total Chromatographable Organics (TCO) via GC/FID analysis in the laboratory. The marker compounds are n-heptane and n-heptadecane because their boiling points are 98°C and 302°C, respectively. The non-volatile organics are determined by a gravimetric procedure known as Grav from the same pooled extracts of the Method 0010 train components as the semivolatile organic components. Because there are no isotopically labeled surrogates used within the sampling and analysis regime of this method, the only accuracy measurement for this sampling method is the blank train samples prepared and analyzed in the same manner as the actual field samples. The TCO/Grav blank train results will be used to blank correct the test TCO/Grav results.

5.3.12 Cascade Impactor PSD

A cascade impactor with quartz fiber substrates is used to collect samples for particle size distribution via gravimetric means. The size distribution of the particulate captured on the individual substrates is determined by drying to a constant weight and measuring the change in weight.

5.4 COMPLETENESS

Data completeness represents the percentage of valid data collected from the total number of valid tests conducted. Completeness is usually expressed as a percentage and calculated based on the number of samples reaching the laboratory for analysis. The completeness objective for the test will be met (100%

completeness) if three valid test runs are obtained for each test condition. Samples resulting from test runs that are judged invalid based on field performance indicators or aborted runs will not be submitted to the laboratory for analysis. Because the possibility exists that a sample may be lost or broken, the data from each individual analytical parameter may not be 100 percent complete for all test runs. The impact of any occurrence of sample loss will be assessed with regard to the objective of obtaining valid runs and will be discussed in the test report. The completeness objective of this test program is to generate sufficient data for the regulatory agencies to judge the performance of the system. An overall completeness objective is to meet at least 90% of the total data quality objectives established in the QAPP. Contingency samples have been incorporated into the sampling design in an effort to gather complete data. Archive samples of most feeds and residues are collected as a contingency for breakage or analytical difficulties.

5.5 REPRESENTATIVENESS, SENSITIVITY AND COMPARABILITY

The sampling procedures chosen for the test are, wherever possible, approved EPA or American Society for Testing Materials (ASTM) sampling methods that are typically employed on incinerator tests. The use of standard sampling methods affirms sample representativeness.

Sensitivity for this test is a function of the sample matrix, the sample size, and the analytical detection limit. The sample sizes chosen for each sample matrix are such that the collected sample is greater than the sample volume/mass required for each analytical method to obtain an acceptable quantitation limit for the project. Calculations have been provided as part of the PDT plan to indicate that the selected sample sizes and analytical methods are appropriate for critical test determinations.

Use of standard, approved sampling and analysis methods, standardized data reduction procedures, and QC samples will provide data that is technically defensible and is comparable from test run to test run, test condition to test condition.

6.0 SAMPLING AND MONITORING PROCEDURES

6.1 GENERAL

The objectives of this test program are the collection of representative feed, process, and stack gas samples that will demonstrate compliance of the RF system with the applicable performance and emissions standards, and provide emissions data for the post-test risk assessment conducted under RCRA Omnibus authority [40 CFR 270.32(b)(2)]. To meet these objectives requires minimizing the potential sources sample contamination or bias imparted to the samples by the sampling equipment, ambient conditions, handling, and preservation. The test program samples will be collected using the methods summarized in Table 6-1. The total numbers of field samples expected to be generated during the WCAI system tests are summarized in Tables 6-2. Sampling procedures are included in Attachment A to the Performance Demonstration Test Plan and are incorporated here by reference.

Guidelines followed to determine sampling equipment to be used, sampling points, and the frequency at which samples are to be taken are presented in Section 5.0 of the PDT plan, and are incorporated here by reference. The reference sources for the standard sampling method references include: Appendix A to 40 CFR 60, *Test Methods and Procedures, New Source Performance Standards*, 40 CFR 60 (EPA); *Test Methods for Evaluating Solid Waste, SW-846*, Third Edition, 1986 and updates (SW-846); and the *American Society for Testing and Materials (ASTM) Annual Book of ASTM Standards*. Regulatory observer approval will be requested if significant deviations from planned procedures are encountered during the testing.

All stack sampling equipment and glassware will be prepared prior to the test according to the method specifications. Following each run, the samples will be recovered from the trains. The sample recovery procedures include prescribed rinses of the trains, which serve a dual purpose of sample recovery and decontamination of the train in preparation for the next run. Rinses that are not included in the sample recovery will be placed into a waste solvent container and disposed of by WCAI.

Process samples will be collected using dedicated sampling equipment (scoops, jars, etc.) at each sampling location, thus eliminating the potential for cross contamination from one sample matrix to another. New sampling containers are used for each test run. If the same equipment will be used for more than one run, the equipment will be decontaminated by thorough washing with detergent, water and, any additional rinses required by the specified sampling and analytical protocol for which the equipment will be used. Any decontamination solution generated, will be collected by the facility operators for proper disposal.

During the test program, the RF system will be operated and tested at the conditions specified in the respective PDT plan. The following samples will be collected during the test:

- Feed Samples:
 - Spent activated carbon
- Process Samples:
 - Makeup water
 - Caustic
 - Scrubber blowdown
 - POTW discharge
- Stack Gas Samples
 - SW-846 Method 0030 VOST for Volatile POHCs and PICs
 - SW-846 Method 0023A for PCDD/PCDFs
 - EPA Method 29 for Metals
 - EPA Method 0061 for Hexavalent Chromium
 - EPA Method 26A for Particulate, HCl and Cl₂
 - SW-846 Method 0010 for SVOC and PICs and OCP
 - SW-846 Method 0010 for PAH and PCB
 - SW-846 Method 0010 for TCO/Grav Organics
 - SW-846 Method 0040 for Total Volatiles
 - CARB 501 for Particle Size Distribution (PSD)
 - Temporary CEMS for THC (EPA Method 25A)
 - Installed CEMS for Carbon Monoxide and Oxygen

Sample tracking is documented using unique sample numbering applied to every sample (refer to Figure 7-5), completed sample collection forms, completed chain of custody (COC) forms, completed request for analysis (RFAs) forms, and sample collection checklists (Figures 7-1, 7-2, and 7-3).

6.2 FIELD QUALITY CONTROL SAMPLES

Field QC samples will be collected during the test to provide an indication of quality assurance for the test samples. The field QC samples include: spiked resin blanks for VOST, Method 0023A and Method 0010 for SVOC and PICs, PAH, and PCB; field and trip blanks for VOST; reagent blanks for all sampling trains; and blank trains for Method 29, Method 0023A, Method 0010 for SVOC and PICs, PAH, OCP, and PCB, and Method 0010 TCO/Grav. Table 6-2 includes the field QC samples that will be collected.

6.2.1 Spiked Resin Blanks

During the preparation of the Tenax and Tenax/Charcoal tubes sets for the test program, two VOST Tenax and Tenax/charcoal resin tube pairs will be spiked with standard EPA contract laboratory program (CLP) surrogate spike compounds. These samples will be analyzed to determine by the VOST tube preparation laboratory to demonstrate the resin is free of background contamination, and to confirm that efficient surrogate recoveries are achievable.

Two XAD-2 resin traps prepared for the Method 0010 SVOC/OCP and PAH/PCB sampling trains will be spiked with sampling surrogates and internal standards. These samples will be extracted and analyzed for SVOCs, PAHs, OCP, and PCBs by the XAD-2 trap preparation laboratory to demonstrate the resin is free of background contamination, and to confirm that efficient surrogate recoveries are achievable.

Two XAD-2 resin traps prepared for the Method 0023A sampling train will be spiked with sampling surrogates and internal standards. These samples will be extracted and analyzed for dioxins and furans by the XAD-2 trap preparation laboratory to demonstrate the resin is free of background contamination, and to confirm that efficient surrogate recoveries are achievable.

Two unspiked XAD-2 resin traps prepared for the Method 0010 TCO/Grav train will be extracted and analyzed by the XAD-2 trap preparation laboratory to demonstrate the resin is free of background contamination.

6.2.2 Feed and Process Samples

Standard methods, procedures, and dedicated sampling equipment will be used for the collection of process samples associated with this test program. The Process Sampling Coordinator monitors the process sampling during the testing to assure that proper documentation is completed and that adherence to prescribed sampling procedures is observed. Sample tracking is documented using the sample numbering system (Figure 7-5), completed RFAs and COCs, the field sampling record, and sample collection checklist (Figures 7-1, 7-2, and 7-3).

6.2.3 Stack Gas Samples

During the test program, the Stack Sampling Coordinator and the Sample Custodian are responsible for monitoring the sampling team's adherence to the standard stack sampling procedures, especially sampling train preparation; leak checks and recoveries (including blank trains); and reagent, field, and trip blanks. The Stack Sampling Coordinator is responsible for operation and recovery of the stack sampling equipment and stack gas samples. The Sample Custodian is responsible for preparing the stack gas samples for shipment to the laboratory. Sampling train calibration procedures are discussed in Section 8.0.

EPA Methods 1 and 2 will be used to determine the number and location of sampling traverse of isokinetic sampling locations. Documentation of the Methods 1 and 2 will be included in the stack sampling report.

During each test run, Tedlar bag samples of stack gas will be collected for determining the stack gas carbon dioxide and oxygen concentrations using EPA Method 3 (Orsat Analysis). The stack gas carbon dioxide and oxygen concentrations are used to determine the stack gas molecular weight. The Tedlar bag samples may be collected from the exhaust of one of the isokinetic sampling trains or using a separate impinger and vacuum pump setup. Carbon dioxide and oxygen calibration gases will be used as reference standards for the Orsat apparatus. As an alternate to Orsat analysis, calibrated CEMS may be used for oxygen and carbon dioxide determination.

Stack gas moisture content will be determined for each isokinetic sampling train via EPA Method 4 (sampling train moisture gain). Isokinetic sampling trains silica gel impingers will be filled with fresh, dry indicating silica gel at the beginning of the test program. During the sampling train recovery process, and subsequent test runs, each indicating silica gel impinger will be inspected prior to reuse to verify that sufficient capacity remains for moisture absorption during the next test run. Silica gel more than 50% utilized will be discarded and the impinger recharged with fresh dry indicating silica gel.

6.2.3.1 VOST

The VOST will be used to sample stack gas for the selected volatile POHC and volatile PICs. The VOST data will be used to assess POHC DRE and volatile PIC emissions. The VOST sampling apparatus will be inspected and leak checked prior to each test run. Four pairs of VOST tubes will be used during each sampling run to sample a nominal 20 liters of stack gas per tube set. Three of the four pairs will be analyzed by Method 5041A/8260B. The fourth set pair will serve as an archive set in the event of sample breakage during shipping and handling. During the analysis, the stack gas and VOST audit Tenax and Tenax/charcoal tube sets will be analyzed separately to assess breakthrough. Field and trip blank Tenax and Tenax/charcoal tube sets are also analyzed separately.

6.2.3.2 Method 0010 for SVOC and PICS, PAHs, OCPs, and PCBs

Two Method 0010 sampling trains will be used to sample stack gas semivolatile organic compound (SVOC) PICs including polynuclear aromatic hydrocarbons (PAHs), organochlorine pesticides (OCP), and polychlorinated biphenyls (PCBs). During each test run, the Method 0010 sampling trains will be assembled and leak checked. The sampling train will be operated to sample a minimum of three dry standard cubic meters of stack gas during each sampling run. The sampling time to achieve the target sample volume will depend upon the probe nozzle size and sampling rate. However, sampling rate will

not exceed one dry standard cubic foot per minute. At the end of each run, the sampling train will be disassembled and all train samples collected.

The Method 0010 sampling train components will be prepared for analysis following SW-846 Method 3542. Method 3542 results in three separate fractions for analysis of the sampling train. Surrogate compounds discussed below are applied to each fraction.

The first fraction, the front-half fraction, consists of the solvent probe rinses and the particulate filter. The front half samples will be combined. Surrogate compounds for the SVOCs, PAHs, OCPs, and PCBs are spiked directly onto the filter before the Soxhlet extraction. The sample is then Soxhlet extracted for 18 hours using methylene chloride.

The second fraction consists of the XAD-2 resin and condenser rinse. The XAD-2 resin and condenser rinse samples will be combined. Surrogate compounds for the SVOCs PAHs, OCPs, and PCBs are spiked directly onto the XAD-2 resin before the Soxhlet extraction. The sample is then Soxhlet extracted for 18 hours using methylene chloride.

The third fraction consists of the condensate impinger catch and rinses. The condensate impinger catch is volumetrically or gravimetrically measured in the field, and this information is added to the stack sampling data sheet to calculate the moisture content of the stack gas. In the laboratory, the condensate impinger sample is placed in a separatory funnel and surrogate compounds for the SVOCs, PAHs, OCPs, and PCBs are added. The sample is then pH adjusted to acid ($\text{pH} < 2$) or basic ($\text{pH} > 11$), and is then separatory funnel extracted using methylene chloride and deionized water. The methylene chloride extract layer is then removed and fresh methylene chloride added to the separatory funnel. The methylene chloride/water separatory extraction is then repeated with the pH adjusted to either acid or basic, depending on the original adjustment. The second methylene chloride extract layer is then removed and combined with the earlier acid or basic methylene chloride extract.

For SVOCs, four types of spiking materials will be applied to the Method 0010 sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD-2 resin tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the SVOCs found using the Method 0010 sampling method.
- Surrogate Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.

- Semivolatile Internal Standard Compounds - These compounds are applied to the sample extract just prior to GC/MS analysis. These compounds applied to the samples are used to determine if the continuing calibration internal standards are still appropriate to use to calculate the associated compound concentrations.
- Matrix Spike Compounds - These compounds are spiked onto the condensate portion of the Method 0010 samples and onto a blank XAD resin. The matrix spike compounds are spiked onto an aliquot of the Method 0010 condensate and blank resin sample before GC/MS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.

For OCPs, four types of spiking materials will be applied to the Method 0010 sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD-2 resin tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the SVOCs found using the Method 0010 sampling method.
- Surrogate Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.
- Organochlorine Pesticides Internal Standard Compounds - These compounds are optional and may be applied to the sample extract just prior to GC/MS analysis. These compounds applied to the samples are used to determine if the continuing calibration internal standards are still appropriate to use to calculate the associated compound concentrations.
- Matrix Spike Compounds - These compounds are spiked onto the condensate portion of the Method 0010 samples and onto a blank XAD resin. The matrix spike compounds are spiked onto an aliquot of the Method 0010 condensate and blank resin sample before GC/MS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.

For PAHs five types of spiking materials will be applied to the Method 0010 sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD-2 resin tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the PAHs found using the Method 0010 sampling method.
- Internal Standard Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. These standards are used to measure the concentration of the analytes and surrogates.
- PAH Recovery Standards- Isotopically labeled compounds applied to the Soxhlet extracts just before HRGC/HRMS analysis. Recovery standards are added to the sample are used to estimate the recovery of the internal standard. Recovery of the internal standard is an indicator of the overall performance.
- Matrix Spike Compounds - These compounds are spiked only to the condensate portion of the Method 0010 samples and a blank XAD resin. The matrix spike compounds are spiked onto an aliquot of the Method 0010 condensate and a blank XAD resin sample

before HRGC/HRMS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.

- Alternate Standard – This compound is added to the impinger prior to extraction. The alternate standard is used to estimate extraction efficiency for the PAH impingers.

For PCBs five types of spiking materials will be applied to the Method 0010 sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD-2 resin tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the PCBs found using the Method 0010 sampling method.
- Isotopically Labeled Analogs - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. These standards are used to measure the concentration of the analytes and surrogates. Concentrations for compounds isotopically labeled analogs added to the sample are determined using the isotope dilution technique.
- PCB Cleanup Standards- Cleanup standards are added after extraction and just prior to clean-up to assess the clean-up of the sample.
- Matrix Spike Compounds - These compounds are spiked only to the condensate portion of the Method 0010 samples and a blank XAD resin. The matrix spike compounds are spiked onto an aliquot of the Method 0010 condensate and a blank XAD resin sample before HRGC/HRMS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.
- PCB Internal standards – These compounds are applied to the sample extract just prior to GC/MS analysis. These compounds applied to the samples are used to determine if the continuing calibration internal standards are still appropriate to use to calculate the associated compound concentrations. Concentrations for compounds without isotopically labeled analogs are determined using the internal standard method.

This test program includes target lists of SVOCs PAHs, OCPs, and PCBs for the Method 0010 SVOC PIC sampling. Commensurate with the procedures allowed under at 40 CFR 63.1208(b)(1)(iii) for the Method 0023A analysis for dioxin/furan congeners, any target SVOC, OCP, or PAH compound that is non-detect in all three analysis fractions of the Method 0010 sampling train will be counted as zero in determining the emissions data for use in the post-test risk assessment conducted under RCRA Omnibus authority [40 CFR 270.32(b)(2)]. For any of the non-target SVOC compounds tentatively identified using a library search for all SW-846 Method 8270 analytes not found during all three runs of the test, the tentatively identified compounds (TICs) will be averaged with zeros for the runs where they are not identified.

6.2.3.3 Method 0023A

A Method 0023A sampling train will be used to sample stack gas dioxin and furans. During each test run, the Method 0023A sampling train will be assembled and leak checked. As required by 40 CFR 63.1208(b)(1)(ii), the Method 0023A sampling train will be operated a minimum of 180 minute (3 hours) to

sample a minimum of 2.5 dry standard cubic meters of stack gas during each sampling run. At the end of each run, the sampling train will be disassembled and all train samples collected. In accordance with 40 CFR 63.1208(b)(1)(iii), any dioxin/furan congener that is non-detect will be counted as zero in determining compliance.

The Method 0023A sampling train front half and back half components are prepared and analyzed as two separate fractions. Surrogates compounds discussed below are applied to each fraction.

The first fraction, the front-half fraction, consists of the solvent probe rinses and the particulate filter. The front half samples will be combined. Internal standard compounds for the PCDD/PCDFs are spiked directly onto the filter before the Soxhlet extraction. The sample is then Soxhlet extracted for 18 hours using methylene chloride followed by 18 hours of extraction using toluene. The methylene chloride and toluene front-half extracts are combined and then blown down. Recovery standards are then added prior to the final concentration for HRGC/HRMS analysis via SW-846 Method 8290.

The second fraction consists of the XAD-2 resin and condenser rinse. The XAD-2 resin and condenser rinse samples will be combined. Internal standard compounds for the PCDD/PCDFs are spiked directly onto the XAD-2 resin before the Soxhlet extraction. The sample is then Soxhlet extracted for 18 hours using methylene chloride followed by 18 hours of extraction using toluene. The methylene chloride and toluene back-half extracts are combined and then blown down. Recovery standards are then added prior to the final concentration for HRGC/HRMS analysis via SW-846 Method 8290.

Three types of spiking materials will be applied to the Method 0023A sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the dioxin and furans found using the Method 0023A sampling method.
- Isotope Dilution Internal Standard Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.
- Dioxin and Furan Recovery Standards- Isotopically labeled compounds applied to the Soxhlet extracts just before GC/MS analysis. These compounds are provide the relative response factors which are used to calculate analyte concentrations.

6.2.3.4 Method 29

An EPA Method 29 sampling train will be used to sample stack gas for the project target metals. Samples are subjected to acid digestion using nitric and hydrofluoric acid in either a parr bomb or microwave pressure relief vessel. Non-mercury metals will be analyzed by SW-846 Method 6010B

[inductively coupled argon plasma spectroscopy (ICP or ICAP)] or 6020 [inductively coupled argon plasma spectroscopy/mass spectroscopy (ICP/MS or ICAPMS)]. Mercury will be analyzed for by SW-846 Method 7470 [cold vapor atomic absorption spectroscopy (CVAA or CVAAS)]. Accuracy and precision are measured through the use of a matrix spike and matrix spike duplicate. Two blank trains are collected and spiked to provide the MS/MSD analysis. The five Method 29 sampling train fractions will undergo seven separate analyses as follows:

- The nitric acid probe rinse and the particulate filter will be combined and digested in the laboratory as the front half composite sample and analyzed for Hg and the non-Hg target metals.
- The condensate knockout impinger (impinger 1) and the HNO₃/H₂O₂ impingers (impingers 2-3) catches will be digested in the laboratory and analyzed for Hg and the non-Hg target metals.
- The empty impinger (impinger 4) catch will be digested in the laboratory and analyzed for Hg.
- The KMnO₄/H₂SO₄ impinger (impingers 5-6) catches will be digested in the laboratory and analyzed for Hg.
- The 8N HCl rinse of impingers 5-6 will be digested in the laboratory and analyzed for Hg.

6.2.3.5 Method 0061

A Method 0061 sampling train will be used to sample for hexavalent chromium during Test 2. Stack samples are filtered, preconcentrated and analyzed directly using ion chromatography following SW846 Method 7199. Accuracy and precision are determined through the use of duplicate analysis, laboratory control samples, and matrix spikes.

6.2.3.6 EPA Method 26A

An EPA Method 26A sampling train will be used to sample for particulate, HCl and Cl₂, during each test run. The stack gas is sampled by bubbling the gas through impingers containing 0.1N H₂SO₄ (acid) and 0.1N NaOH (basic) solutions in series. In the acid impinger solution, HCl gas is captured. Any Cl₂ passes through to the acid impinger and is captured in the basic impinger solution. The chloride concentrations of the acidic and basic impinger samples are analyzed separately for chloride ion, and are reported as HCl and Cl₂ catches respectively. During the sampling train recovery, the basic Cl₂ catch is preserved as by addition of 0.008% sodium thiosulfate (Na₂S₂O₃). The sodium thiosulfate preserves the Cl₂ sample by converting any Cl₂ captured as hypochlorite ion (OCl⁻)¹⁰ the more stable chloride ion (Cl⁻). Precision for these samples are determined through the use of duplicate analyses of all calibration standards, all QC samples and all field samples. Accuracy is determined by matrix spike/ matrix spike duplicate analyses. Additional matrix specific quality control is provided by separate matrix specific calibrations being analyzed prior to sample analysis.

The stack gas particulate emissions are determined by weighing the tare weighted particulate filter to determine the differential weight of the particulate collected by the Method 26A sampling train. Samples are dried to a constant weight to the nearest 0.1 mg. Constant weight shall mean a difference between two consecutive weighings of no more than 0.5 mg difference or more than 1 percent of the total weight less the tare weight. This differential weight is added to the weight of the residue remaining after evaporation of the acetone probe and filter holder rinses. The reported particulate catch is the sum of the particulate filter differential weight and the probe rinse residue weight.

6.2.3.7 Method 0010 for TCO/Grav

A Method 0010 sampling train will be used to sample stack gas total chromatographable organics (TCO and gravimetric organics (Grav). During each test run, the Method 0010 sampling train will be assembled and leak checked. The sampling train will be operated to sample a minimum of three dry standard cubic meters of stack gas during each sampling run. The sampling time to achieve the target sample volume will depend upon the probe nozzle size and sampling rate. However, sampling rate will not exceed one dry standard cubic foot per minute. At the end of each run, the sampling train will be disassembled and all train samples collected.

In the laboratory, the TOC/Grav front and back half rinses, particulate filter and XAD-2 resin are combined and Soxhlet extracted with methylene chloride. The final pooled extract volume must be no less than 5 mL. Since the extracts for total organics determinations are analyzed by GC/FID and gravimetric techniques, no sampling surrogate, isotopically-labeled standards, or internal standards associated with GC/MS analysis (Method 8270) are added to the extractors or sample extracts. The combined methylene chloride extracts are split into four portions and used as follows:

- One portion of the extract is used for semivolatile organic analysis using TCO GC/FID analysis according to EPA Guidance for Total Organics. Details of the method are described in Appendix C of the Guidance.
- Two portions of the methylene chloride extract are used for nonvolatile organic analysis using gravimetric mass (Grav) method according to EPA Guidance for Total Organics. Details of the method are described in Appendix D of the Guidance.
- The final portion is used as an archive sample.

6.2.3.8 Cascade Impactor for PSD

A cascade impactor will be used to sample for particle size distribution (PSD). The Cascade impactor employs a series of plates and filters used to separate particles in the stack gas into aerodynamic particle size ranges based on the flow of gas through small holes in each plate. The stack sampling train is operated for a time selected by the sampling team to obtain enough particle mass (without overloading individual filters) for particle size determination. The filter substrates are carefully recovered from the

sampling train, placed into individual petri dishes, and sealed. Each filter substrate is weighed in a manner similar to EPA Method 5, and the particle mass on each filter is then used to determine the particle size distribution.

6.2.4 Blank Trains and Reagent Blanks

Blank train samples are the samples recovered from sampling trains that have been assembled and charged with all the required chemical reagents and collection media in the same manner as the sampling trains used to sample the stack gases. The sampling trains are leak checked and heated to temperature in a location near the stack. The sampling train remains sealed at the stack location for a period equivalent to the length of time the corresponding sampling train is operated during the test run. The blank train is then recovered in the same way that actual stack gas sampling trains are recovered. The recovered blank train components are labeled as blank train samples and submitted for analysis with the actual stack gas train samples. The results of the blank train samples provide an indication of possible contamination introduced to the samples by reagents, glassware, sampling environment, and sampling recovery. The blank train samples for the stack sampling trains used during this test program will be collected as summarized in Tables 6-2. For this test program one baseline test, and up to two optional, additional tests, for a maximum of nine test runs total, two Method 29 blank trains, one for a matrix spike sample and one for a matrix spike duplicate sample, will be collected. One each blank train of the each the Method 0010 SVOC/OCP, Method 0010 PAH/PCB, Method 0023A, and Method 0010 TCO/Grav trains will be collected during the testing.

Reagent blanks are samples of the reagent source solvents, solutions, and other media used in stack sampling. Reagent blank samples for the Method 26A, Method 29, Method 0023A, Method 0010 SVOC/OCP, Method 0010 PAH/PCB, and Method 0010 TCO/Grav sampling trains used during this test program will be collected as summarized in Table 6-2.

6.2.5 Field Blanks

Field blanks are sampling media that are handled at the sampling location in the same manner as the actual test samples. However, these media are not used to collect stack gas samples. The field blank samples will be collected and analyzed to demonstrate that the sample handling procedures do not expose the samples to contaminants. This test program includes collecting one pair of VOST tubes as field blanks once during each test run (three samples total). Each field blank VOST tube pair is opened in the field by the VOST operator during the sampling run and are allowed to remain open for a period equivalent to the time required for a tube pair change-out during testing. The field blank pair is then sealed up, labeled and handled in the same manner as other VOST samples. The compounds found in field blanks reflect exposure to field fugitives, laboratory contaminants and resin degradation products, and are used to assess any contamination that can impact test results.

6.2.6 Trip Blanks

Trip blanks are similar to field blanks in that they are used to assess contamination resulting from sample shipment. With each shipment of VOST samples from the test site to the laboratory, a pair of VOST tubes that have remained sealed as shipped from the laboratory to the field to be used as trip blanks. Additionally, pair of volatile organic analysis (VOA) vials filled with deionized (DI) water is included with the VOST samples shipped from the test site back to the laboratory. The trip blank and DI water analyses demonstrate that the samples are not exposed to contamination during transport from the field to the laboratory.

7.0 SAMPLE HANDLING, TRACEABILITY, AND HOLDING TIMES

7.1 SAMPLE CUSTODY

A sample will be considered to be in the custody of a person if it is in his or her possession, in his or her sight, or secured by that person in an approved location accessible only to authorized personnel.

The analytical laboratory will prepare the sampling media and sample reagents according to the specifications of the methods as described in the PDT plan and will ship them to the site in sealed containers.

During the test, once the samples are transferred from the sampling technician to the Sample Custodian, sample custody becomes the responsibility of the Sample Custodian until the samples arrive at the analytical laboratory. When overnight couriers are utilized, the air bill will serve to document the transfer of custody from the Sample Custodian to the courier. The courier's air bill becomes part of the chain of custody (COC) record. Upon transfer of the samples from the courier to the analytical laboratory, sample custody will be maintained by the analytical laboratory performing the analyses. Samples for organic analysis (VOST, Method 0023A, POHC, etc.) will be kept on ice ($4\pm 2^{\circ}\text{C}$) and shipped to the analytical laboratory in sealed, insulated shipping containers (ice chests). All ice on shipped samples shall be triple bagged in ziplock bags to prevent leakage of water during shipping. Samples not requiring chilling (particulate, chloride, metals, properties, etc.) may be shipped in sealed, insulated shipping containers (ice chests) without ice. **If dry ice is used to preserve organic samples during shipping, the shipment packaging and placarding shall be conducted in strict accordance with the International Air Transport Association (IATA) regulations.**

Collected samples will be shipped from the site to the laboratory in sealed containers with chain of custody (COC) and request for analysis (RFA) forms. Example COC and RFA forms are presented as Figures 7-1 and 7-2 respectively. Prior to shipping any samples, the condition of these samples will be documented on the chain of custody by the Sample Custodian. Conditions to be verified and documented on the COC include but are not limited to the following: sample temperatures for organic analyses samples (measurement will be taken using the temperature blank), condition of all containers, level of sample within all containers (to be marked on the outside of the container), and type of packing material used. RFAs will be checked against the COCs to verify there is a RFA for every sample being shipped. **Before shipping the samples from the site, the Sample Custodian shall make three photocopy sets of all COCs and RFAs. One copy will be retained by the Sample Custodian, and one copy each will be provided to the Test Coordinator and the QAO.**

Upon receipt, the shipping containers will be opened by the Laboratory Analysis Coordinator or his designee and inspected. The receiver will verify that the container contents correspond with the COC. Any damage to the contents of the shipping container or deviations from the original shipment documents will be noted on the COC and the receiver will accept custody for the shipment by an exchange of signatures with the delivering agent.

Upon receipt of samples at the laboratory, the sample shipping containers will be opened and all sample containers inspected. A labeled temperature blank (labeled VOA vial with water) will be shipped in every container with samples for organic analysis expressly for the purpose of determining sample temperatures. **The Laboratory Analysis Coordinator or designee will, immediately upon opening the sample packaging, open the temperature blank and measure the temperature of the water inside the temperature blank using a thermometer. This temperature will be recorded on the COCs and any applicable laboratory documentation (sample receipt log).** Containers will then be secured in a location accessible only to authorized personnel. Samples for organic analysis shall be secured in refrigerated sample storage. The COC forms are used specifically to track the samples. To provide specific instructions to the analysts, the RFAs will accompany the respective COCs.

Transfer of custody to and within the analytical laboratory is addressed in the Laboratory's QA Manual. Upon completion of analysis, samples will be maintained at the laboratory under chain of custody until they are released for proper disposal.

7.2 SAMPLE LABELING

An example sample label format is presented in Figure 7-4. Each sample container will be labeled to show the source of the sample as WCAI; the project identification; sampler's initials; laboratory to which the sample will be shipped; an unique alphanumeric sample number; date and time; sample description; test number; and run number. If a single sample requires multiple containers, the number of the container and the total number of containers will be noted on the label.

Project samples will be tracked via the assigned unique alphanumeric sample numbers. The sample number will appear on the sample label, the RFA and the COC. The alphanumeric system of sample identification for this project is presented in Figure 7-5. The numbering system presented will result in unique numbers being assigned to every sample.

7.3 PROCESS SAMPLE COLLECTION FORMS

While a process sample is being taken in the field, the sampling technician will complete a field sampling record. An example field sampling record is presented as Figure 7-3. The field sampling record will be

filled out in its entirety for every sample. This will provide information to be used in the final report. The sampling technician shall provide the completed field sampling record and COC form to the Sample Custodian.

7.4 SAMPLE COLLECTION CHECKLIST

Prior to start of testing, a master list of the samples required for the test will be compiled by the Sample Custodian or QAO. This list will identify the samples by their assigned unique alphanumeric sample numbers (refer to Figure 7-5) and the analytical test(s) required. As field samples are acquired and routed through the Sample Custodian, the samples will be checked off against the master list to ensure that all of the appropriate samples have been taken.

7.5 REQUEST FOR ANALYSIS/CHAIN OF CUSTODY

The Sampling Technician and Sample Custodian will complete the COC and RFA forms for every sample. Each sample may consist of several sub-samples. Each individual component of the sample will be listed separately on the COC with its own unique alphanumeric sample identification number. The samples will be preserved as needed and secured in a shipping container by the sampler and must remain in his or her possession until it is presented to the Sample Custodian. The Sample Custodian will secure the samples in a location accessible only to authorized personnel until custody is transferred to a courier for delivery to the laboratory.

Each sample container will be clearly identified using standard container labels. It is imperative that information on the COC form, RFA form and the container label match in every respect. Example COC and RFA forms are presented in Figures 7-1 and 7-2. The label format is shown in Figure 7-4. Planned sample identification codes are shown in Figure 7-5. An individual trained in Federal Department of Transportation (DOT) and International Air Transport Association (IATA) regulations will package the samples for overnight courier shipment to be in compliance with the applicable portions of these regulations.

7.6 SAMPLE PRESERVATION

Table 7-1 shows the appropriate containers, preservation, and holding times for all samples to be collected during the test.

The location of the VOST sample holding area is of special importance. The sample containers for volatiles will be stored in a clean area separate from the sample preparation area. High concentration volatile organic samples (recovered elements of other sampling trains, waste feed samples, etc.) will be

segregated to prevent inadvertent contamination of the VOST samples. The VOST tube sample pairs will be preserved before and after sampling by placing them on triple bagged ice in a dedicated sample cooler. To preclude contamination from solvents and process samples, VOST samples will be shipped on triple bagged ice in dedicated shipping containers separate from all other test samples.

XAD-2 traps for the Method 0023A, Method 0010 SVOC/OCP, Method 0010 PAH/PCB, and Method 0010 TCO/Grav trains will be preserved before and after sampling by placing them on triple bagged ice in a dedicated sample cooler. The balance of the Method 0023A, Method 0010 SVOC/OCP, Method 0010 PAH/PCB, and Method 0010 TCO/Grav train sample components will be preserved after sample collection by placing them on triple bagged ice in a dedicated sample cooler. Other organic analysis samples (e.g., process samples for POHC) will be preserved after sample collection by placing them on triple bagged ice in a dedicated sample cooler.

For non-organic analysis samples (particulate, chloride, metals, properties, etc.), sample preservatives (if applicable, refer to Table 7-1) will be used as required by the target analyte. These samples will be stored in dedicated sample coolers. These samples do not require chilling on ice or refrigeration.

8.0 SPECIFIC CALIBRATION PROCEDURES AND FREQUENCY

The objective of this section is to assure that process instrumentation, gas sampling equipment, and analytical instruments are performing properly before conducting the test and analyzing samples. Equipment and instruments used to generate data for determining compliance with performance requirements or to establish quantitative allowable operating limits will be calibrated according to the manufacturer's instructions, prior to and/or during the test as necessary.

The calibration procedures are separated into groups according to the personnel who will perform them. WCAI operations personnel will calibrate the process instruments. Stack sampling equipment will be calibrated by the stack sampling contractor and analytical instruments will be calibrated by the contracted laboratory personnel. The calibration procedures for process instrumentation stack gas sampling, and laboratory analytical instruments are described in the following subsections.

8.1 PROCESS INSTRUMENTATION

Prior to the start of testing, the parameter continuous monitoring system (CMS) (thermocouples, flow meters, pressure transducers, etc.) and the continuous emission monitoring system (CEMS) (installed CO and O₂ monitors) will be calibrated in accordance with the facility standard operating procedures.

During testing, the installed carbon monoxide and oxygen CEMS, the monitors will be calibrated daily. The zero and span checks will be considered a verification of the data quality from these monitors.

CMS and CEMS data will be reported on 1-minute intervals and will be archived in the CMS data acquisition system.

8.2 STACK SAMPLING EQUIPMENT

Sampling equipment is calibrated according to the criteria specified in the reference method being employed. In addition, the guidelines set forth in the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods (EPA-600/4-77-027b) will be followed. Dry gas meters, orifices, nozzles, pitot tubes, etc. are calibrated in accordance with this document. The range of the calibration is specified for all environmental measurements to encompass the range of probable experimental values. This approach ensures that all results are based upon interpolative analyses rather than extrapolative analyses.

Calibrations are designed to include, where practical, at least three measurement points evenly spaced over the range. This practice minimizes the probability that false assumptions of calibration linearity will be made. In addition it is common practice to select, when practical, at least one calibration value approximating the levels anticipated in the actual measurement. Typically, calibration frequency is dictated by the need to demonstrate the stability of the calibration value over the course of measurements. Calibrations are made both pre- and post-test to accomplish the demonstration of stability.

Following the test program, calibrations are checked on all relevant items of sampling equipment to ensure the validity of data collected in the field. New items for which calibration is required are calibrated before initial field use. Equipment whose calibration status may change with use or time is inspected in the field before testing begins and again upon return from each field use. When an item of equipment is found to be out of calibration, it is repaired and recalibrated or retired from service. All equipment is periodically recalibrated in full, regardless of the outcome of these regular inspections.

Data obtained during calibrations are recorded on standardized forms, which are checked for completeness and accuracy by management personnel. Data reduction and subsequent calculations are performed using standard procedures, and are computerized where appropriate. Calculations are checked at least twice for accuracy. Copies of calibration forms are included in the test or project reports.

Emissions sampling equipment requiring calibration include pitot tubes, pressure gauges, thermometers, dry gas meters, and barometers. The following sections elaborate on the calibration procedures for these specific equipment items.

8.2.1 Pitot Tubes

All Type S pitot tubes, whether separate or attached to a sampling probe, are inspected in accordance with the geometry standards contained in EPA Method 2. All Type S pitot tubes are calibrated over an eight-point range with a wind tunnel. A calibration coefficient is calculated for each pitot tube.

Each pitot tube is inspected visually upon return from the field. If a visual inspection indicates damage or raises doubt that the pitot remains in accordance with the EPA geometry standards, the pitot tube is first calibrated, then repaired and recalibrated. The acceptance limits are listed in Table 8-1.

8.2.2 Differential Pressure Gauges

Some meter consoles are equipped with 10-inch water column (W.C.) inclined-vertical manometers. Fluid manometers do not require calibration other than leak-checks. Manometers are leak-checked in the field prior to each test series and again upon return from the field.

8.2.3 Digital Temperature Indicator

One digital temperature indicator is used to determine the flue gas temperature, probe temperature, oven temperature, "train temperature" and dry gas meter temperature. The digital temperature indicator is calibrated over a seven-point range (32°F-450°F) using an ASTM mercury-in-glass thermometer as a reference. The calibration is acceptable if the agreement is within $\pm 2\%$ or 2°F from 50°F-180°F.

8.2.4 Dry Gas Meter and Orifice

A calibrated wet test meter is used to calibrate the dry gas meter and orifice. The full calibration procedure is used to obtain the calibration factor of the dry gas meter. Full calibrations are performed using a calibrated wet test meter as a reference standard.

8.2.4.1 Dry Gas Meter

Each metering system receives a full calibration at the time of purchase and quarterly. Upon request, a post-test calibration can be performed after each field use. If the calibration factor deviates by less than five percent from the initial value, the test data are acceptable. If it deviates by more than 5%, the meter is recalibrated and the meter coefficient (initial or recalibrated) that yields the lowest sample volume for the test runs is used.

EPA Method 5 requires another full calibration anytime the post-test calibration check indicates that the calibration factor has changed by more than 5%. Standard practice is to recalibrate the dry gas meter quarterly and check the orifice calibration during and after each field use.

8.2.4.2 Orifice

An orifice calibration factor is calculated for each of the eighteen flow settings during a full calibration. The arithmetic average of the values obtained during the calibration is used.

8.2.5 Barometer

Each field barometer is adjusted before each test series to agree within ± 0.1 inches of a reference aneroid barometer. The reference barometer is checked against the station pressure value (corrected for elevation difference) reported by the National Weather Service.

8.3 LABORATORY ANALYTICAL EQUIPMENT

The laboratory instruments will be calibrated as specified by the appropriate method before analyzing the test samples. The laboratory instrument calibration procedures are based on instructions in the

referenced analytical methods and are summarized, along with other routine quality control checks, in Table 8-2. The calibrations performed and the results will be reported as appropriate to assure the quality of data in the laboratory sample analysis report.

9.0 ANALYTICAL PROCEDURES

Analytical procedures and methods are summarized in Table 9-1. Individual sampling and analytical methods are described in detail in Attachments A and B of the PDT plan and are incorporated here by reference. Tables 9-2 and 9-3 represent the volatile and semivolatile products of incomplete combustion (PICs) which will be targeted during the analysis of the test samples. These lists represent the target compound list (TCL) to be used by the laboratory. In addition to the TCL compounds, non-TCL peaks greater than 10% of the nearest internal standard will be tentatively identified using a library search for all SW-846 Method 8260 and 8270 analyses. Table 9-4 lists the target PAH analytes for CARB Method 429. Table 9-5 lists the target PCB analytes for EPA Method 1668. Table 9-6 lists the target dioxin and furan analytes for SW-846 Method 8290. Table 9-7 lists the target metal analytes by SW-846 Methods 6010 or 6020, and 7470. Table 9-8 lists the target organochlorine pesticides analytes for SW-846 Method 8081.

All analyses will be performed by a laboratory qualified in the categories of sample analysis delineated in this section. Laboratory qualifications can be submitted upon request. The following is a list of the analytical reference methods for the procedures presented in Table 9-1:

- Test Methods for Evaluating Solid Waste, SW-846 (SW-846), Third Edition, November 1986 and Updates
- Sampling and Analysis Methods for Hazardous Waste Incineration, EPA 600/8-84-002.
- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series
- Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.
- Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020.
- Performance Specification 4B, Appendix B, 40 CFR 60.

10.0 SPECIFIC INTERNAL QUALITY CONTROL CHECKS

10.1 DEFINITIONS

The various types of QA/QC checks that may be performed as part of the test, both for sampling and analysis, are defined below. One or more of these QA/QC checks are associated with each measurement system in order to assess the compliance of the data to the DQOs established in Section 5.0. Table 10-1 is a summary of all the sample analyses and their associated internal quality control checks associated with this test program.

Audit Sample An audit sample is a field or alternate laboratory prepared blank spike submitted to the test laboratory to assess accuracy or potential sample degradation.

Blank, Field A field blank is a sampling train or sampling component that is set-up in the field but is not used for test sampling. The field blank is used to assess background contamination that may affect the representativeness of the field samples.

Blank, Media A sample of unused sampling media analyzed to ensure the media are uncontaminated. This type of sample may also be referred to as a “reagent blank” (see below).

Blank, Method A method blank is a sample of unused media that is prepared and analyzed in the test laboratory to assess background contamination that may exist in the laboratory, on glassware, or in the analytical system.

Blank, Reagent A sample of unused reagent(s) used to demonstrate the absence of contamination in the reagents.

Blank, Spike A blank spike is a laboratory prepared sample of blank media that is spiked with a known amount of target analyte(s) used to assess the accuracy of the analytical method.

Blank, System An aliquot of uncontaminated reagent used to clean out the analytical system after high level samples have been analyzed or before analysis begins.

Blank, Trip A trip blank is an unused sample component that is shipped to the field along with the sampling equipment/media and/or returned to the laboratory without having been exposed to field conditions. If contamination is encountered in the field blank(s), the trip blank is analyzed to assess

whether or not the contamination originates in the field, is inherent in the equipment/media, or results from exposure during shipping and handling.

Breakthrough Check The result of the analysis of a secondary component (i.e., sorbent tube) in a sampling train is compared to the result of the primary component to assess whether or not the primary component has successfully captured the target analytes. If the result of the secondary component analysis is high compared to the primary component analysis, the possibility exists that the analytical results may be artificially low.

Calibration Check A standard solution from a source other than the calibration standards used to verify the integrity of an instrument's calibration.

Calibration Standards High purity compounds or mixtures of compounds used to adjust the response of an analytical instrument. The laboratory will use traceable standards and submit standard preparation logs as part of the deliverables package.

Contingency Sample An archived portion of a field sample from the same location as other field samples that is collected and held in case of breakage or QA/QC failure during the handling or analysis of the primary sample. This type of sample is sometimes referred to as an “archive sample.”

Continuing Calibration Verification A mid-point standard, from the same Calibration source as the initial calibration solution analyzed periodically to verify that calibration conditions have not drifted from the initial calibration.

Duplicate Analysis A duplicate is a sample that is split in the laboratory and prepared and analyzed twice. The results of the two analyses are compared as a measure of precision.

Duplicate Injection A second analysis of a single sample preparation. This QC test may be used to assess analytical QC failures, matrix interferences, or as a measure of analytical system precision.

Initial Calibration A series of analyses of solutions, that have known concentrations, used to establish the correspondence between the amount of an analyte present in the solution and the instrument's response across the expected analytical range of the samples. Initial calibrations also establish retention time windows for identification purposes in chromatographic methods.

Interference Check An interference check sample is analyzed, for ICP analysis only, to assess the possible error in analytical results arising from the interaction of various metals in the sample under the conditions of analysis.

Internal Standard Recovery Internal standards are non-target spikes added to samples for quantitation purposes. The percent recovery of the internal standards is checked to assess whether or not significant matrix interferences may affect the accuracy and precision of analytical results.

Performance Evaluation (PE) Sample See Audit Sample.

Proficiency Test A series of blank spikes analyzed in the test laboratory to demonstrate an analyst's ability to successfully perform the method with acceptable precision and accuracy.

Replicate One of a series of identical samples or splits of a single sample used to assess precision.

Serial Dilution The result of the analysis of a highly contaminated sample, run undiluted, is compared to the results for the same sample after serial dilution. The two results are expected to match to within method specified criteria. This test is a measure of the linearity of ICP calibration and the analysis technique.

Spike, Field See Audit Sample.

Spike, Matrix Spike of the known or controlled amount of an actual target analyte to an actual sample matrix that is then analyzed for that analyte. The percent recovery of the spiked analyte provides a measure of the matrix bias.

Surrogates Non-target or isotopically labeled analytes spiked into field samples as a measure of method efficiency and accuracy.

10.2 SPECIFIC QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

A variety of QC checks are required both in the field and in the laboratory to ensure the collection of samples that accurately represent the field conditions under study, to assess compliance with the Data Quality Objectives (DQOs), and to assess biases in the measurement system.

10.2.1 Field Activities

In order to ensure the representativeness of samples collected during the test, and to ensure integrity of field measurements, a variety of QC checks and controls will be implemented throughout the sampling program. These checks and controls will include:

- Standard forms and/or standard field notebooks will be used to document field activities and for data collection. The data collection forms and field notebooks will be reviewed routinely by senior staff for accuracy, completeness, and internal consistency.
- The strict adherence to detailed operating procedures as documented in the various project controlling documents and related SOPs will be enforced by experienced senior technical staff.
- Project personnel will be selected based on appropriate levels of training and experience and will receive project specific training prior to working on-site. Training will include health and safety requirements; security requirements; briefings on overall project goals, objectives, and schedules; and, specific technical training related to their assigned tasks. Training will be documented in the project files.
- Routine calibration will be performed on measurement systems and sampling equipment including metering systems, thermocouples, barometers, rotameters, and pitot tubes. Guidance related to equipment calibration is provided in Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods and Quality Assurance/Quality Control Procedures for Hazardous Waste Incinerators, Appendix A. The detailed specifications, acceptance criteria, and corrective action requirements are presented in Section 8.0 of this QAPP. All calibrations will be documented and the documentation maintained in the project files.
- Leak checks will be performed according to method specifications before and after sampling.
- Field QC samples will be routinely submitted including audit (PE) samples, field blanks, media blanks, reagent blanks, trip blanks, and contingency samples. The frequency of submittal for these field QC samples and other field samples are provided in Tables 5-1 and 8-2.
- Field audits/surveillance will be performed periodically by the QAO to assess conformance to specifications. If nonconforming conditions are noted, the corrective action provisions of the QA plan will be invoked.

10.2.2 Laboratory Activities

Standard laboratory QA procedures, required of each laboratory, provide discussions related to QA/QC checks and controls within the laboratory. Specific data quality objectives, calibration requirements, acceptance criteria, and corrective action requirements for this test program are presented in Table 5-1 and Table 8-2 of this plan.

In addition to the requirements referenced above the laboratory will provide for quality control of sampling media and sample collection equipment. Sorbents used in the organic sampling trains will be prepared according to method specifications. Samples of the prepared media will be tested according to the intended method of use and analysis prior to shipping media to the field. The results of these tests will be retained in the laboratory's files for future reference.

11.0 DATA REDUCTION, DATA VALIDATION, AND DATA REPORTING

11.1 DATA REDUCTION

11.1.1 General Principles

11.1.1.1 Field

Data reduction will occur for the field measurements at the point of sampling. At the point of sampling, the data as measured by the field instrument will be reported in the field notebooks and/or on any forms required for the project.

11.1.1.2 Office

After the field event, the data may be further reduced to data tables, trend analysis tables, or graphs. At any point where manual transcriptions of data take place, an editing function will be invoked to ensure accuracy of the transcriptions.

Upon the return of the analytical results from the laboratory and after data validation, the data will be further reduced to data tables. The data tables will contain the following information:

- Information identifying exactly the samples represented on the tables (e.g. sample location, matrix, etc.),
- The compounds for which the samples were tested,
- The results for each compound, and
- The data flags as applied by the laboratory or by data validators (if used).

11.1.1.3 Laboratory

Data reduction in the laboratory is covered in the Laboratory's QA Manual and SOPs. The laboratory's data reduction process will include at a minimum the following.

- Transcription of data results from raw data printouts to data report forms. This will include any calculations required to report the data in the required units.
- Transcription of QA/QC data onto summary forms to provide the required information for evaluation of the validity of the data. The requirement for each type of data is included in section 11.3.

11.1.2 Specific Data Reduction Requirements

11.1.2.1 GC and GC/MS Techniques

Organic analyses will all be conducted using gas chromatography (GC) techniques. The VOC, SVOC, PAH, OCP, PCB and PCDD/PCDF analyses will employ mass spectral (MS) detectors. Although the principles of operation and specific methods of calibration differ according to the analyte specific methods, the general data reduction scheme is the same for all of these tests. The individual methods should be consulted for details. A summary of the data reduction scheme is presented below.

Depending on the specific method, analytical instrumentation is calibrated at 3 to 5 points covering the expected analytical range. The gas chromatograph/flame ionization detector (GC/FID) or gas chromatograph/electron capture detector (GC/ECD) methods generally employ an external standard calibration technique while the GC/MS methods employ an internal standard technique. For GC/FID and GC/ECD methods, a calibration factor (CF) is calculated using the following formula:

$$CF = \frac{R}{M}$$

Where: CF = Calibration Factor

R = Response or Area of the GC Peak

M = Mass Injected (in nanograms)

The calibration factors must agree to within method specified criteria for the percent relative standard deviation (%RSD). The formula for %RSD is given below.

$$\% \text{ RSD} = \frac{s}{\text{avg } CF} \times 100$$

Where: %RSD = Percent Relative Standard Deviation

σ = Standard Deviation of the Calibration Factors

avg CF = Average Calibration Factor

For GC/MS calibrations a response factor (RF) is used rather than the CF. The formula for the RF is:

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}$$

Where: RF = Response Factor

A_s = Response for the Analyte

A_{is} = Response for the Internal Standard

C_s = Concentration of the Analyte

C_{is} = Concentration of the Internal Standard

The RFs must also pass a test of the %RSD in order for the calibration to be considered valid.

When samples are analyzed, the area of the peak produced by a given analyte is compared to the CF or RF to arrive at an analytical result according to the following formula:

$$M_x = A_x \times CF$$

Where:

M_x = The Mass of Analyte in the Sample

A_x = The Response of the Analyte in the Sample

CF = The Calibration (or Response) Factor

Samples containing more of an analyte than the instrument is calibrated for, will have a dilution performed, if such a dilution is practical given the sample preparation method. In that case M_x is multiplied by the dilution factor to arrive at the final result. If, under the circumstances of the method, a dilution is not possible, the analytical result must be considered estimated.

To arrive at a concentration in the gas sample the mass of any sub-samples must be added together and then compared to the volume of gas sampled according to the following formula:

$$C_x = \frac{(M_1 + M_2 + \dots M_n)}{V}$$

Where: C_x = The Concentration of the Analyte in the Gas Sample
 M_n = The Result (Mass) for Each Component in the Sampling Train
 V = The Volume of Gas Sampled

11.1.2.2 Analysis of Metals by ICP and Atomic Absorption

The analysis of metals also begins with an instrument calibration at 2 to 6 points, depending on the specific analytical method. For inductively coupled argon plasma spectroscopy (ICP) for non-mercury (non-Hg) metals analyses and cold vapor atomic adsorption spectroscopy (CVAA) for mercury (Hg) analyses, instruments are profiled and calibrated according to the instrument manufacturer's instructions. A calibration blank and a QC check standard are then analyzed to ensure appropriate instrument response. A percent recovery (%R) is calculated according to the following formula:

$$\%R = \frac{Found}{True} \times 100$$

Where: %R = Percent Recovery
Found = The Result of the Analysis
True = The Expected Result

The %R is expected to be within method specifications before analysis can begin. The calibration is verified periodically according to method specifications using the same technique.

Atomic absorption instruments are calibrated at three to five points. A linear regression is performed and a correlation coefficient is calculated to assess linearity of the curve. It is beyond the scope of this document to provide a detailed explanation of the statistics supporting linear regression and the calculation of the correlation coefficient. Reference can be made to any standard statistics text for additional information. Calibration checks are performed as above and periodically verified.

Analytical results for metals are read directly from the instrument in terms of concentration. Dilution factors must be used as discussed above if applicable. In order to combine the results of the sub-samples of a metals sampling train, the concentration is converted back to mass using the following formula:

$$M_{xs} = C_{xs} \times V_{xs}$$

Where: M_{xs} = Mass in the Sub-sample

C_{xs} = Concentration in the Sub-sample

V_{xs} = Volume of the Sub-sample

The concentration in the gas sample can then be calculated.

11.1.2.3 Ion Chromatography

Anions, such as chloride, are separated on the ion chromatograph using a system comprising separator columns, guard columns, and eluents. The system is calibrated at a minimum of 3 points and the calibration is verified with a mid-range standard. Samples are quantitated in the same manner as given above.

11.1.2.4 Direct Reading Instruments

Gravimetric, temperature, pressure, flow, and CEMS data are directly read from the measurement instrumentation. The instrumentation will be calibration checked prior to the test, and routinely prior to reading measurements, however, no data reduction beyond formatting into tables is expected

11.2 DATA VALIDATION

The results of all sample analysis and all QA/QC sample analysis (100% of the laboratory data) will be compared, step by step, by the QAO or his/her designee, to the specifications given in Tables 5-1 and 8-2. The data validation criteria outlined in: Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis, (1994) and Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis, (1994); prepared by USEPA Data Review Work Group will be followed as applicable to the individual methods used. Any sample data associated with a QC check that fail to meet the target criteria established in these tables will be flagged in the final report, and an assessment of the impact, if any, of missing the target data quality objective will be provided. Additional guidance will be found in the analytical methods and EPA/625/6-89/023, Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration.

Each laboratory providing analysis will be required to provide the results based on the method detection limit (MDL) and either derived reliable detection limits (RDLs) or practical quantitation limits (PQLs) for all non-isotope dilution method compounds. Non-detects for the isotope dilution methods will be determined

using the method specific SW-846 definition of a estimated detection limit (EDL) without the use of empirical factors or other mathematical manipulations specific to the laboratory. Results reported between the MDL or EDL and the RDL or PQL will be flagged as estimated. The laboratory must provide include with each data package the basis for and any calculations or statistical methods employed in determining the detection limits used (MDL, EDL and PQL).

Particular attention will be paid to the results of blank data. Analytical data will not be routinely corrected for contamination. They are however evaluated on a case-by-case basis for possible blank correction. A "B" flag will be applied to the samples associated with contaminated blanks such that this information may be assessed in the final report. Risk data will be evaluated against all associated blanks to determine if sample results are less than five times (ten times for common laboratory contaminants) the concentration reported in the blanks. Sample results reported at concentrations less than 5X/10X the blank concentration will be considered not present in the sample and will be qualified as not detected (ND or U).

The output from the data validation process will be a summary comparison of the QA/QC results to the specified data quality objectives, a review and discussion of any deficiencies identified in the data assessments of laboratory performance, and, overall precision and accuracy, representativeness and completeness of the data set.

Detailed procedures for the internal review of data in the laboratory are found in the laboratories QA Manuals and related standard operating procedures (SOPs).

11.3 DATA REPORTING

11.3.1 Experimental Data

Experimental data that will be reported as part of the final test report will include:

- All relevant field measurements in raw and tabular form. This will include, but not necessarily be limited to, calibration data for field instruments, velocity and gas flow measurements, and temperature and pressure measurements.
- Process monitoring data
- All CEMS data to include CO, O₂, and THC
- Analytical laboratory data for all laboratory measurements.

The laboratory deliverable package is expected to include the following elements:

The following forms for all organics analyses using Gas Chromatography/Mass Spectroscopy methods:

- Case narrative and sample identification cross reference

- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Organic analysis data sheet for samples, blanks, and QC analysis (CLP Form 1 or equivalent)
- System monitoring compound/surrogate recoveries summary (CLP Form 2 or equivalent)
- Duplicate analysis summary (CLP Form 3 or equivalent if MS/MSD)
- QC Check Sample summary
- Method blank summary (CLP Form 4 or equivalent) and results
- Instrument performance check summary – tuning reports (CLP Form 5 or equivalent)
- Initial calibration summary (CLP Form 6 or equivalent)
- Continuing calibration check (CLP Form 7 or equivalent)
- Internal standard area and RT summary (CLP Form 8 or equivalent)
- Internal standard recovery summary (PCDD/PCDFs, PAHs and PCBs)
- DDT/Endrin breakdown standards
- Raw data: run logs, mass spectra, quantitation reports, manual integration, and chromatograms for samples, tunes, calibrations, and QC samples, sample preparation logs, and run logs.
- Standards preparation logs and certificate is required
- Sample receipt information including temperature and pH information if preservation is required
- Documentation of all nonconformances and the actions taken
- Examples of all calculations performed
- Detection limits including method detection limits and sample quantitation limits
- Any performance evaluation samples provided
- Percent solids or percent moisture for soil samples

The following forms for all organics analyses using Gas Chromatography:

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Organic analysis data sheet for samples, blanks, and QC samples including retention times of required for the analysis (CLP Form 1 or equivalent)
- System monitoring compound/surrogate recoveries summary (CLP Form 2 or equivalent)
- Duplicate analysis summary (CLP Form 3 or equivalent if MS/MSD)
- QC Check Sample summary
- Method blank summary and results (CLP Form 4 or equivalent)

- Initial calibration summary (CLP Form 6 or equivalent)
- Calibration verification summary (CLP Form 7 or equivalent)
- Graphic Representation of Curve Fit, with Correlation Coefficient
- Analytical Sequence (run logs)
- Raw data: quantitation reports, manual integrations, and chromatograms, and retention times for each column in all field and QC samples, sample preparation logs, and run logs.
- Standards preparation logs and certificates (if applicable)
- Sample receipt information including temperature and pH information if preservation is required
- Documentation of all nonconformances and the actions taken
- Examples of all calculations performed
- Compound confirmation (if required for the analysis)
- Peak Resolution Summary (if required for analysis)
- Retention time window determination
- Detection limits including method detection limits and sample quantitation limits
- Any performance evaluation samples provided
- Percent solids or percent moisture for soil samples

The following forms for all metals analyses:

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Inorganic analysis data sheet (CLP Form 1 or equivalent)
- Initial and continuing calibration verification (CLP Form 2 or equivalent)
- Blanks summary (CLP Form 3 or equivalent)
- Spike sample recovery/Post digest spike sample recovery (CLP Form 5 or equivalent)
- Interference checks sample results (CLP Form 4 or equivalent)
- Serial dilution results (CLP Form 9 or equivalent)
- Duplicate results (CLP Form 6 or equivalent)
- Laboratory control sample results (CLP Form 7 or equivalent)
- Raw data for samples, blanks, QC samples, calibrations, and instrument checks, sample preparation logs and, instrument run logs.
- Documentation of all nonconformances and the actions taken
- Sample receipt information including temperature and pH information if preservation is required
- Examples of all calculations performed

- Detection limits including method detection limits, instrument detection limits, and quantitation limits
- Method of standard additions data
- ICP-AES inter-element correction (IEC) factors
- ICP-MS tunes
- ICP-MS internal standards relative intensity summary
- Any performance evaluation samples provided
- Standards preparation logs and certificates (if applicable)
- Percent solids or percent moisture for soil samples
- AA – wavelengths used for analysis

The following forms for all inorganic non-metals analyses: (as appropriate for analysis)

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references
- Inorganic analysis data sheet
- Calibration summaries
- Method blank results summary
- Sample spike recovery
- Duplicate sample results
- Laboratory Control Sample.
- Raw data, chromatograms, area printouts, sample preparation logs and, instrument run logs
- Examples of all calculations performed
- Documentation of all nonconformances and the actions taken
- Sample receipt information including temperature and pH information if preservation is required
- Standards preparation logs and certificates (if applicable)
- Percent solids or percent moisture for soil samples
- Logbook pages (gravimetric)
- Scale calibrations (gravimetric)

11.3.2 Reporting of Tentatively Identified Compounds

In addition to the target analytes identified for volatile and semivolatile organic stack gas analysis, there are generally a number of non-target components observed in the chromatogram. Attempts to identify and quantify these unknown chromatographic peaks can improve the percentage of identified organic

compounds and reduce overall uncertainty. However, because the instrument is not calibrated for these unknown compounds, the identification and quantitative analysis is tentative until the identification is confirmed by the analysis of a standard. EPA OSW risk assessment guidance recommends that TICs be considered “identified” compounds for purposes of site-specific risk assessments to ensure that appropriate credit is given to defensible efforts to identify the maximum number of organic compounds.

To identify non-target TICs, the mass spectrum can be searched against a computerized library of reference mass spectra. A forward library search selects the largest mass spectral peaks from the unknown mass spectrum and looks for reference spectra in the library that contain the peaks of the unknown. A reverse library search looks for the peaks in the reference spectrum that occur in the unknown mass spectrum. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library matches should the analyst assign a tentative identification. Any components that are identified are referred to as TICs, since no reference standard was analyzed at the same time as the unknown. Without calibration of the instrument with the actual compound, TICs are quantified using the nearest-eluting internal standard with a relative instrument response factor of 1.00. The resulting concentration is considered “estimated,” because the response factor is not compound-specific. An unknown level of error in the quantitation is introduced using the response factor of 1.00; this level of error will vary from compound to compound.

Methods 8260/8270 present guidelines for identification of TICs, and these guidelines are summarized below:

- Relative intensities of major ions in the reference mass spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample mass spectrum.
- The relative intensities of the major ions should agree within ± 20 percent. Example: for an ion with an abundance of 50 percent in the standard spectrum, the corresponding sample ion abundance should lie between 30 and 70 percent.
- Molecular ions present in the reference mass spectrum should be present in the sample mass spectrum.
- Ions present in the sample mass spectrum but not in the reference mass spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference mass spectrum but not in the sample mass spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library programs can sometimes create these discrepancies.

If, in the judgment of the experienced mass spectral interpreter, no valid tentative identification can be made, the compound should be reported as “unknown.” The mass spectral interpreter should give

additional classification of the unknown compound, if possible (i.e., unknown aromatic compound, unknown hydrocarbon, unknown chlorinated compound). If a probable molecular weight can be distinguished, this molecular weight should also be reported. The experienced interpreter should apply this experience and judgment to the mass spectral interpretations supplied by the computerized library search. For example, if a hydrocarbon occurring 40 minutes into the chromatographic analysis is identified by the computer as “octane,” analytical judgment dictates that this identification is scientifically illogical and the compound should be reported as “unknown hydrocarbon.” By no means should the computer identifications be accepted uncritically.

11.3.3 Project Reporting Format

The format for the PDT Report final report is outlined in Figure 11-1.

11.3.4 Detection Limits and Data Reduction

Detection limits for organic compounds will be derived as recommended by the U.S. EPA Office of Solid Waste (OSW) in the “Human Health Risk Assessment Protocol” published in July 1998. This protocol recommends that non-isotope dilution methods quantify non-detects using the method detection limit (MDL), derived reliable detection limits (RDL). The MDL is defined in 40 CFR Part 136 Appendix A. Each laboratory will be required to provide MDLs for each non-isotope dilution target compound along with the RDLs. Isotope dilution methods quantify non-detects using estimated detection limits (EDLs) as defined in SW-846 Method 8290.

Detection limits for inorganic compound compliance data will be derived according to the referenced analytical method and the laboratory's standard operating procedures. Each laboratory will be required to provide detection limits for each inorganic analyte.

Analytical results that are reported as “Not Detected” (ND) or “Below the detection Limit” (BDL) in compliance demonstration samples will be handled in the following manner. The analytical result will be reported as “ND”, “BDL”, or “U” and the appropriate detection limit as discussed above will be shown. The detection limit will be used as the assumed sample concentration in all subsequent calculations. For analyses of single component samples (e.g., feed or residue samples) that are reported as “ND” or “BDL”, the results of all subsequent calculations using those values will be accompanied by a “less than” (“<”) sign. For analyses of multi-component samples (e.g., VOST, Method 0010), where the analytical results must be combined for use in subsequent calculations, the detection limit will be used for each component reported as “ND” or “BDL”. Results of calculations utilizing values derived from multi-component analyses where the analyte of concern was non-detect in one or more components but not in all of the components, the result will be accompanied by a “<” sign. If the analyte of concern in a multi-component sample is non- detect in all the components of a particular sample, the result will be shown with a “<” sign,

and will also be marked as “ND” or “BDL”. Where destruction and removal efficiency or similar performance measurements are calculated using emissions rates reported with a “<” value, the resulting performance measurement will be accompanied by a “greater than” (“>”) sign.

For the purpose of risk assessment, if all results for a compound are found to be nondetect, one half of the detection limit will be use.

11.3.5 Final Case Files

At a minimum, the following documents will be retained upon the completion of the project in the final case file, which must be maintained at the WCAI facility for a period at least three years:

- All legal documents and orders,
- All field documents including those used for preliminary field activities,
- Copies of all analytical data,
- Copies of the final report and background documents, and
- All correspondence relating to the project as well as corrective action requests.

12.0 ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES

12.1 SAMPLING EQUIPMENT

All equipment used in emission testing measuring systems must be maintained in good operating order. To achieve this objective, a routine preventive maintenance program is necessary. Procedures used in this program follow those outlined in Maintenance Calibration and Operation of Isokinetic Source Sampling Equipment, Publication No. APTD-05-76 and Volume III of the Quality Assurance Handbook for Air Pollution Measurement Systems.

The potential impact of equipment malfunction on data completeness is minimized through two complementary approaches. First, an equipment maintenance program is part of routine operations. The maintenance program's strengths include:

- Trained technicians experienced in the details of equipment maintenance and fabrication,
- Adequate spare parts inventory, and
- The availability of tools and specialized equipment.

The second approach is based upon equipment redundancy. Backup equipment, spare parts and tools are included on the materials transported to the field for each sampling task. This approach allows the sampling team to respond to equipment breakage or malfunction in a timely fashion, minimizing the quantity of lost data.

For field equipment, preventive maintenance schedules are based on the results of routine inspections and on accumulated experience. At a minimum, equipment will be inspected prior to the beginning of and at the conclusion of each test. A record of each inspection (Figure 12-1) will be kept as part of the final case file. Maintenance schedules for continuous emissions monitors follow manufacturer's recommendations.

Each item of field test equipment is assigned a unique, permanent identification number. An effective preventive maintenance program is necessary to ensure data quality. Each item of equipment returning from the field is inspected before it is returned to storage. During the course of these inspections, items are cleaned, repaired, reconditioned and recalibrated where necessary. Each item of equipment transported to the field for this test program is inspected again before being packed to detect equipment problems that may originate during periods of storage. This minimizes lost time on the job site due to equipment failure. Occasional equipment failure in the field is unavoidable despite the most rigorous inspection and maintenance procedures. For this reason, adequate spare parts are kept in a central

location so the sampling contractor can quickly respond to the job site with replacement equipment for all critical sampling train components.

12.2 LABORATORY INSTRUMENTS

The laboratories perform regular maintenance on all analytical instruments. An inventory of replacement parts is kept to prevent downtime. Manufacturers' service representatives are also contracted, as required, for major instrument repairs.

Preventive and routine maintenance is covered in each of the laboratories' QA Manuals and SOPs or in accordance with manufacturer's recommendations (i.e., instrument manuals). Daily maintenance (such as replacement of injector septa, etc.) is covered in instrument SOPs. Inoperative equipment is tagged as non-usable until repairs are performed. Logbooks are maintained for each instrument to record usage, maintenance, and repairs.

12.3 PROCESS INSTRUMENTS

On-site personnel perform regular maintenance on all process instrumentation. Routine and preventive maintenance procedures are documented and updated as required. Where appropriate, manufacturers' recommendations for maintenance of process instruments are followed. Operators conduct daily reviews of process instrumentation by noting suspicious or inconsistent readings. Maintenance logs are used to record the frequency and type of repairs necessary for process instruments. Process instruments used to demonstrate compliance with operating limits will be calibrated prior to the test. Records of these calibrations will be included in the final test report.

13.0 ASSESSMENT PROCEDURES FOR ACCURACY, PRECISION, & COMPLETENESS

The QA activities implemented in this study will provide a basis for assessing the accuracy and precision of the analytical measurements. Section 5.0 discusses the QA activities that will generate the accuracy and precision data for each sample type. The generalized forms of the equations that will be used to calculate accuracy and precision are presented below.

13.1 ACCURACY

When a reference standard material is used in the analysis, percent Accuracy (A) will be calculated as follows:

$$A = \frac{\text{Found concentration}}{\text{True concentration}} \times 100$$

Percent analyte Recovery (R) will be calculated as follows:

$$R = \frac{X - N}{S} \times 100$$

Where X is the experimentally determined value, N is the amount of native material in the sample, and S is the amount of spiked material of the species being measured. Recoveries are used to determine accuracy when standards are not available, or are not appropriate for a given matrix.

13.2 PRECISION

When less than three analyses of the same parameter are available, precision will be calculated as a Relative Percent Difference (RPD) from the average of replicate measurements according to:

$$RPD = \frac{(X_1 - X_2)}{\text{Average } X} \times 100$$

Where X_1 and X_2 are the highest and lowest results of replicate measurements.

Where three or more analyses of the same parameter are available, the precision will be determined as the Relative Standard Deviation (RSD) according to:

$$\text{RSD} = \frac{\text{Standard deviation}}{\text{Average X}} \times 100$$

13.3 COMPLETENESS

Completeness of data generated from a test program is usually calculated as follows:

$$\% \text{ Completeness} = \frac{\text{Valid data}}{\text{Expected data}} \times 100$$

Data completeness is defined in Section 5.0 of this QAPP as the percentage of valid data collected from the total number of valid tests conducted. Three valid test runs, at each test condition, are required for the test to be completed. If an individual sample from a test run is lost or broken, the data for that individual analytical parameter may not be 100% complete. This, however, may not invalidate the test run. The completeness objective for this test program is to generate sufficient data for the regulatory agencies to judge the performance of the system.

14.0 AUDIT PROCEDURES, CORRECTIVE ACTION, AND QA REPORTING

14.1 PERFORMANCE AND SYSTEM AUDITS

This section presents information related to the procedures used by the QA staff to assess conformance of the project staff to the specifications contained in the relevant project controlling documents. Further, auditing may be employed to assess the ability of subcontractors to successfully perform the work.

14.1.1 Field Audits

The QAO assigned to the project will conduct audits of the operations at the site to ensure that work is being performed in accordance with the various project controlling documents and associated standard operating procedures. A checklist appropriate to the activities scheduled during the audit will be used. The audit will cover, but not necessarily be limited to, such areas as:

- Conformance to SOPs
- Completeness and accuracy of documentation
- Chain of custody procedures
- Compliance with Health and Safety requirements.

These audits will occur at the start or end of each significant phase of the project.

14.1.2 Performance Evaluations

The QAO and/or the Regulatory Agencies may submit Performance Evaluation (PE) samples (referred to elsewhere as "Audit Samples") to the laboratory as indicated on Tables 5-1 and 6-2. PE samples may be submitted for analysis to demonstrate analytical performance on an as required basis.

14.1.3 Office Audits

The QAO will also conduct periodic audits of the case files. These audits will assess the completeness of the files and verify that all of the appropriate information is included in the files.

14.1.4 Laboratory Audits

WCAI or its appointed representative may choose to audit the laboratories at any time during the course of the project on an as-required basis to assess the laboratory's ability to successfully perform the work and to ensure mutual agreement between WCAI and the laboratory with regard to the scope of work, QA/QC requirements, and deliverable requirements. Reasonable notice will be provided prior to any on-site inspection of the laboratory.

14.2 CORRECTIVE ACTION

The following procedures have been established to ensure that nonconforming conditions, such as malfunctions, deficiencies, deviations and errors are promptly investigated, documented, evaluated and corrected. Every person employed in the test is expected to function as a QC inspector to ensure the quality of the final product. Quality, as it relates to this project, is defined as "performing the work according to the agreed upon specifications contained in the PDT plan and relevant SOPs or causing the specification to be changed *in a controlled manner*." Each individual is encouraged to identify any condition adverse to the successful completion of the work or any modification to the specifications that might result in a better end product. These improvements might be framed in terms of higher quality, greater safety, greater efficiency, and/or lower cost. However, it can not be stressed strongly enough, that only documented and approved changes to the specifications are allowable.

14.2.1 Field

When a nonconforming condition or an opportunity for improvement is noted at the site or contractor location, the corrective action provisions of this plan will be invoked to identify the condition and recommend corrective action. Condition identification, cause, reference documents and the corrective action planned to be taken will be documented and reported at a minimum to the employee's immediate supervisor.

A Corrective Action Request (CAR), as shown in Figure 14-1, should be used to identify the adverse condition or opportunity for improvement, reference document(s) and recommended corrective action(s). The CAR is directed to the Test Coordinator. The Test Coordinator affixes his signature and the date to the corrective action block that states the cause of the condition(s) and corrective action(s) to be taken. The Test Coordinator is responsible for first notifying the regulatory agency representative of any problems or deviations from the QAPP, or PDT plan identified in the CAR. The Test Coordinator then forwards the requested response to the QAO for follow-up and filing. The QAO maintains the log for status control of CARs and responses confirms the adequacy of the intended corrective action(s) and verifies its implementation. The QAO will issue and distribute copies of completed CARs to the originator, Test Coordinator, WCAI Test Project Manager, and the involved contractor(s) if any. CARs are transmitted to the project file for future reference, and are incorporated into the final test report.

Testing activities may be impacted by a number of factors, including process interruptions, operating conditions which are outside of specifications, inclement weather, or sampling train difficulties. A set of field troubleshooting guidelines has been developed and presented in Table 14-1 to assist in recognizing and resolving these issues in the field.

14.2.2 Laboratory

The laboratories' QA Manuals and the related SOPs, contain detailed discussions of corrective actions to be taken if established criteria fail during laboratory analysis. The laboratory has the responsibility to immediately notify the Test Coordinator and/or QAO when any analytical QC nonconformance occurs, so a mutually acceptable course of action can be pursued.

14.3 QA REPORTS TO MANAGEMENT

The QAO will provide a written report to the Test Coordinator. This report will address:

- Overview of activities and significant events related to QA/QC
- Summary of audit results
- Review of corrective action request status
- Laboratory QA/QC reports
- Data validation reports
- Summary of significant changes in procedures or QA/QC programs
- Recommendations.

Upon project completion, a Final QA Report will be issued, assessing the overall degree of project conformance to specifications and the impact of any nonconforming conditions on data quality that may affect management decisions. This report will be incorporated into the final test report.

The nature of the laboratories' Quality Assurance reports is provided in their respective Laboratory Quality Assurance Manuals and SOPs. Where no other specifications exists, the laboratory must conform to the provisions given in this section.

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Spent Activated Carbon	Chloride & Elemental analysis	90 – 110% of reference value for analysis of a known material conducted once per test series for each property of concern	<10% RPD for duplicate analyses conducted for one sample from each matrix. (viscosity – every sample in duplicate)	An optional matrix spike/matrix spike duplicate may be conducted if desired. If conducted, the desired spiking level is 2 times the native concentration or 10 times the detection limit, whichever is greater. Accuracy and precision objectives are as stated for reference material and duplicate analyses.
Spent Activated Carbon, Makeup Water, Scrubber Blowdown, POTW Discharge & Caustic	Volatile organics	Matrix Spike % recoveries as specified in Table 5-2 Surrogate % recoveries (Table 5-2) spiked onto every field sample	< 35% RPD for duplicate preparation and analysis conducted for one sample from each matrix <35% PRD for matrix spike/matrix spike duplicate analysis	Analysis of one method blank per sample batch carried through all preparation and analysis steps. Results should be less than the lowest calibration standard.
Spent Activated Carbon, Makeup Water, Scrubber blowdown, POTW Discharge & Caustic	Semivolatile organics	50 - 130% recovery of isotopically labeled POHCs or appropriate surrogates spiked into every sample prior to preparation and analysis. 50 - 130% recovery of each POHC spiked into an aliquot of one sample from each matrix at 2 times the apparent concentration in the unspiked sample or 10 times the detection limit, whichever is greater. (Matrix Spike).	< 35% RSD of spike recoveries between samples for each matrix. < 35% RPD for duplicate analyses conducted for one sample from each matrix. < 35% RPD for duplicate analysis of sample spiked with POHCs at 2X or 10X level. (Matrix Spike Duplicate).	Analysis of one method blank per sample batch, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Spent Activated Carbon, Makeup Water, Scrubber blowdown, POTW Discharge & Caustic	Metals	70 - 130% recovery of each metal of concern spiked into an aliquot of one sample from each matrix. The spiking level should be the greater of either 1 - 2 times the apparent concentration in the unspiked sample, or at least 10 times the detection limits. (Matrix Spike).	<p>< 35% RPD for duplicate analyses conducted for one sample from each matrix. This criterion only applies to individual metals with an apparent concentration greater than the lowest calibration standard used in the analyses.</p> <p>and/or</p> <p>< 35% RPD for duplicate analysis of the spiked sample. (Matrix Spike Duplicate).</p>	Analysis of one method blank per sample batch, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	Volatile Organics (Method 0030, VOST)	<p>75 - 125% recovery of standards (independent of calibration standards) spiked onto 2 VOST tube pairs and analyzed prior to sample analysis.</p> <p>Surrogate % recoveries as specified in Table 5-2 spiked onto every field sample (VOST tubes and condensate).</p> <p>50 - 150% of true value for analysis of samples collected from EPA audit cylinder (if requested and provided).</p>	<p>< 25% RSD between spike recoveries from 2 VOST tube pairs analyzed prior to sample analysis.</p> <p>< 35% RSD of surrogate spike recoveries between each field sample.</p>	<p>Separate analysis of front and back tubes from each pair should show less than 30% of the front tube concentration on the back tube. This criterion is not applicable for a particular compound if the back tube contains less than 75 ng of that compound.</p> <p>One pair of field blank tubes is analyzed for every test run (6 samples). Should be less than the lowest calibration standard.</p> <p>One pair of trip blank tubes, accompanying each tube shipment from the field, should be analyzed if the field blanks show contamination. Should be less than the lowest calibration standard.</p> <p>One pair of laboratory blank tubes (prepared in the same batch as the field sample tubes, and archived) should be analyzed if trip blanks show contamination. Should be less than the lowest standard.</p> <p>System blanks are analyzed daily before sample analysis, and between high-level samples. Should be less than the lowest standard.</p>

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	PCDD/PCDFs (Method 0023A)	<p>70 - 130% recovery of isotopically labeled PCDD/PCDF pre-sampling surrogates spiked onto each sorbent resin tube prior to sampling.</p> <p>40 - 130% recovery of isotopically labeled tetra- through hexa-chlorinated PCDD/PCDF internal surrogate standards spiked onto train components prior to extraction.</p> <p>25 - 130% recovery of isotopically labeled hepta- and octa- chlorinated PCDD/PCDF internal surrogate standards spiked onto train components prior to extraction.</p> <p>50 – 150% recovery of isotopically labeled alternate standards spiked onto the extract prior to cleanup standards and recovery standards spiked onto the extract following cleanup.</p> <p>50 - 150% of true value for analysis of an audit sample (if requested/provided)</p>	<p>< 30% RSD of spike recoveries between samples for labeled compounds spiked prior to sampling.</p> <p>< 60% RSD of spike recoveries between samples for internal quantitation standards</p>	<p>Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train is performed to assess contamination.</p> <p>Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be conducted to assess contamination.</p>

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	Hexavalent chromium (Method 0061)	<p>60 - 140% recovery of Cr⁺⁶ spiked into one sample preparation. The spiking level should be the greater of either 1 - 2 times the apparent concentration in the unspiked sample, or at least 10 times the detection limits.</p> <p>90 - 110% of the true value for analysis of an audit sample obtained from the regulatory agency (if requested).</p>	< 30% RPD for duplicate analysis of every sample including the spiked sample.	Analysis of one set of reagent blanks, carried through all preparation and analysis steps, should be less than 5% of sample levels or below the detection limit.
Stack gas	Semivolatile organics (Method 0010)	<p>Recovery of isotopically labeled POHCs or appropriate surrogates spiked into each sample as listed in Table 5-2</p> <p>Recovery of POHCs or appropriate surrogates spiked onto a blank XAD-2 resin trap and one condensate sample in the laboratory as specified in Table 5-2. (Matrix Spike)</p>	<p>< 40% RPD (or < 35% RSD, if greater than 4 determinations are made) of spike recoveries between field samples.</p> <p>< 50% RPD for duplicate injection from one run. This criterion is relaxed to < 100% RPD if the compound is found at a concentration below the lowest calibration standard.</p> <p>< 50% RPD for duplicate preparation and analysis of spiked blank XAD-2 resin trap. (Matrix Spike Duplicate)</p>	<p>Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train should be less than 20% of the sample levels or below the detection limit.</p> <p>Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.</p>

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	PCBs (Method 0010) (Draft EPA Method 1668A)	<p>Recovery of isotopically labeled PCBs or appropriate surrogates spiked into each train component prior to extraction as specified in Table 5-2.</p> <p>Recovery of PCBs spiked onto a blank XAD-2 resin trap and one condensate sample in the laboratory as specified in Table 5-2. (Matrix Spike)</p>	<p>< 40% RPD (or < 35% RSD, if greater than 4 determinations are made) of spike recoveries between field samples.</p> <p>< 50% RPD for duplicate injection from one run. This criterion is relaxed to < 100% RPD if the particular compound is found at a concentration below the lowest calibration standard.</p> <p>< 50% RPD for duplicate preparation and analysis of spiked blank XAD-2 resin trap. (Matrix Spike Duplicate)</p>	<p>Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train should be less than 20% of the sample levels or below the detection limit.</p> <p>Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.</p>
Stack gas	PAHs (Method 0010) (CARB Method 429)	<p>50 - 150% recovery of isotopically labeled PAH compounds spiked onto each sorbent resin tube prior to sampling</p> <p>50 - 150% recovery of isotopically labeled internal quantitation standards spiked onto train components prior to extraction</p> <p>50 - 150% recovery of PCBs spiked onto a blank XAD-2 resin trap and one condensate sample in the laboratory. (Matrix Spike)</p>	<p>< 30% RSD of spike recoveries between samples for labeled compounds spiked prior to sampling.</p> <p>< 60% RSD of spike recoveries between samples for internal quantitation standards</p>	<p>Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train is performed to assess contamination</p> <p>Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be conducted to assess contamination</p>

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	OCP (Method 0010) (SW846 Method 8081)	<p>Recovery of appropriate surrogates spiked into each sample as listed in Table 5-2</p> <p>Recovery of appropriate surrogates spiked onto a blank XAD-2 resin trap and one condensate sample in the laboratory as specified in Table 5-2. (Matrix Spike)</p>	<p>< 40% RPD (or < 35% RSD, if greater than 4 determinations are made) of spike recoveries between field samples.</p> <p>< 50% RPD for duplicate injection from one run. This criterion is relaxed to < 100% RPD if the compound is found at a concentration below the lowest calibration standard.</p> <p>< 50% RPD for duplicate preparation and analysis of spiked blank XAD-2 resin trap. (Matrix Spike Duplicate)</p>	<p>Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train should be less than 20% of the sample levels or below the detection limit.</p> <p>Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.</p>
Stack gas	Total semivolatile and nonvolatile organics (Method 0010)	<p>TCO – Daily QC sample \pm 15% of actual value</p> <p>GRAV – Audit sample \pm 20% of actual value</p>	<p>TCO - <15% RPD between analysis of one replicate sample per test</p> <p>GRAV - <20% RPD between duplicate analysis of each sample.</p>	<p>Analysis of one method blank for recovery reagents and XAD/filter carried through all preparation and analysis steps should be conducted to assess contamination.</p> <p>Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train is performed to assess contamination</p>

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	Total volatile organics (Method 0040)	Tedlar Bag - 80 - 120% of the true value for analysis of a gas with known hydrocarbon concentration placed into a clean bag and analyzed prior to sample analysis.	< 50% RPD for duplicate analysis of known gas	Analyze one gas bag filled with zero air or zero nitrogen, and one water blank with each field sample should be less than 20% of the sample levels or below the detection limit. Results are evaluated on a case-by-case basis for possible blank correction.
Stack Gas	Metals (Method 29)	<p>75 - 125% recoveries for each metal of concern spiked into an aliquot of one sample preparation from each train component. The spiking level should be the greater of either 1 - 2 times the apparent concentration in the unspiked sample, or at least 10 times the detection limits.</p> <p>70 - 125 % recoveries for spiked blank filters for each metal of concern.</p> <p>75 - 125% recoveries for preparation and analysis of two complete blank trains spiked with each metal of concern. The spiking level should be the greater of either 1 - 2 times the expected concentration in the field samples, or at least 10 times the detection limit.</p>	<p>< 20% RPD between analyses of the two blank spiked filters for each metal of concern.</p> <p>< 20% RPD between analyses of the two spiked blank trains.</p> <p>For mercury only, < 25% RPD for duplicate analysis conducted for every sample.</p>	Analysis of one set of reagent blanks, carried through all preparation and analysis steps. Results are evaluated on a case-by-case basis for possible blank correction.
Stack Gas	Particulate (Method 26A)	99 – 101% agreement for balance calibration check with Class-S weights prior to and after all tare weighing and after all tare weighing and gravimetric determinations.	Duplicate weighing of each sample must be within 0.5 mg or 1% total tare weight, whichever is greater.	None

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	HCl/Cl ₂ (Method 26A)	85 – 115% recovery of chloride spiked into an aliquot of both acidified and alkaline impinger solutions at less than 3 times the apparent concentration in the unspiked sample or 10 times the detection limit, whichever is greater. (Matrix Spike)	<25% RPD for duplicate analysis conducted for each acidified and each alkaline impinger solution sample. This criterion is relaxed to <50% RPD if the sample concentration is less than 5 times the detection limit. <25% RPD for duplicate analysis of each spiked sample. (MS/MSD)	Analysis of one method blank analyzed in duplicate per sample batch, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.
Stack gas	Particle Size distribution (PSD) via Cascade Impactor (CARB Method 501)	N/A	N/A	Analysis of one set of blank filter media to establish analysis baseline.
Stack gas	Carbon monoxide (permanent CEMS)	Daily calibration checks (high and low range) within 3% of span.	40 CFR 63 Subpart EEE Appendix; PA Continuous Source Monitoring Manual	N/A
Stack gas	Oxygen (permanent CEMS)	Daily calibration checks within 0.5% oxygen.	40 CFR 63 Subpart EEE Appendix; PA Continuous Source Monitoring Manual	N/A
Stack gas	THC/VOC (Temporary CEMS)	Pre- and Post-run calibration checks within 3% of respective span calibration gases	40 CFR 63 Subpart EEE Appendix; PA Continuous Source Monitoring Manual; EPA Method 25A	N/A

$$\text{Relative Percent Difference (RPD)} = \frac{\text{highest value} - \text{lowest value}}{\text{average value}} \times 100$$

$$\% \text{ Recovery} = \frac{\text{found} - \text{native}}{\text{amount spiked}} \times 100$$

$$\% \text{ Accuracy} = \frac{\text{found concentration}}{\text{actual concentration}} \times 100 \text{ (for reference materials)}$$

$$\text{Relative Standard Deviation} = \frac{\text{standard deviation}}{\text{average value}} \times 100$$

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits	
Spent activated carbon feed, Makeup Water, Scrubber Blowdown, POTW Discharge & Caustic	Volatile Organics in Organic & Aqueous Liquid Matrices and Solid Matrices	Surrogate Spikes	Toluene-d ₈	50 – 130%
			4-Bromofluorobenzene	50 – 130%
			1,2-Dichloroethane-d ₄	50 – 130%
	Matrix Spikes	Chlorobenzene	50 – 130%	
		Tetrachloroethene	50 – 130%	
		1,1-Dichloroethene	50 – 130%	
		Trichloroethene	50 – 130%	
		Benzene	50 – 130%	
	Internal Standards (area count compared to Continuing calibration)	Toluene	50 – 130%	
		Fluorobenzene	50 – 200%	
Chlorobenzene-d ₅		50 – 200%		
Makeup Water, Scrubber blowdown, POTW Discharge & Caustic	Semivolatile Organics in Organic & Aqueous Liquid Matrices	Surrogate Spikes	1,4-dichlorobenzene-d ₄	50 – 200%
			Nitrobenzene-d ₅	23 - 120%
			2-Fluorobiphenyl	30 - 115%
			Phenol-d ₅	24 - 113%
			2-Fluorophenol	25 - 121%
	Internal Standards (area count compared to Continuing calibration)	2,4,6-Tribromophenol	19 - 122%	
		1,4-Dichlorobenzene-d ₄	50 – 200%	
		Naphthalene-d ₈	50 – 200%	
		Acenaphthene-d ₁₀	50 – 200%	
		Phenanthrene-d ₁₀	50 – 200%	
Chrysene-d ₁₂	50 – 200%			
Perylene-d ₁₂	50 – 200%			

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Spent activated carbon feed	Semi-volatile Organics Solid Matrices		
	Sampling Surrogate (to XAD-2 resin before field use)	¹³ C ₃ -labeled Naphthalene	50 - 150%
	Surrogate Spikes (to sample fraction before extraction)	Nitrobenzene-d ₅	23 - 120%
		2-Fluorobiphenyl	30 - 115%
		Phenol-d ₅	24 - 113%
		2-Fluorophenol	25 - 121%
		2,4,6-Tribromophenol	19 - 122%
	Matrix Spikes (Blank Spiked Resin)	Phenol	25 - 150%
		2-Chlorophenol	26 - 150%
		1,4-Dinitrophenol	28 - 150%
		N-Nitroso-di-n-propylamine	41 - 150%
		1,2,4-Trichlorobenzene	38 - 150%
		4-Chloro-3-methylphenol	26 - 150%
		Acenaphthene	31 - 150%
		4-Nitrophenol	11 - 150%
2,4-Dinitrotoluene		28 - 150%	
Pentachlorotoluene		17 - 150%	
Pyrene	35 - 150%		
Internal Standards (area count compared to Continuing calibration)	1,4-Dichlorobenzene-d ₄	50 - 200%	
	Naphthalene-d ₈	50 - 200%	
	Acenaphthene-d ₁₀	50 - 200%	
	Phenanthrene-d ₁₀	50 - 200%	
	Chrysene-d ₁₂	50 - 200%	
Perylene-d ₁₂	50 - 200%		
Stack Gas Volatile Organics (Method 0030)	VOST Solid Matrices		
	Surrogate Spikes	Toluene-d ₈	50 - 130%
		4-Bromofluorobenzene	50 - 130%
		1,2-Dichloroethane-d ₄	50 - 130%
	Internal Standards (area count compared to Continuing calibration)	Fluorobenzene	50 - 200%
		Chlorobenzene-d ₅	50 - 200%
		1,4-dichlorobenzene-d ₄	50 - 200%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas Volatile Organics (Method 0030)	VOST Condensate (Aqueous Matrices)		
	Surrogate Spikes	Toluene-d ₈	50 – 130%
		4-Bromofluorobenzene	50 – 130%
		1,2-Dichloroethane-d ₄	50 – 130%
	Matrix Spikes (or Method Spike)	1,1-Dichloroethene	50 – 130%
		Trichloroethene	50 – 130%
		Benzene	50 – 130%
		Toluene	50 – 130%
		Chlorobenzene	50 – 130%
	Internal Standards (area count compared to Continuing calibration)	Fluorobenzene	50 – 200%
Chlorobenzene-d ₅		50 – 200%	
1,4-dichlorobenzene-d ₄		50 – 200%	
Stack Gas PCDD/PCDFs (Method 0023A)	Pre-sampling Surrogate Spikes (to XAD-2 resin before field use and to the filter immediately prior to extraction)	³⁷ Cl ₄ -2,3,7,8-TCDD	70 – 130%
		¹³ C ₁₂ -2,3,4,7,8-PeCDF	70 – 130%
		¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	70 – 130%
		¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	70 – 130%
		¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	70 – 130%
	Internal Surrogate Spikes (to sample fraction before extraction)	¹³ C ₁₂ -2,3,7,8-TCDF	40 – 130%
		¹³ C ₁₂ -2,3,7,8-TCDD	40 – 130%
		¹³ C ₁₂ -1,2,3,7,8-PeCDF	40 – 130%
		¹³ C ₁₂ -1,2,3,7,8-PeCDD	40 – 130%
		¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	40 – 130%
		¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	40 – 130%
		¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	25 – 130%
		¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	25 – 130%
	¹³ C ₁₂ -OCDD	25 – 130%	
	Alternate Standard (to extract before cleanup)	¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	50 – 150%
Recovery Standards (to extract before analysis)	¹³ C ₁₂ -1,2,3,4-TCDD	50 – 150%	
	¹³ C ₁₂ -1,2,3,7,8,9-HxCDD	50 – 150%	

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas Semivolatile Organics (Method 0010)	Semi-volatile Organics Solid Matrices (Filter and XAD-2 Resin)		
	Sampling Surrogate (to XAD-2 resin before field use)	¹³ C ₃ -labeled Naphthalene	50 - 150%
	Surrogate Spikes (to sample fraction before extraction)	Nitrobenzene-d ₅	23 - 120%
		2-Fluorobiphenyl	30 - 115%
		Phenol-d ₅	24 - 113%
		2-Fluorophenol	25 - 121%
		2,4,6-Tribromophenol	19 - 122%
	Matrix Spikes (Blank Spiked Resin)	Phenol	25 - 150%
		2-Chlorophenol	26 - 150%
		1,4-Dinitrophenol	28 - 150%
		N-Nitroso-di-n-propylamine	41 - 150%
		1,2,4-Trichlorobenzene	38 - 150%
		4-Chloro-3-methylphenol	26 - 150%
		Acenaphthene	31 - 150%
		4-Nitrophenol	11 - 150%
2,4-Dinitrotoluene		28 - 150%	
Pentachlorotoluene		17 - 150%	
Pyrene		35 - 150%	
Internal Standards (area count compared to Continuing calibration)	1,4-Dichlorobenzene-d ₄	50 - 200%	
	Naphthalene-d ₈	50 - 200%	
	Acenaphthene-d ₁₀	50 - 200%	
	Phenanthrene-d ₁₀	50 - 200%	
	Chrysene-d ₁₂	50 - 200%	
Perylene-d ₁₂	50 - 200%		
Stack Gas Semivolatile Organics (Method 0010)	Semi-volatile Organics Aqueous Matrices (Condensate)		
	Surrogate Spikes (to sample fraction before extraction)	Nitrobenzene-d ₅	35 - 114%
		2-Fluorobiphenyl	43 - 116%
		Phenol-d ₅	10 - 94%
		2-Fluorophenol	21 - 100%
		2,4,6-Tribromophenol	10 - 123%
	Matrix Spikes or Method Spike)	Phenol	12 - 150%
		2-Chlorophenol	27 - 150%
		1,4-Dinitrophenol	36 - 150%
		N-Nitroso-di-n-propylamine	41 - 150%
		1,2,4-Trichlorobenzene	39 - 150%
		4-Chloro-3-methylphenol	23 - 150%
		Acenaphthene	46 - 150%
		4-Nitrophenol	10 - 150%
		2,4-Dinitrotoluene	24 - 150%
Pentachlorotoluene		9 - 150%	
Pyrene	26 - 150%		

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas PCBs (Method 0010)	PCBs Solid or Aqueous Matrices (Filter and XAD-2 Resin, or Condensate)		
	Sampling Surrogate Spikes * (to XAD-2 resin before field use)	¹³ C ₁₂ -2,4,4'-Tri-CB ¹³ C ₁₂ -2,3,3',5,5'-PeCB ¹³ C ₁₂ -2,2',3,3',5,5',6-HpCB	60 - 140% 60 - 140% 60 - 140%
	Surrogate Spikes (to sample fraction before extraction)	¹³ C ₁₂ -3,3',4,4'-TCB ¹³ C ₁₂ -2,3,3',4,4'-PeCB ¹³ C ₁₂ -2,3',4,4',5-PeCB ¹³ C ₁₂ -3,3',4,4',5-PeCB ¹³ C ₁₂ -2,3,3',4,4',5-HxCB ¹³ C ₁₂ -2,3,3',4,4',5'HxCB ¹³ C ₁₂ -2,3',4,4',5,5'-HxCB ¹³ C ₁₂ -3,3',4,4',5,5'-HxCB ¹³ C ₁₂ -2,2',3,4,4',5,5'-HpCB ¹³ C ₁₂ -2,3,3',4,4',5,5'-HpCB ¹³ C ₁₂ -DCB ^a	24 – 169% 21 – 178% 21 – 178% 21 – 178% 26 – 152% 26 – 152% 26 – 152% 26 – 152% 26 – 152% 23 – 143% 23 – 143% 26 - 152%
	Cleanup Standard Spikes (to extract before cleanup)	¹³ C ₁₂ -3,4,4',5-TCB ¹³ C ₁₂ -2,3,4',5,5'-PeCB	35 – 197% 35 – 197%
	Internal Standard Spikes * (to extract after cleanup and before analysis)	¹³ C ₁₂ -2,2',5,5'-TCB ¹³ C ₁₂ -2,2',4,5,5'-PeCB ¹³ C ₁₂ -2,2',3,4,4',5'-HxCB ¹³ C ₁₂ -2,2',3,3',5,5',6-HpCB	40 – 120% 40 – 120% 40 – 120% 40 – 120%
	Matrix Spike * (blank spiked resin)	3,3',4,4'-TCB 2,3,3',4,4'-PeCB 2,3,4,4',5-PeCB 2,3,4,4',5-PeCB 2',3,4,4',5-PeCB 3,3',4,4',5-PeCB 2,3,3',4,4',5-HxCB 2,3,3',4,4',5'-HxCB 2,3',4,4',5,5'-HxCB 3,3',4,4',5,5'-HxCB 2,2',3,3',4,4',5-HpCB 2,2',3,4,4',5,5'-HpCB 2,3,3',4,4',5,5'-HpCB	60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas OCPs (Method 0010)	Organochlorine Pesticides Solid Matrices (Filter and XAD-2 Resin)		
	Sampling Surrogate (to XAD-2 resin before field use)		
	Surrogate Spikes (to sample fraction before extraction)	Decachlorobiphenyl Tetrachloro-m-xylene	50 – 150% 50 – 150%
	Matrix Spikes (Blank Spiked Resin)	Aldrin	70 – 130%
		α -BHC	70 – 130%
		β -BHC	70 – 130%
		γ -BHC	70 – 130%
		δ -BHC	70 – 130%
		Chlorobenzilate	70 – 130%
		α -Chlordane	70 – 130%
		γ -Chlordane	70 – 130%
		DBCP	70 – 130%
		4,4'-DDD	70 – 130%
		4,4'-DDE	70 – 130%
		4,4'-DDT	70 – 130%
		Diallate	70 – 130%
		Dieldrin	70 – 130%
		Endosulfan I	70 – 130%
		Endosulfan II	70 – 130%
		Endosulfan sulfate	70 – 130%
Endrin		70 – 130%	
Endrin aldehyde	70 – 130%		
Endrin ketone	70 – 130%		
Heptachlor	70 – 130%		
Heptachlor epoxide	70 – 130%		
Isodrin	70 – 130%		
Methoxychlor	70 – 130%		
Toxaphene	70 – 130%		
		70 – 130%	
Internal Standards (optional) (area count compared to Continuing calibration)	Pentachloronitrobenzene	50 – 200%	

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas OCP (Method 0010)	Organochlorine Pesticides Aqueous Matrices (Condensate)		
	Surrogate Spikes (to sample fraction before extraction)	Decachlorobiphenyl Tetrachloro-m-xylene	50 – 150% 50 – 150%
	Matrix Spikes (or Method Spike)	Aldrin	70 – 130%
		α -BHC	70 – 130%
		β -BHC	70 – 130%
		γ -BHC	70 – 130%
		δ -BHC	70 – 130%
		Chlorobenzilate	70 – 130%
		α -Chlordane	70 – 130%
		γ -Chlordane	70 – 130%
		DBCP	70 – 130%
		4,4'-DDD	70 – 130%
		4,4'-DDE	70 – 130%
4,4'-DDT	70 – 130%		
Diallate	70 – 130%		
Dieldrin	70 – 130%		
Endosulfan I	70 – 130%		
Endosulfan II	70 – 130%		
Endosulfan sulfate	70 – 130%		
Endrin	70 – 130%		
Endrin aldehyde	70 – 130%		
Endrin ketone	70 – 130%		
Heptachlor	70 – 130%		
Heptachlor epoxide	70 – 130%		
Isodrin	70 – 130%		
Methoxychlor	70 – 130%		
Toxaphene	70 – 130%		
Internal Standards (optional) (area count compared to Continuing calibration)	Pentachloronitrobenzene	50 – 200%	

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits	
Stack Gas PAHs (Method 0010)	PAH Solid or Aqueous Matrices (Filter and XAD-2 Resin, or Condensate)			
	Sampling Surrogate (to XAD-2 resin before field use)	d ₁₀ -Fluorene d ₁₄ -Terphenyl	50 – 150% 50 – 150%	
	Internal Standard Spikes (to sample fraction before extraction)		Naphthalene-d ₈	50 – 150%
			2-Methylnaphthalene	50 – 150%
			Acenaphthene-d ₁₀	50 – 150%
			Phenanthrene-d ₁₀	50 – 150%
			Fluoranthene-d ₁₀	50 – 150%
			Benzo(a)anthracene-d ₁₂	50 – 150%
			Chrysene-d ₁₂	50 – 150%
			Perylene-d ₁₂	50 – 150%
			Benzo(b)fluoranthene-d ₁₂	50 – 150%
			Benzo(d)fluoranthene-d ₁₂	50 – 150%
			Benzo(a)pyrene-d ₁₂	50 – 150%
			Benzo(g,h,i)perylene-d ₁₂	50 – 150%
			Indeno(1,2,3-c,d)pyrene-d ₁₂	50 – 150%
	Dibenzo(a,h)anthracene-d ₁₄	50 – 150%		
	Alternate Standard (add before extraction)	Anthracene-d ₁₀	50 – 150%	
	Recovery Standards (after extraction and before GC/MS)		Acenaphthene-d ₁₀	50 – 150%
			Pyrene-d ₁₀	50 – 150%
			Benzo(e)pyrene-d ₁₂	50 – 150%
	Matrix Spike (blank spiked resin)		Naphthalene	50 – 150%
			2-Methylnaphthalene	50 – 150%
			Acenaphthalene	50 – 150%
Acenaphthylene			50 – 150%	
Fluorene			50 – 150%	
Phenanthrene			50 – 150%	
Anthracene			50 – 150%	
Fluoranthene			50 – 150%	
Pyrene			50 – 150%	
Benzo(a)anthracene			50 – 150%	
Chrysene			50 – 150%	
Benzo(b)fluoranthene			50 – 150%	
Benzo(k)fluoranthene			50 – 150%	
Benzo(e)pyrene			50 – 150%	
Benzo(a)pyrene			50 – 150%	
Perylene			50 – 150%	
Indeno(1,2,3-cd)pyrene			50 – 150%	
Dibenz(a,h)anthracene	50 – 150%			
Benzo(ghi)perylene	50 – 150%			

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

* CARB 428 limits. None provided in EPA Method 1668A

a No limits provided for this surrogate. The most stringent limits from the other surrogates were used.

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
VOLATILE ORGANICS					
Acetone	67-64-1	2.00E-01	9.44E-02	3.33E+00	6.10E-06
Acrylonitrile	107-13-1	9.00E-01	4.25E-01	1.50E+01	2.75E-05
Benzene	71-43-2	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Bromochloromethane	74-97-5	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Bromodichloromethane	75-27-4	4.00E-02	1.89E-02	6.67E-01	1.22E-06
Bromoform	75-25-2	6.00E-02	2.83E-02	1.00E+00	1.83E-06
Bromomethane	74-83-9	5.00E-02	2.36E-02	8.33E-01	1.53E-06
2-Butanone (MEK)	78-93-3	3.00E-01	1.42E-01	5.00E+00	9.15E-06
Carbon Disulfide	75-15-0	3.00E-02	1.42E-02	5.00E-01	9.15E-07
Carbon Tetrachloride	56-23-5	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Chlorobenzene	108-90-7	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Chlorodibromomethane	124-48-1	6.00E-02	2.83E-02	1.00E+00	1.83E-06
Chloroethane	75-00-3	4.00E-02	1.89E-02	6.67E-01	1.22E-06
Chloroform	67-66-3	3.00E-02	1.42E-02	5.00E-01	9.15E-07
Chloromethane	74-87-3	1.00E+00	4.72E-01	1.67E+01	3.05E-05
1,2-Dibromoethane (a)	106-93-4				
Dibromomethane	74-95-3	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Dichlorodifluoromethane	75-71-8	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1-Dichloroethane	75-34-3	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,2-Dichloroethane	107-06-2	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1-Dichloroethene	75-35-4	4.00E-02	1.89E-02	6.67E-01	1.22E-06
1,2-Dichloropropane	78-87-5	1.00E+00	4.72E-01	1.67E+01	3.05E-05
Dicyclopentadiene (a)	77-73-6				
Ethylbenzene	100-41-4	3.00E-02	1.42E-02	5.00E-01	9.15E-07
2-Ethyl-1-methylbenzene (a)	611-14-3				
2-Hexanone	591-78-6	2.00E-01	9.44E-02	3.33E+00	6.10E-06
Iodomethane	74-88-4	1.00E+00	4.72E-01	1.67E+01	3.05E-05
Methyl methacrylate (a)	80-62-6				
Methylene Chloride	75-09-2	1.00E+00	4.72E-01	1.67E+01	3.05E-05
Propylbenzene (a)	103-65-1				
Styrene	100-42-5	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1,2,2-Tetrachloroethane	79-34-5	3.00E-02	1.42E-02	5.00E-01	9.15E-07
Tetrachloroethene	127-18-4	6.00E-02	2.83E-02	1.00E+00	1.83E-06
Tetrahydrofuran (a)	109-99-9				
Triethylamine (a)	121-44-8				
Toluene	108-88-3	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1,1-Trichloroethane	71-55-6	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1,2-Trichloroethane	79-00-5	8.00E-02	3.78E-02	1.33E+00	2.44E-06
Trichloroethene	79-01-6	1.00E+00	4.72E-01	1.67E+01	3.05E-05
Trichlorofluoromethane	75-69-4	1.00E-01	4.72E-02	1.67E+00	3.05E-06
1,2,3-Trichloropropane	96-18-4	2.00E-02	9.44E-03	3.33E-01	6.10E-07
1,2,4-trimethylbenzene (a)	95-63-6				
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	2.00E-01	9.44E-02	3.33E+00	6.10E-06
Vinyl Acetate	108-05-4	1.00E-01	4.72E-02	1.67E+00	3.05E-06
Vinyl Chloride	75-69-4	1.00E-03	4.72E-04	1.67E-02	3.05E-08
m & p-Xylenes	108-38-3/106-42-3	2.00E-03	9.44E-04	3.33E-02	6.10E-08
o-Xylene	95-47-6	2.00E-03	9.44E-04	3.33E-02	6.10E-08
Xylenes (total)	1330-02-7	2.00E-03	9.44E-04	3.33E-02	6.10E-08

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
SEMIVOLATILE ORGANICS					
Aniline	62-53-3	1.50E+01	1.42E-01	5.00E+00	9.15E-06
Benzoic Acid (a)	65-85-0	2.00E+01	1.89E-01	6.66E+00	1.22E-05
Benzyl Alcohol	100-51-6	4.00E+01	3.77E-01	1.33E+01	2.44E-05
Bis(2-chloroethoxy) Methane	111-91-1	2.00E+00	1.89E-02	6.66E-01	1.22E-06
Bis-(2-chloroethyl) Ether	111-44-4	2.00E+00	1.89E-02	6.66E-01	1.22E-06
Bis(2-ethylhexyl) Phthalate	117-81-7	5.00E+00	4.72E-02	1.67E+00	3.05E-06
4-Bromophenyl-phenyl Ether	101-55-3	2.00E+00	1.89E-02	6.66E-01	1.22E-06
Butylbenzylphthalate	85-68-7	3.00E+00	2.83E-02	9.99E-01	1.83E-06
4-Chloroaniline	106-47-8	1.00E+01	9.43E-02	3.33E+00	6.10E-06
4-Chloro-3-methylphenol	59-50-7	4.10E+00	3.87E-02	1.37E+00	2.50E-06
2-Chloronaphthalene	91-58-7	1.50E+00	1.42E-02	5.00E-01	9.15E-07
2-Chlorophenol	95-57-8	2.20E+00	2.08E-02	7.33E-01	1.34E-06
4-Chlorophenyl-phenyl Ether	7005-72-3	2.20E+00	2.08E-02	7.33E-01	1.34E-06
Dibenzofuran	132-64-9	2.20E+00	2.08E-02	7.33E-01	1.34E-06
Di-n-butylphthalate	84-74-2	1.30E+00	1.23E-02	4.33E-01	7.93E-07
1,2-Dichlorobenzene	95-50-1	2.00E+00	1.89E-02	6.66E-01	1.22E-06
1,3-Dichlorobenzene	541-73-1	2.30E+00	2.17E-02	7.66E-01	1.40E-06
1,4-Dichlorobenzene	106-46-7	2.40E+00	2.26E-02	7.99E-01	1.46E-06
3,3'-Dichlorobenzidine	91-94-1	1.30E+01	1.23E-01	4.33E+00	7.93E-06
2,4-Dichlorophenol	120-83-2	3.00E+00	2.83E-02	9.99E-01	1.83E-06
Diethyl Phthalate	84-66-2	2.80E+00	2.64E-02	9.33E-01	1.71E-06
2,4-Dimethylphenol	105-67-9	9.80E+00	9.25E-02	3.26E+00	5.98E-06
Dimethylphthalate	131-11-3	1.60E+00	1.51E-02	5.33E-01	9.76E-07
1,3-Dinitrobenzene	99-65-0	1.00E+01	9.43E-02	3.33E+00	6.10E-06
4,6-Dinitro-2-methylphenol	534-52-1	1.40E+01	1.32E-01	4.66E+00	8.54E-06
2,4-Dinitrophenol	51-28-5	2.90E+01	2.74E-01	9.66E+00	1.77E-05
2,4-Dinitrotoluene	121-14-2	3.10E+00	2.92E-02	1.03E+00	1.89E-06
2,6-Dinitrotoluene	606-20-2	2.60E+00	2.45E-02	8.66E-01	1.59E-06
Di-n-octyl Phthalate	117-84-0	5.00E+00	4.72E-02	1.67E+00	3.05E-06
1,4-Dioxane	123-91-1	1.00E+01	9.43E-02	3.33E+00	6.10E-06
Diphenylamine	122-39-7	3.00E+00	2.83E-02	9.99E-01	1.83E-06
Hexachlorobenzene	118-74-1	2.10E+00	1.98E-02	7.00E-01	1.28E-06
Hexachlorobutadiene	87-68-3	2.90E+00	2.74E-02	9.66E-01	1.77E-06
Hexachlorocyclo-pentadiene	77-47-4	2.30E+01	2.17E-01	7.66E+00	1.40E-05
Hexachloroethane	67-72-1	3.80E+00	3.58E-02	1.27E+00	2.32E-06
Isophrone	78-59-1	5.00E+00	4.72E-02	1.67E+00	3.05E-06
2-Methylphenol	95-48-7	6.10E+00	5.75E-02	2.03E+00	3.72E-06
3/4-Methylphenol	106-44-5	5.20E+00	4.91E-02	1.73E+00	3.17E-06
2-Nitroaniline	88-74-4	8.00E+00	7.55E-02	2.66E+00	4.88E-06
3-Nitroaniline	99-09-2	6.00E+00	5.66E-02	2.00E+00	3.66E-06
4-Nitroaniline	100-01-6	3.00E+00	2.83E-02	9.99E-01	1.83E-06
Nitrobenzene	98-95-3	4.70E+00	4.43E-02	1.57E+00	2.87E-06
2-Nitrophenol	88-75-5	8.10E+00	7.64E-02	2.70E+00	4.94E-06
4-Nitrophenol	100-02-7	1.00E+01	9.43E-02	3.33E+00	6.10E-06
N-Nitrosodimethylamine	62-44-2	5.00E+00	4.72E-02	1.67E+00	3.05E-06
N-Nitrosodiphenylamine	86-30-6	5.00E+00	4.72E-02	1.67E+00	3.05E-06
N-Nitroso-di-n-propylamine	621-64-7	2.50E+00	2.36E-02	8.33E-01	1.52E-06
2,2'-oxybis (1-Chloropropane)	108-60-1	1.10E+01	1.04E-01	3.66E+00	6.71E-06
Pentachlorobenzene	608-93-5	1.20E+01	1.13E-01	4.00E+00	7.32E-06
Pentachlorophenol	87-86-5	2.80E+00	2.64E-02	9.33E-01	1.71E-06
Phenol	108-95-2	1.00E+01	9.43E-02	3.33E+00	6.10E-06
1,2,4-Trichlorobenzene	120-82-1	4.40E+00	4.15E-02	1.47E+00	2.68E-06
2,4,5-Trichlorophenol	95-95-4	3.10E+00	2.92E-02	1.03E+00	1.89E-06
2,4,6-Trichlorophenol	88-06-2	2.40E+00	2.26E-02	7.99E-01	1.46E-06

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
POLYAROMATIC HYDROCARBONS					
Acenaphthene	83-32-9	8.00E-03	7.55E-05	2.66E-03	4.88E-09
Acenaphthylene	208-96-8	8.10E-03	7.64E-05	2.70E-03	4.94E-09
Anthracene	120-12-7	9.70E-03	9.15E-05	3.23E-03	5.91E-09
Benzo(a)anthracene	56-55-3	8.80E-03	8.30E-05	2.93E-03	5.37E-09
Benzo(b)fluoranthene	205-99-2	9.90E-03	9.34E-05	3.30E-03	6.04E-09
Benzo(k)fluoranthene	207-08-9	9.80E-03	9.25E-05	3.26E-03	5.98E-09
Benzo(g,h,i)perylene	191-24-2	6.90E-03	6.51E-05	2.30E-03	4.21E-09
Benzo(a)pyrene	50-32-8	7.80E-03	7.36E-05	2.60E-03	4.76E-09
Benzo(e)pyrene	192-97-2	7.80E-03	7.36E-05	2.60E-03	4.76E-09
Chrysene	218-01-9	1.00E-02	9.43E-05	3.33E-03	6.10E-09
Dibenzo(a,h)anthracene	53-70-3	1.20E-02	1.13E-04	4.00E-03	7.32E-09
Fluoranthene	206-44-0	9.20E-03	8.68E-05	3.06E-03	5.61E-09
Fluorene	86-73-7	8.50E-03	8.02E-05	2.83E-03	5.18E-09
Indeno(1,2,3-cd)pyrene	193-39-5	1.20E-02	1.13E-04	4.00E-03	7.32E-09
2-Methylnaphthalene	91-57-6	2.00E-02	1.89E-04	6.66E-03	1.22E-08
Naphthalene	91-20-3	5.00E-01	4.72E-03	1.67E-01	3.05E-07
Perylene	198-55-0	9.00E-03	8.49E-05	3.00E-03	5.49E-09
Phenanthrene	85-01-8	8.50E-03	8.02E-05	2.83E-03	5.18E-09
Pyrene	129-00-0	7.20E-03	6.79E-05	2.40E-03	4.39E-09
ORGANOCHLORINE PESTICIDES					
1,2-Dibromo-3-chloropropane	96-12-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
4,4'-DDD	72-54-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
4,4'-DDE	72-5-9	1.00E+00	9.43E-03	3.33E-01	6.10E-07
4,4'-DDT	50-29-3	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Aldrin	309-00-2	1.00E+00	9.43E-03	3.33E-01	6.10E-07
alpha-BHC	319-84-6	1.00E+00	9.43E-03	3.33E-01	6.10E-07
beta-BHC	319-85-7	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Lindane	58-89-9	1.00E+00	9.43E-03	3.33E-01	6.10E-07
gamma-BHC	319-86-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Chlorobenzilate	510-15-6	1.00E+00	9.43E-03	3.33E-01	6.10E-07
alpha-Chlordane	5103-71-9	1.00E+00	9.43E-03	3.33E-01	6.10E-07
gamma-Chlordane	5103-74-2	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Diallate	2303-16-4	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endosulfan I	959-98-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endosulfan II	33213-65-9	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endosulfan sulfate	1031-07-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endrin	72-20-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endrin Ketone	53494-70-5	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Heptachlor	76-44-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Heptachlor epoxide	1024-57-3	1.00E+00	9.43E-03	3.33E-01	6.10E-07

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
POLYCHLORINATED BIPHENYLS					
3,4,3',4'-Tetrachlorobiphenyl	32598-13-3	5.00E-04	4.72E-06	1.67E-04	3.05E-10
3,4,4',5'-Tetrachlorobiphenyl	70362-50-4	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,3',4'-Pentachlorobiphenyl	32598-14-4	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,5,4'-Pentachlorobiphenyl	74472-37-0	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,4,5,3',4'-Pentachlorobiphenyl	31508-00-6	5.00E-04	4.72E-06	1.67E-04	3.05E-10
3,4,5,2',4'-Pentachlorobiphenyl	65510-44-3	5.00E-04	4.72E-06	1.67E-04	3.05E-10
3,4,5,3',4'-Pentachlorobiphenyl	57465-28-8	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,5,3',4'-Hexachlorobiphenyl	38380-98-4	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,3',4',5'-Hexachlorobiphenyl	68782-90-7	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,4,5,3',4',5'-Hexachlorobiphenyl	52663-72-6	5.00E-04	4.72E-06	1.67E-04	3.05E-10
3,4,5,3',4',5'-Hexachlorobiphenyl	32774-16-6	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,5,3',4',5'-Heptachlorobiphenyl	39635-31-9	5.00E-04	4.72E-06	1.67E-04	3.05E-10
DIOXINS AND FURANS					
2,3,7,8-TCDD	1746-01-6	6.00E-06	5.66E-08	2.00E-06	3.66E-12
2,3,7,8-TCDF	51207-31-9	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,7,8-PeCDD	40321-76-4	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,7,8-PeCDF	57117-41-6	5.00E-06	4.72E-08	1.67E-06	3.05E-12
2,3,4,7,8-PeCDF	57117-31-4	5.00E-06	4.72E-08	1.67E-06	3.05E-12
1,2,3,6,7,8-HxCDD	57653-85-7	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,4,7,8-HxCDD	39227-28-6	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,7,8,9-HxCDD	19408-74-3	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,6,7,8-HxCDF	57117-44-9	4.00E-06	3.77E-08	1.33E-06	2.44E-12
1,2,3,4,7,8-HxCDF	70648-26-9	5.00E-06	4.72E-08	1.67E-06	3.05E-12
1,2,3,7,8,9-HxCDF	72918-21-9	6.00E-06	5.66E-08	2.00E-06	3.66E-12
2,3,4,6,7,8-HxCDF	60851-34-5	5.00E-06	4.72E-08	1.67E-06	3.05E-12
1,2,3,4,6,7,8-HpCDD	35822-39-4	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,4,6,7,8-HpCDF	67562-39-4	7.00E-06	6.60E-08	2.33E-06	4.27E-12
1,2,3,4,7,8,9-HpCDF	55673-89-7	9.00E-06	8.49E-08	3.00E-06	5.49E-12
Total OCDD	3268-87-9	1.00E-05	9.43E-08	3.33E-06	6.10E-12
Total OCDF	39001-02-0	2.00E-05	1.89E-07	6.66E-06	1.22E-11

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
METALS					
Aluminum	7429-90-5	8.40E+00	1.40E-01	4.94E+00	9.05E-06
Antimony	7440-36-0	1.20E+00	2.00E-02	7.06E-01	1.29E-06
Arsenic	7440-38-2	6.00E-01	1.00E-02	3.53E-01	6.46E-07
Barium	7440-39-3	7.00E-01	1.17E-02	4.12E-01	7.54E-07
Beryllium	7440-41-7	3.00E-01	5.00E-03	1.77E-01	3.23E-07
Cadmium	7440-43-9	3.00E-01	5.00E-03	1.77E-01	3.23E-07
Chromium (Total)	7440-47-3	5.00E-01	8.33E-03	2.94E-01	5.39E-07
Chromium (Hexavalent)	7440-47-3	1.50E-01	2.50E-03	8.83E-02	1.62E-07
Cobalt	7440-48-4	1.50E+00	2.50E-02	8.83E-01	1.62E-06
Copper	7440-50-8	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Lead	7439-92-1	3.00E+00	5.00E-02	1.77E+00	3.23E-06
Manganese	7439-96-5	9.00E-01	1.50E-02	5.30E-01	9.70E-07
Mercury	7439-97-6	1.60E+00	2.67E-02	9.42E-01	1.72E-06
Nickel	7440-02-0	1.50E+00	2.50E-02	8.83E-01	1.62E-06
Selenium	7782-49-2	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Silver	7440-22-4	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Thallium	7440-28-0	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Vanadium	7440-62-2	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Zinc	7440-66-6	8.00E-01	1.33E-02	4.71E-01	8.62E-07

- (a) Compound not on typical target analyte list, and does not have proven sampling and/or analytical performance. Detection limit estimated.
- (b) Naphthalene can be analyzed by low resolution GC/MS as a semivolatile compound or by high resolution GC/MS as a PAH compound.
- (c) Compound will be analyzed as a target compound if adequate QC can be demonstrated. If not, the compound will be reported as a TIC, if present.
- (d) Compound not included in the list of SW-846 Method 8270 compounds, and will be reported as a TIC, if present.
- (e) Marginal recovery/reproducibility in field validation studies with Method 0010/3542/8270.

Basis for stack gas flow rate (dscfm)	3878
Assumed VOC sample size (L, dry, std.)	60
Assumed SVOC sample size (dscf)	106
Assumed PAH sample size (dscf)	106
Assumed PCDD/PCDF sample size (dscf)	106
Assumed metals sample size (dscf)	60

Note: All estimates for PCDD/PCDFs and PAHs are estimated EDLs. Other compounds are estimated MDLs.
 SVOC values are based on a 2-way split of a dedicated sampling train (analyzed fraction and archive fraction).

Note: All estimates are for the entire sampling train. For example, volatile organics include three sets of VOST tubes plus condensate.

dscf = dry standard cubic feet
 dscm = dry standard cubic meters
 Standard conditions 68°F, 29.92 in. Hg

Table 6-1 Sample Collection Locations, Equipment, and Methods

Location ^a	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method ^b
1	Spent Activated Carbon (1-Volatiles) (1-Semivolatiles) (1 – Metals) (1 - Properties) (1-Archive)	Conveyor	Teflon scoop 4L glass jug, 250 ml jar (VOA) 1L glass bottles with teflon lined lids	1 scoop per grab; 250 ml volatiles 1L semivolatiles 1L properties 1L metals 1L archive	Collect a grab sample at each 15-minute interval during each test run. Grab samples will be combined in a glass jug to build run composite. Collect four 1-liter samples and one 250 ml VOA jar of the homogenized composite at the end of the test run.	SW-846, Vol. II, Chapter 9, Section 9.3
2	Makeup water (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA 1L semivolatiles 1L metals 1L archive	Collect one pair of 40 ml VOA vials at the beginning of the test; Fill 4L bottle at beginning of test. Fill three 1-liter samples from the 4L bottle.	SW-846, Vol. II, Chapter 9, Section 9.2
3	Caustic (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA 1L semivolatiles 1L metals 1L archive	Collect one pair of 40 ml VOA vials at the beginning of the test; Fill 4L bottle at beginning of test. Fill three 1-liter samples from the 4L bottle.	SW-846, Vol. II, Chapter 9, Section 9.2
4	Scrubber Blowdown (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA ~200 ml per grab; 1L semivolatiles 1L metals 1L archive	Collect one pair of 40 ml VOA vials at each 30 minute interval; Collect a ~200 ml grab sample at each 30-minute interval during each test run. Grab samples will be combined in a glass jug to build run composite. Collect three 1-liter samples of the homogenized composite at the end of the test run.	SW-846, Vol. II, Chapter 9, Section 9.2
5	POTW Discharge (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA ~200 ml per grab; 1L semivolatiles 1L metals 1L archive	Collect one pair of 40 ml VOA vials at each 30 minute interval; Collect a ~200 ml grab sample at each 30-minute interval during each test run. Grab samples will be combined in a glass jug to build run composite. Collect three 1-liter samples of the homogenized composite at the end of the test run.	SW-846, Vol. II, Chapter 9, Section 9.2

Table 6-1 Sample Collection Locations, Equipment, and Methods

Location ^a	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method ^b
Stack (6)	Stack gas M29	Port	EPA Method 29 multiple metals sampling train	Minimum 120 minutes ^{c,d}	Collect integrated sample for metals and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5, and 29.
Stack (6)	Stack gas M0061	Port	SW-846 Method 0061 hexavalent chromium sampling train	Minimum 120 minutes ^{c,d}	Collect integrated samples for hexavalent chromium and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0061
Stack (6)	Stack gas M26A	Port	EPA Method 26A sampling train	Minimum 120 minutes ^{c,d}	Collect integrated sample for particulate, hydrogen chloride, and chlorine. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5, and 26A
Stack (6)	Stack gas M0010-SV	Port	SW-846 Method 0010 sampling train	Minimum 3 dry standard cubic meters ^{c,d}	Collect integrated sample for semivolatile organics, organochlorine pesticides, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010.
Stack (6)	Stack gas M0010-P	Port	Combined SW-846 Method 0010, EPA CARB Method 429 sampling train	Minimum 3 dry standard cubic meters ^{c,d}	Collect integrated sample for PAHs, PCBs, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010; CARB Method 429.

Table 6-1 Sample Collection Locations, Equipment, and Methods

Location ^a	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method ^b
Stack (6)	Stack gas M0010-TOE	Port	SW-846 Method 0010 sampling train	Minimum 3 dry standard cubic meters ^{c,d}	Collect integrated samples for total semivolatile organics, total nonvolatile organics, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010; EPA TOE Guidance
Stack (6)	Stack gas M0023A	Port	SW-846 Method 0023A sampling train	Minimum 3 hours and 2.5 dry standard cubic meters ^{c,d}	Collect integrated sample for PCDD/PCDFs, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0023A.
Stack (6)	Stack gas M0030	Port	SW-846 Method 0030 volatile organic sampling train	4 tube pairs per run; 40 minutes per tube pair. Up to 20 liters of stack gas per tube pair	Collect four pairs of sorbent tubes and stack gas condensate for volatile organics during each run.	SW846-0030 (VOST)
Stack (6)	Stack gas M0040	Port	SW-846 Method 0040 sampling train	25 – 50 liters	Collect representative sample through a heated sample probe and filter; through a condenser and into a Tedlar bag. Transport dried sample and condensate to GC/FID.	EPA Methods 1 through 5; SW846-0040; EPA TOE Guidance.
Stack (6)	Stack gas PSD	Port	Cascade impactor	As required	Collect particle size distribution samples on multiple substrates	Cascade impactor mfr. instructions
Stack (6)	Stack gas CEMS	Port	Temporary CEMS THC	Continuous	Continuously monitor stack gas for total hydrocarbons during each run	EPA Method 25A
Stack (7)	Stack gas CEMS	Port	Installed CEMS CO	Continuous	Continuously monitor stack gas carbon monoxide during each run.	40 CFR 63 Subpart EEE Appendix; PS 4B

Table 6-1 Sample Collection Locations, Equipment, and Methods

Location ^a	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method ^b
Stack (7)	Stack gas CEMS	Port	Installed CEMS O ₂	Continuous	Continuously monitor stack gas oxygen during each run.	40 CFR 63 Subpart EEE Appendix; PS 4B

- a Refer to Figure 5-1 of the Comprehensive Performance Test Plan.
- b “SW846” refers to Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates.
 “EPA Method” refers to New Source Performance Standards, Test Methods and Procedures, Appendix A, 40 CFR 60.
 “CARB” refers to California Air Resources Board Methods.
 “PS 4B” refers to Performance Specification 4B, 40 CFR 60.
- c The exact volume of gas sampled will depend on the isokinetic sampling rate.
- d Isokinetic sampling trains include:
- Collecting one set of bag samples (or using CEM) for oxygen and carbon dioxide analysis to determine stack gas molecular weight (EPA Method 3)
 - Performing stack gas velocity, pressure, and temperature profile measurement for each sampling location (EPA Method 2)
 - Determining the moisture content of the stack gas for each sampling train (EPA Method 4)

Table 6-2. Summary of Expected Trial Burn Field Samples

Sample Matrix	Container	Routine Samples or Field Splits (a) (No. per Run)	Number of Runs	Total Samples Collected During Test
Spent Activated Carbon				
Physical/chemical properties	1L glass	1	3	3
Volatile organics	250 mL VOA jar	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		5		15
Makeup Water				
Volatile organics	40 ml VOA vials	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		4		12
Caustic				
Volatile organics	40 ml VOA vials	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		4		12
Scrubber Blowdown				
Volatile organics	40 ml VOA vials	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		4		12
POTW Discharge				
Volatile organics	40 ml VOA vials	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		4		12
Stack Gas M0023A				
Filter	Glass petri dish	1	3	3
XAD-2 Resin trap	Glass trap	1	3	3
Front half acetone/methylene chloride and toluene rinses	500 ml amber glass	1	3	3
Back half acetone/methylene chloride and toluene rinses	500 ml amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	1
XAD-2 Resin trap (blank train)	Glass trap	NA	NA	1
Front half acetone/methylene chloride and toluene rinses (blank train)	500 ml amber glass	NA	NA	1
Back half acetone/methylene chloride and toluene rinses (blank train)	500 ml amber glass	NA	NA	1
XAD-2 spiked resin trap blanks	Glass trap	NA	NA	2
Acetone reagent blank	500 ml amber glass	NA	NA	1
Methylene chloride reagent blank	500 ml amber glass	NA	NA	1
Toluene reagent blank	500 ml amber glass	NA	NA	1
Audit sample (XAD-2 resin trap)	Glass trap	NA	NA	1
Subtotal		4		22
Stack Gas M0010-SV (Modified for SVOCs and OCP)				
Filter	Glass petri dish	1	3	3
XAD-2 Resin trap	Glass trap	1	3	3
Front half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Back half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Condensate	1 liter amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	1
XAD-2 Resin trap (blank train)	Glass trap	NA	NA	1
Front half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
Back half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
XAD-2 Resin trap blanks	Glass trap	NA	NA	2
Acetone reagent blank	500 ml amber glass	NA	NA	1
Methylene chloride reagent blank	500 ml amber glass	NA	NA	1
Subtotal		5		23
Stack Gas M0010-P (Modified for PAH and PCB)				
Filter	Glass petri dish	1	3	3
XAD-2 Resin trap	Glass trap	1	3	3
Front half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Back half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Condensate	1 liter amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	1
XAD-2 Resin trap (blank train)	Glass trap	NA	NA	1
Front half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
Back half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
Subtotal		5		23

Table 6-2. Summary of Expected Trial Burn Field Samples

Sample Matrix	Container	Routine Samples or Field Splits (a) (No. per Run)	Number of Runs	Total Samples Collected During Test
XAD-2 Resin trap blanks	Glass trap	NA	NA	2
Acetone reagent blank	500 ml amber glass	NA	NA	1
Methylene chloride reagent blank	500 ml amber glass	NA	NA	1
Subtotal		5		23

Table 6-2. Summary of Expected Trial Burn Field Samples

Sample Matrix	Container	Routine Samples or Field Splits (a) (No. per Run)	Number of Runs	Total Samples Collected During Test
Stack Gas VOST M0030				
Tenax resin tube	Glass culture tube	4	3	12
Tenax resin/charcoal tube	Glass culture tube	4	3	12
Condensate	40 ml VOA	1	3	3
Tenax resin tube field blank	Glass culture tube	1	3	3
Tenax resin/charcoal tube field blank	Glass culture tube	1	3	3
Tenax resin tube spiked resin blank	Glass culture tube	NA	NA	1
Tenax resin/charcoal tube spiked resin blank	Glass culture tube	NA	NA	1
Tenax resin tube trip blank (1 per shipment to lab)	Glass culture tube	NA	NA	1
Tenax resin/charcoal tube trip blank (1 per shipment to lab)	Glass culture tube	NA	NA	1
Tenax resin tube audit samples (4 tubes x 1 cylinder)	Glass culture tube	4	NA	4
Tenax resin/charcoal tube audit samples (4 tubes x 1 cyl)	Glass culture tube	4	NA	4
Subtotal		19		45
Stack Gas Metals Method 29				
Filter	Glass petri dish	1	3	3
Nitric acid probe rinse	500 ml amber glass	1	3	3
Acidified peroxide impinger solution and rinses	1 liter amber glass	1	3	3
Initially empty impinger solution and rinse	500 ml amber glass	1	3	3
Permanganate impinger solution and rinse	500 ml amber glass	1	3	3
HCl rinse of permanganate impingers (if used)	500 ml amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	2
Nitric acid probe rinse (blank train)	500 ml amber glass	NA	NA	2
Acidified peroxide impinger solution and rinses (blank train)	500 ml amber glass	NA	NA	2
Initially empty impinger solution and rinse (blank train)	500 ml amber glass	NA	NA	2
Permanganate impinger solution and rinse (blank train)	500 ml amber glass	NA	NA	2
HCl rinse of permanganate impingers (if used) (blank train)	500 ml amber glass	NA	NA	2
Nitric acid solution reagent blank	500 ml amber glass	NA	NA	1
Nitric acid/hydrogen peroxide solution reagent blank	500 ml amber glass	NA	NA	1
Acidified potassium permanganate solution reagent blank	500 ml amber glass	NA	NA	1
HCl solution reagent blank (if used)	500 ml amber glass	NA	NA	1
Filter blank	Glass petri dish	NA	NA	1
Subtotal		6		35
Stack Gas Hexavalent Chromium M0061				
Impinger solution and rinses	1 liter polyethylene	1	3	3
Filter and Residue	Glass vial	1	3	3
HNO3 Rinses	500 ml glass	1	3	3
KOH reagent blank	500 ml polyethylene	NA	NA	1
HNO3 Reagent Blank	500 ml polyethylene	NA	NA	1
Water reagent blank	500 ml polyethylene	NA	NA	1
Field spiked KOH solution (audit sample)	500 ml polyethylene	NA	NA	1
Subtotal		3		13
Stack gas Method 26A				
Filter	Petri dish	1	6	6
Front half acetone rinse	500 ml amber glass	1	6	6
Acid impinger liquid	500 ml amber glass	1	6	6
Alkaline impinger liquid	500 ml amber glass	1	6	6
Acetone reagent blank	500 ml amber glass	NA	NA	1
Sulfuric acid solution reagent blank	500 ml amber glass	NA	NA	1
Sodium hydroxide solution reagent blank	500 ml amber glass	NA	NA	1
Deionized water reagent blank	500 ml amber glass	NA	NA	1
Subtotal		4		28
Stack Gas M0010-TOE (TCO/GRAV)				
Filter	Glass petri dish	1	3	3
XAD-2 Resin trap	Glass trap	1	3	3
Front half methanol/methylene chloride rinses	500 ml amber glass	1	3	3
Back half methanol/methylene chloride rinses	500 ml amber glass	1	3	3
Condensate	1 liter amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	1
XAD-2 Resin trap (blank train)	Glass trap	NA	NA	1
Front half methanol/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
Back half methanol/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
XAD-2 Resin trap blanks	Glass trap	NA	NA	2
Filter blank	Glass petri dish	NA	NA	1
methanol/methylene chloride reagent blank	500 ml amber glass	NA	NA	1
Subtotal		5		23

Table 6-2. Summary of Expected Trial Burn Field Samples

Sample Matrix	Container	Routine Samples or Field Splits (a) (No. per Run)	Number of Runs	Total Samples Collected During Test
Stack Gas M0040				
Sample bag	Tedlar bag	1	3	3
Condensate	40 ml VOA	1	3	3
Field blank bag	Tedlar bag	1	3	3
Condensate blank	Tedlar bag	1	3	3
Subtotal		4		12
PSD (Cascade Impactor)				
Filter	Perti dish stack	1	3	3
Subtotal		1		3
TOTAL		69		266

(a) "Field Splits" are separate portions of the same sample, placed into individual containers.
 "Field Duplicates" are separate samples collected from the same sampling point.

Table 7-1. Sample Containers, Preservation, and Holding Times

Parameter	Sample Name	Containers	Preservation	Maximum Holding Time
Volatile organics	Makeup water, caustic, scrubber blowdown, POTW discharge	Glass VOA, Teflon-lined septum	Chill 4°C	14 days
	Spent Activated Carbon	Glass VOA, Teflon-lined septum	Chill 4°C	14 days
	Stack gas VOST tubes	Glass tube	Chill 4°C	14 days
	Stack gas VOST condensate	Glass VOA, Teflon-lined septum	Chill 4°C	14 days
	Stack gas M0040 bags	Tedlar bag	Protect from sunlight	72 hours
	Stack gas M0040 condensate	Glass VOA, Teflon-lined septum	Chill 4°C	14 days
SVOC, PAH, OCP, and PCB	Makeup water, caustic, scrubber blowdown (SVOC only), POTW discharge (SVOC only)	Glass bottle, Teflon-lined cap	Chill 4°C	14 days until extraction, 40 days after extraction
	Spent Activated Carbon	Glass bottle, Teflon-lined cap	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0010 filter	Glass petri dish	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0010 sorbent tube	Standard cartridge wrapped in aluminum foil	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0010 liquids	Glass bottle, Teflon-lined cap	Chill 4°C	14 days until extraction, 40 days after extraction
PCDD/PCDF	Stack gas M0023A filter	Glass petri dish	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0023A sorbent tube	Standard cartridge wrapped in aluminum foil	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0023A liquids	Glass bottle, Teflon-lined cap	Chill 4°C	14 days until extraction, 40 days after extraction
Metals (except Cr VI)	Makeup water, caustic, scrubber blowdown, POTW discharge	Glass bottle	NA	180 days/28 days for Hg
	Spent Activated Carbon	Glass bottle	NA	180 days/28 days for Hg
	Stack gas M29 filter	Petri dish	None required	180 days/28 days for Hg
	Stack gas M29 liquids	Glass bottle	None required	180 days/28 days for Hg

Table 7-1. Sample Containers, Preservation, and Holding Times

Parameter	Sample Name	Containers	Preservation	Maximum Holding Time
Hexavalent chromium	Stack gas M0061 liquids	Polyethylene bottle	Chill 4°C pH >8.5	14 days
Total Semivolatile and Non-volatile organics	Stack gas M0010 XAD-2 sorbant tube	Standard cartridge wrapped in aluminum foil	Chill 4°C	14 days until extraction, 40 days after extraction
Total Semivolatile and Non-volatile organics Cont	Stack gas M0010	Glass petri dish	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0010 liquids	Glass bottle, Teflon-lined cap	Chill 4°C	14 days until extraction, 40 days after extraction
Stack gas HCl/Cl ₂	Stack gas M26A liquids	Glass bottle, Teflon lined cap	None required	30 days
Stack gas particulate	Stack gas M26A and cascade impactor	Petri dish	None required	NA
	Stack gas M26A front half rinses	Glass bottle, Teflon lined cap	None required	NA

Table 8-1. Sampling Equipment Calibration Requirements

Stack Gas Parameter	Quality Parameter	Method of Determination	Frequency	Criteria
Gas Flow	Pitot tube angle & dimensions	Measurements with a vernier micrometer and angle indicator or wind tunnel	Post-test	To specifications in EPA Method 2
	Barometer	Calibrated vs. lab Hg-in-glass barometer	Pre-test	Within 0.2 in. Hg
	Stack gas thermocouple	Calibrated vs. ASTM Hg-in-glass thermometer	Pre-test & Post-test	Within 1.5% as R
Isokinetic Sampling Trains	Dry gas meter	Calibrated against a reference test meter	Pre-test & Post-test	Y within 0.05 of pre-test Y; H@ within 0.15 of pre-test
	Probe nozzle	Measurements with vernier micrometer to 0.001 in.	On-site Pre-test	Maximum difference in any two dimensions within 0.004 in.
	Dry gas meter thermocouples	Calibrated vs. ASTM Hg-in-glass thermometer	Post-test	Within 5 degrees F
	Triple beam balance	Calibrated vs. standard weights	Post-test	Within 0.5g

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Volatile organics by GC/MS (SW846 8260)	Initial calibration	3 - 5 standards bracketing expected concentrations	Prior to sample analysis	Variability of average RRF less than or equal to 30% RSD for POHCs and CCCs SPCCs (chlorobenzene and 1,1,2,2-tetrachloroethane) will be ≥ 0.3 , and SPCCs (chloromethane, 1,1-dichloroethane, and bromoform) will be ≥ 0.1	Recalibrate
	Continuing calibration	Midlevel standard	Prior to sample analysis, then every 12 hours or after sample set	RRF for POHCs and CCCs within 25% difference of the initial calibration average RRF. SPCCs (chlorobenzene and 1,1,2,2-tetrachloroethane) will be ≥ 0.3 , and SPCCs (chloromethane, 1,1-dichloroethane, and bromoform) will be ≥ 0.1	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check.
	Consistency in chromatography	For MS methods, monitor internal standard retention time and area. For non-MS methods, monitor retention time windows for compounds of interest.	Every sample, standard, and blank	Retention time within 30 seconds of last calibration check. Area within -50 to +100% of last calibration check	Perform calibration standard check. Reanalyze sample if possible, or flag data.
	Calibration check or LCS	Analysis of independent calibration check standard	In association with each initial calibration	Within 3 std. deviations of historical mean (laboratory specific)	Recalibrate and recheck.
	Method Blank	Analysis of blank	Analyze one with each analytical batch	Result less than method detection limit	Flag data and discuss in case narrative.

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Semivolatile organics GC/MS (SW846 8270)	Initial calibration	5 standards bracketing expected concentrations. Critical level should be at least 10 times higher than lowest standard	Prior to sample analysis	Variability of average RRF less than or equal to 30% RSD for CCCs. SPCCs greater than or equal to 0.05.	Recalibrate
	Continuing calibration	Midlevel standard	Prior to sample analysis, then every 12 hours or after sample set	RRF for CCCs within 30% of initial calibration average RRF. SPCCs greater than or equal to 0.05.	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check.
	Consistency in chromatography	For MS methods, monitor internal standard retention time and area. For non-MS methods, monitor retention time window for compounds of interest.	Every sample, standard, and blank	Retention time within 30 seconds of last calibration check. Area within -50 to +100% of last calibration check	Perform calibration standard check. Reanalyze sample if possible, or flag data.
	Calibration check	Analysis of independent calibration check standard	In association with each initial calibration	Within 3 std. deviations of historical mean (laboratory specific)	Recalibrate and recheck.
	Method Blank	Analysis of blank	Analyze one with each analytical batch	Results less than method detection limit	Flag data and discuss in the case narrative.
Organochlorine Pesticides (SW846 – 8081)	Initial calibration	5 standards bracketing expected concentrations. Critical level should be at least 10 times higher than lowest standard	Prior to sample analysis	Mean CF %RSD \leq 20%	Recalibrate

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
	Continuing calibration	Midlevel standard	Prior to sample analysis, then every 12 hours or after sample set	CF for each analyte $\leq 15\%D$ from the mean CF for the Initial calibration .	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check.
Organochlorine Pesticides (SW846 – 8081) Cont.	Consistency in chromatography	For MS methods, monitor internal standard retention time and area. For non-MS methods, monitor retention time window for compounds of interest.	Every sample, standard, and blank	Retention time within 30 seconds of last calibration check. Area within -50 to +100% of last calibration check	Perform calibration standard check. Reanalyze sample if possible, or flag data.
	Calibration check	Analysis of independent calibration check standard	In association with each initial calibration	Within 3 std. deviations of historical mean (laboratory specific)	Recalibrate and recheck.
	Method Blank	Analysis of blank	Analyze one with each analytical batch	Results less than method detection limit	Flag data and discuss in the case narrative.
PCDD/PCDF by High Res. GC/MS (SW846 8290)	Initial Calibration	All five high resolution concentration calibration solutions must be used for the initial calibration	Prior to sample analysis	The %RSD for the mean RRF from the 17 unlabeled standards must not exceed $\pm 20\%$, and those for the 9 labeled reference compounds must not exceed $\pm 30\%$.	Recalibrate
	Continuing Calibration	Midlevel standard	At the beginning and end of each 12 hour shift	RFs must be within $\pm 20\%$ of the initial calibration mean RRF for unlabeled standards and $\pm 30\%$ for labeled standards	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check.

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
	Retention time window verification and GC column performance	Monitor retention times	Start of each 12 hour shift	Compliance with Section 8.2.1 of Method 8290	Correct according to method
	Method Blank	Analysis of blanks	Analyze one with each analytical batch	Results less than method detection limit	Flag data as discussed in case narrative

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
PCBs by High Resolution GC/MS (EPA 1668)	Initial Calibration	All five high resolution concentration calibration solutions must be used for the initial calibration	Prior to sample analysis	< 35% coefficient of variation over the five-point range for the average RF to be used.	Recalibrate or use the complete calibration curve over the five-point range.
	Continuing Calibration	Midlevel standard	At the beginning and end of each 12 hour shift	RFs must be within $\pm 30\%$ of the initial calibration average RF	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check.
	Retention times and GC resolution	Monitor retention times	Start of each 12 hour shift	Compliance with Section 15.4 of Method 1668	Correct according to method
	Method blank	Analysis of blanks	Analyze one with each analytical batch	Results less than method detection limit	Flag data as discussed in case narrative
Total Volatiles (Method 0040)	Initial calibration	Minimum of 3 standards bracketing expected concentrations analyzed in duplicate	Prior to sample analysis	Laboratory specific	N/A
	Daily Calibration check	Midlevel standard	At least one per day	$\pm 10\%$ of true value	Reanalyze standard and if necessary recalibrate system
	Blanks	Tedlar bag blanks and water blanks	One with each sample	Less than 20% of field sample	Evaluate for possible blank correction
TCO (Method 0010)	Initial calibration	Minimum of 3 standards	Prior to sample analysis	Correlation coefficient ≤ 0.97	Recalibrate
	Daily Calibration check	Midlevel standard	At least one per day	$\pm 15\%$ of true value	Reanalyze standard and if necessary recalibrate system
	Method Blank	Analysis of blanks	One with each batch	< 20% of sample results	Evaluate for possible contamination

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Metals by ICP or AAS (SW846 6010 ICP and SW846 7470 CVAA)	Initial calibration	Multiple standards (AAS) or 1 standard (ICP) and a calibration blank, bracketing the expected concentrations. Critical level should be at least twice the lowest calibration standard.	Prior to sample analysis	Correlation coefficient of linear plot >0.995 (AAS). Not applicable for ICP.	Recalibrate
	Reagent blank	Analysis of blank	After every 10 samples and at end of analysis	Less than instrument detection limit (IDL)	Reanalyze if greater than the reporting limit and discuss in case narrative if greater than the IDL
	Calibration check	Analysis of independent calibration check standard	Once after initial calibration	90 - 110% of theoretical value	Reanalyze and recalibrate, if necessary
	Serial dilution	Analysis of serial dilution (DF=5)	Once per matrix for high level analytes (ICP only)	90 - 110% of undiluted sample value (ICP samples > 50 times the IDL)	Flag data; discuss in case narrative
	Post digestion spike	Analysis of post digest spike, spiked at 2 to 5 times the original sample value	Each sample analyzed by GFAAS One sample per test for ICP	85 - 115% of theoretical value	Flag data; discuss in case narrative
	Calibration accuracy (ICP only)	Reanalysis of high level standard	After every initial calibration	90 - 110% of theoretical value	Recalibrate and recheck
	Interference check (ICP only)	Analysis of interference sample	After every initial calibration and at the end of each run	80 - 120% of theoretical value	Recalibrate and recheck

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
	Continuing calibration	Midlevel standard and blank	Beginning and end of each analysis period and after every 10 samples	AAS - Midlevel standard 80 - 120% of theoretical value; blank <50% of lowest calibration standard. ICP - Midlevel standard 90 - 110% of theoretical value; blank <50% of lowest calibration standard or within 3 SD of average blank.	Identify and correct problems; reanalyze samples run since last acceptable continuing calibration check.
Hexavalent chromium by ion chromatography (SW846 7199)	Initial calibration	≥4 standards bracketing expected concentrations	Prior to and following sample analysis	RPD of response of the two standards at each concentration <10% RPD. Also the average of the standards must yield a relative accuracy of 93 - 107% when calculated versus the linear regression curve.	Reanalyze calibration standards and samples.
	Calibration check or LCS	Analyze independent check standard.	Once per test	90 - 110% of true value	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze.
	Sample response	Analyze each sample in duplicate	Every sample	<10% RPD of instrument response	Reanalyze samples
Particulate and Gravimetric	Calibration check	Calibration of balance with standardized weights	Prior to analysis, between each group of sample weighings, and at the end of each day.	99 – 101% of theoretical value	Recalibrate and recheck sample weights.

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Chloride by Ion Chromatography	Initial Calibration	4 standards bracketing expected concentrations Note: Separate calibrations are required for the acid and alkaline samples	Prior to sample analysis	Linear correlation coefficient >0.995	Recalibrate
	Retention time check for ion identification	Determine average retention time for ions of interest or relative retention time of several ions for every calibration curve	Prior to sample analysis	Average Retention Time - Sample identification is positive if results are within retention time window of standards Relative Retention Time – Sample identification is positive if results are within 3 SD of average RRT	Ions of interest are not present if criteria are not met.
Chloride by Ion Chromatography Cont.	Control check sample	Midlevel independent standard analyzed in duplicate	Beginning and end of each analysis period and after every 10 samples	90 – 110% of theoretical value	Repeat calibration check. If second check fails criteria, regenerate analytical system and reanalyze all samples since last acceptable calibration check.
	Reagent blank (ICB and CCBs)	Analysis of blanks	Immediately following the ICV and following each CCV.	Less than 1 mg/L	Contamination source must be found and corrected. All samples analyzed since the last acceptable CCB must be reanalyzed.
	Reagent blank (ICB and CCBs)	Analysis of blanks	Immediately following the ICV and following each CCV.	Less than 1 mg/L	Contamination source must be found and corrected. All samples analyzed since the last acceptable CCB must be reanalyzed.

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
PAH by High Resolution GC/MS (CARB 429)	Initial Calibration	5 standards bracketing expected concentrations.	Prior to sample analysis	Mean RRF must be no greater than 30% for both unlabeled analytes and internal standards	Recalibrate
	Continuing Calibration	Midlevel standard	At the beginning and end of each analysis period, or at the beginning of each 12-hour shift if the laboratory operates during consecutive 12-hour shifts	RRFs must be within $\pm 30\%$ of the initial calibration mean RRF for all analytes (both labeled and unlabeled)	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since last successful check.

Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
PAH by High Resolution GC/MS (CARB 429) Cont.	Retention time window verification and GC column performance	Monitor retention times	Each daily calibration	Retention times for any internal standard must not change by more than 30 seconds from the most recent calibration	Inspect the chromatographic system for malfunctions and make the necessary corrections. Document acceptable performance with a new initial calibration curve.
	Calibration check of LCS	Analysis of independent calibration check standard	Two per batch of fifteen or less samples	Percent recovery for both standards should be between 50 – 150% and the relative percent difference should be less than 50%	Repeat the test for those analytes which failed to meet the performance criteria
	Method blank	Analysis of blanks	Analyze one with each analytical batch	Results less than method detection limit or less than 5% of the analyte concentration in the field sample.	Flag data and if blank results are greater than 5% of the field sample concentrations, reanalyze samples.

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Samples per Run	Total Field Samples for Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Spent Activated Carbon	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Chloride	1	3	SW846-5050	Ion chromatography (SW846-9056)
	Total metals	1	3	Acid digestion (SW846-3050)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
	Elemental	1	3	NA	(ASTM D5373) with (ASTM D3176) as an alternate
Makeup Water	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Total metals	1	3	Acid digestion (SW846-3020)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
Caustic	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Total metals	1	3	Acid digestion (SW846-3020)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
Scrubber Blowdown	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Total metals	1	3	Acid digestion (SW846-3020)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
POTW Discharge	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Total metals	1	3	Acid digestion (SW846-3020)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Samples per Run	Total Field Samples for Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Stack gas M0030	VOCs + TICs (tenax + tenax/charcoal tubes) (Note 2)	(Note 3)	(Note 3)	Thermal desorption, trap (SW846-5041A)	GC/MS (SW846-8260)
	VOCs + TICs (condensate) (Note 2)	1	3	Purge and trap	GC/MS (SW846-8260)
Stack gas M0040	Total VOCs	1	3	Purge and trap for condensate Direct injection for gas	GC/FID (Guidance for Total Organics, App. A and E)
Stack gas M0010-SV (low res analysis)	Semivolatile Organics & TICs (Note 4)	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	OCP (Note 5)	1	3	Solvent extraction (SW846-3542)	GC (SW-846-8081)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M0010-P (high res analysis)	PCB (Note 7)	1	3	Solvent extraction (SW846-3542)	GC/MS (EPA Method 1668)
	PAH (Note 8)	1	3	Solvent extraction (CARB 429)	GC/MS (CARB 429)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Samples per Run	Total Field Samples for Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Stack gas M0010-TOE	Total SVOCs	1	3	Solvent extraction (SW846-3542)	TOC GC/FID (Guidance for Total Organics, Appendix C)
	Total NVOCs	1	3	Solvent extraction (SW846-3542)	Gravimetric Method (Guidance for Total Organics, Appendix D)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M0023A	PCDD/PDCF	1	3	Solvent extraction (SW846-3500)	GC/MS (SW-846 Method 8290)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M29	Metals (Note 9)	1	3	Acid digestion (SW846-3050)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M0061	Hexavalent chromium	1	3	NA	Ion chromatography, post-column reactor (SW846-7199)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Samples per Run	Total Field Samples for Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Stack gas M26A	Hydrogen chloride/Chlorine	1	3	NA	Ion chromatography (SW846-9057)
	Particulate	1	1	NA	Gravimetric (EPA Method 5)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M00023A	PCDD/PCDF	1	3	Solvent extraction (SW846-8290)	GC/MS (SW846-8290; & SW846-0023A)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Flow rate	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas PSD	Particle size distribution	NA	NA	NA	Cascade impactor manufacturer's instructions
Stack gas temporary CEMS	Total hydrocarbons	(Note 10)	(Note 10)	NA	Extractive Analyzers, EPA Method 25A
Stack gas Installed CEMs	Carbon Monoxide	(Note 10)	(Note 10)	NA	Extractive Analyzers, 40CFR 63 Appendix
	Oxygen	(Note 10)	(Note 10)	NA	Extractive Gas Analyzers, 40 CFR 63 Appendix

Note 1: "ASTM" refers to American Society for Testing and Materials, Annual Book of ASTM Standards, Annual Series.
 "SW846" refers to Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and updates.
 "EPA Methods" (Methods 1 through 5 and 23) refer to New Source Performance Standards, Test Methods and Procedures,, App. A, 40CFR 60.
 "CARB" refers to California Air Resources Board methodology adopted January 27, 1987.
 "Guidance for Total Organics" refers to EPA/600/R-96/036, March, 1996.

Note 2: Volatile Target Compounds as listed in this Test Plan, plus tentatively identified compounds.

Note 3: During each sampling run, 4 pairs of VOST tubes (8 samples) will be collected, but only 3 pairs (6 samples) will be analyzed. The extra tube pair provides a contingency in case of breakage or other event that could require analysis of the extra tube pair. Analysis of each tube in each tube pair will be conducted separately.

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Note 4: Semivolatile Target Compounds as listed in this Test Plan, plus tentatively identified compounds.

Note 5: Organochlorinated pesticide (OCP) target compounds as listed in this Test Plan.

Note 6: One set of gas bag samples collected during each stack traverse for Orsat analysis, or CEM.

Note 7: Polychlorinated Biphenyl (PCB) target compounds target compounds as listed in the Plan

Note:8 Polycyclic Aromatic Hydrocarbon (PAH) target compounds as listed in this Plan

Note 9: Metal Target Compounds as listed in this Test Plan.

Note 10: Installed CEMs sampling and analysis is continuous during each run.

Table 9-2. Summary of Target Volatile Organic Analytes

Volatiles	CAS Number
Acetone	67-64-1
Acrylonitrile	107-13-1
Benzene	71-43-2
Bromodichloromethane	75-27-4
Bromochloromethane	74-97-5
Bromoform	75-25-2
Bromomethane	74-83-9
2-Butanone	78-93-3
Carbon Disulfide	75-15-0
Carbon Tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chlorodibromomethane	124-48-1
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
1,2-Dibromoethane ^a	106-93-4
Dibromomethane	74-95-3
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
1,2-Dichloropropane	78-87-5
Dicyclopentadiene ^a	77-73-6
Ethylbenzene	100-41-4
2-Ethyl-1-methylbenzene ^a	611-14-3
2-Hexanone	591-78-6
Iodomethane	74-88-4
Methyl methacrylate ^a	80-62-6
Methylene Chloride	75-09-2
Propylbenzene ^a	103-65-1
Styrene	100-42-5
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Tetrahydrofuran ^a	109-99-9
Triethylamine ^a	121-44-8

Table 9-2. Summary of Target Volatile Organic Analytes

Volatiles	CAS Number
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene ^a	95-63-6
1,1,2-Trichloro – 1,2,2-trifluoroethane (freon 113)	76-13-1
Vinyl Acetate	108-05-4
Vinyl Chloride	75-69-4
m & p Xylenes	108-38-3 & 106-42-3
o-xylene	95-47-6
Xylenes (total)	1330-02-7

a Reported as a TIC if found

Table 9-3. Summary of Target Semivolatile Organic Analytes

Semivolatiles	CAS Number
Aniline	62-53-3
Benzoic Acid	65-85-0
Benzyl alcohol	100-51-6
Bis(2-chloroethoxy) methane	111-91-1
Bis-(2-chloroethyl) ether	111-44-4
Bis(2-ethylhexyl) phthalate	117-81-7
4-Bromophenyl-phenyl ether	101-55-3
Butylbenzylphthalate	85-68-7
4-Chloroaniline	106-47-8
4-Chloro-3-methylphenol	59-50-7
2-Chloronaphthalene	91-58-7
2-Chlorophenol	95-57-8
4-Chlorophenyl-phenyl ether	7005-72-3
Dibenzofuran	132-64-9
Di-n-butylphthalate	84-74-2
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
3,3'-Dichlorobenzidine	91-94-1
2,4-Dichlorophenol	120-83-2
Diethyl phthalate	84-66-2
2,4-Dimethylphenol	105-67-9
Dimethylphthalate	131-11-3
1,3-Dinitrobenzene	99-65-0
4,6-Dinitro-2-methylphenol	534-52-1
2,4-Dinitrophenol	51-28-5
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
Di-n-octyl phthalate	117-84-0
1,4-Dioxane	123-91-1
Diphenylamine	122-39-4
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclo-pentadiene	77-47-4
Hexachloroethane	67-72-1

Table 9-3. Summary of Target Semivolatile Organic Analytes

Semivolatiles	CAS Number
Isophrone	78-59-1
2-Methylphenol	95-48-7
3/4-Methylphenol	108-59-4 & 106-44-5
Cresol (total)	108-59-4, 106-44-5, & 95-48-7
2-Nitroaniline	88-74-4
3-Nitroaniline	99-09-2
4-Nitroaniline	100-01-6
Nitrobenzene	98-95-3
2-Nitrophenol	88-75-5
4-Nitrophenol	100-02-7
N-Nitrosodiphenylamine	86-30-6
N-Nitroso-di-n-propylamine	621-64-7
2,2'-oxybis (1-Chloropropane)	108-60-1
Pentachlorobenzene	82-62-8
Pentachlorophenol	87-86-5
Phenol	108-95-2
1,2,4-Trichlorobenzene	120-82-1
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2

Table 9-4. Summary of Target PAH Analytes

PAH	CAS Number
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(g,h,i)perylene	191-24-2
Benzo(a)pyrene	50-32-8
Benzo(e)pyrene	192-97-2
Chrysene	218-01-9
Dibenzo(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Indeno(1,2,3-cd)pyrene	193-39-5
2-Methylnaphthalene	91-57-6
Naphthalene	91-20-3
Perylene	
Phenanthrene	85-01-8
Pyrene	129-00-0

Table 9-5. Summary of Target PCB Analytes

PCB	IUPAC	CAS Number
3,4,3',4'-Tetrachlorobiphenyl	77	32598-13-3
3,4,4',5-Tetrachlorobiphenyl		70362-50-4
2,3,4,3',4'-Pentachlorobiphenyl	105	32598-14-4
2,3,4,5,4'-Pentachlorobiphenyl	114	74472-37-0
2,4,5,3',4'-Pentachlorobiphenyl	118	31508-00-6
3,4,5,2',4'-Pentachlorobiphenyl	123	65510-44-3
3,4,5,3',4'-Pentachlorobiphenyl	126	57465-28-8
2,3,4,5,3',4'-Hexachlorobiphenyl	156	38380-98-4
2,3,4,3',4',5'-Hexachlorobiphenyl	157	68782-90-7
2,4,5,3',4',5'-Hexachlorobiphenyl	167	52663-72-6
3,4,5,3',4',5'-Hexachlorobiphenyl	169	32774-16-6
2,3,4,5,3',4',5'-Heptachlorobiphenyl	189	39635-31-9
Total Homologue Groups (Sum to Determine total PCBs)		
Monochlorobiphenyls		
Dichlorobiphenyls		
Trichlorobiphenyls		
Tetrachlorobiphenyls		
Pentachlorobiphenyls		
Hexachlorobiphenyls		
Heptachlorobiphenyls		
Octachlorobiphenyls		
Nonachlorobiphenyls		
Decachlorobiphenyls		

Table 9-6. Summary of Target OCP Analytes

OCP	CAS Number
1,2-Dibromo-3-chloropropane	96-12-8
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Aldrin	309-00-2
α -BHC	319-84-6
β -BHC	319-85-7
γ -BHC (Lindane)	58-89-9
δ -BHC	319-86-8
Chlorobenzilate	510-15-6
α -Chlordane	5103-71-9
δ -Chlordane	5103-74-2
Diallate	2303-16-4
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3

Table 9-7. Summary of Target Dioxin/Furan Analytes

Dioxin/Furan Compounds	CAS Number
2,3,7,8-TCDD	1746-01-6
Total TCDD	NA
2,3,7,8-TCDF	51207-31-9
Total TCDF	NA
1,2,3,7,8-PeCDD	40321-76-4
Total PeCDD	NA
1,2,3,7,8-PeCDF	57117-41-6
2,3,4,7,8-PeCDF	57117-31-4
Total PeCDF	NA
1,2,3,6,7,8-HxCDD	57653-85-7
1,2,3,4,7,8-HxCDD	39227-28-6
1,2,3,7,8,9-HxCDD	19408-74-3
Total HxCDD	NA
1,2,3,6,7,8-HxCDF	57117-44-9
1,2,3,4,7,8-HxCDF	70648-26-9
1,2,3,7,8,9-HxCDF	72918-21-9
2,3,4,6,7,8-HxCDF	60851-34-5
Total HxCDF	NA
1,2,3,4,6,7,8-HpCDD	35822-39-4
Total HpCDD	NA
1,2,3,4,6,7,8-HpCDF	67562-39-4
1,2,3,4,7,8,9-HpCDF	55673-89-7
Total HpCDF	NA
Total OCDD	3268-87-9
Total OCDF	39001-02-0

Table 9-8 Summary of Target Metal Analytes

Metal	CAS Number
Aluminum	7429-90-5
Antimony	7440-36-0
Arsenic	7440-38-2
Barium	7440-39-3
Beryllium	7440-41-7
Cadmium	7440-43-9
Chromium	7440-47-3
Cobalt	7440-48-4
Copper	7440-50-8
Lead	7439-92-1
Manganese	7439-96-5
Mercury	7439-97-6
Nickel	7440-02-0
Selenium	7782-49-2
Silver	7440-22-4
Thallium	7440-28-0
Vanadium	7440-62-2
Zinc	7440-66-6

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b	
Total chloride	Spent Activated Carbon	3	Bomb or flask combustion (ASTM Method D-808 or E-442; SW-846 Method 5050)	Ion chromatography of residue (SW-846 Method 9056)	Duplicate	One per test	3	6	
Ash	Spent Activated Carbon	3	NA	Residue after muffle furnace combustion (ASTM Method D-482)	Duplicate	One per test	1	4	
Elemental	Spent Activated Carbon	3	Ultimate analysis (ASTM Method D-3176)	Ultimate analysis (ASTM Method D-3176)	Duplicate	One per test	1	4	
Volatiles	Spent Activated Carbon	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl. Duplicates ^c	6	6	
					Duplicate	One per test	1		
					MS/MSD ^{d,e}	One set test	2		
	Scrubber blowdown	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl.	6	6	
					Duplicate	One per test	1		
					MS/MSD ^{d,e}	One set test	2		
	POTW discharge	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl.	6	6	
					Duplicate	One per test	1		
					MS/MSD ^{d,e}	One set test	2		
	Makeup Water	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl. Duplicates ^c	6	6	
					Duplicate	One per test	1		
					MS/MSD ^{d,e}	One set test	2		
	Caustic	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl.	6	6	
					Duplicate	One per test	1		
					MS/MSD ^{d,e}	One set test	2		
	POHC Spike		3	NA	None; archive for analysis of required	NA	NA	NA	NA
	Analytical system QC	NA	NA	NA	Purge and trap, GC/MS (SW-846 Method 8260)	LCS	One per batch/matrix specific	2 or more	2 or more
						Method Blank	One per batch/matrix specific	2 or more	2 or more

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Semivolatiles	Spent Activated Carbon	3	Waste Dilution(SW-846 Method 3580)	GC/MS (SW-846 Method 8270)	Surrogate spikes incl. duplicates ^c	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD ^{d,e}	One set test	2	
	Scrubber blowdown	3	Waste Dilution(SW-846 Method 3580)	GC/MS (SW-846 Method 8270)	Surrogate spikes incl. duplicates ^c	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD ^{d,e}	One set test	2	
	POTW discharge	3	Waste Dilution(SW-846 Method 3580)	GC/MS (SW-846 Method 8270)	Surrogate spikes	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD ^{d,e}	One set test	2	
	Makeup Water	3	Waste Dilution(SW-846 Method 3580)	GC/MS (SW-846 Method 8270)	Surrogate spikes incl. duplicates ^c	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD ^{d,e}	One set test	2	
	Caustic	3	Liquid-Liquid Extraction (SW-846 Method 3510)	GC/MS (SW-846 Method 8270)	Surrogate spikes incl. duplicates ^c	Every sample	6	6
					Duplicate	One per test	1	
MS/MSD ^{d,e}					One set test	2		
Analytical system QC	NA		GC/MS (SW-8270)	LCS	One per batch/ matrix specific	2 or more	2 or more	
				Method Blank	One per batch/ matrix specific	2 or more	2 or more	
Metals by ICP	Spent Activated Carbon	3	Digestion (SW-846 Method 3051)	ICP (SW-846 Method 6010 or 6020)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	
	Scrubber blowdown	3	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	
	POTW discharge	3	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	
	Makeup Water	3	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Metals (Cont'd)	Caustic	3	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	
	Metals Spiking Solution	1	None; archive for analysis of required	None; archive for analysis of required	NA	NA	NA	NA
	Analytical system QC (aqueous)	NA	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	LCS	One per batch/ matrix specific	2 or more	6 or more
					Serial dilution	One per batch/ matrix specific	2 or more	
Method Blank					One per batch/ matrix specific	2 or more		
Analytical system QC (solids)	NA	Digestion (SW-846 Method 3051)	ICP (SW-846 Method 6010 or 6020)	LCS	One per batch/ matrix specific	2 or more	6 or more	
				Serial dilution	One per batch/ matrix specific	2 or more		
				Method Blank	One per batch/ matrix specific	2 or more		
Mercury by CVAA	Spent Activated Carbon	3	CVAA (SW-846 Method 7471)	CVAA (SW-846 Method 7471)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	
	Makeup Water	3	CVAA (SW-846 Method 7470)	CVAA (SW-846 Method 7471)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	
	Scrubber blowdown	3	CVAA (SW-846 Method 7470)	CVAA (SW-846 Method 7471)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	
	POTW discharge	3	CVAA (SW-846 Method 7470)	CVAA (SW-846 Method 7471)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	
	Caustic	3	CVAA (SW-846 Method 7471)	CVAA (SW-846 Method 7471)	MS ^d	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS ^d	One per test	1	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Mercury by CVAA (cont.)	Analytical system QC (aqueous)	NA	CVAA (SW-846 Method 7470)	CVAA (SW-846 Method 7471)	LCS	One per batch/matrix specific	3 or more	3 or more
					Method Blank	One per batch/matrix specific	2 or more	
	Analytical system QC (solids)	NA	CVAA (SW-846 Method 7471)	CVAA (SW-846 Method 7471)	LCS	One per batch/matrix specific	3 or more	3 or more
					Method Blank	One per batch/matrix specific	2 or more	
VOST for Volatile PICs and TICs	VOST stack sample tube pairs	9	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes ^c	Every sample	18	18
	VOST Condensate	3	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260)	Surrogate spikes ^c	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD ^{d,e}	One set test	2	
	VOST field blank tube pairs	1	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes ^c	Every sample	1	2
	VOST trip blank tube pairs	1	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes ^c	Every sample	1	2
	VOST audit sample tube pairs	2	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes ^c	Every sample	4	4
	Spiked resin blank tube pairs	2	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes ^c	Every sample	2	4
Analytical system QC	NA	NA	Purge and trap, GC/MS (SW-846 Method 8260)	LCS	1 per condensate batch	2 or more	2 or more	
				Method blank	1 per analytical run	2 or more	2 or more	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Dioxin/Furans by Method 0023A	Method 0023A front half composite: filter, and front half of filter holder and probe rinses.	3	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	3	3
					Isotopically labeled surrogate spike	Every sample	3	
	Method 0023A back half composite: XAD-2 resin, and back half of filter holder and condenser rinses.	3	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	3	3
					Isotopically labeled surrogate spike	Every sample	3	
	Method 0023A blank train front half composite: filter, and front half of filter holder and probe rinses.	1	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
					Isotopically labeled surrogate spike	Every sample	1	
	Method 0023A blank train back half composite: XAD-2 resin, and back half of filter holder and condenser rinses.	1	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
					Isotopically labeled surrogate spike	Every sample	1	
	Method 0023A spiked XAD-2 resin blanks	2	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	2	2
					Isotopically labeled surrogate spike	Every sample	2	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Dioxin/Furans by Method 0023A (cont'd)	Method 0023A acetone reagent blanks	1	NA	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
	Method 0023A methylene chloride reagent blanks	1	NA	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
	Method 0023A toluene reagent blanks	1	NA	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
	Analytical system QC	NA	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Method blank	1 per analytical batch	1 or more	1 or more
					Blank spike	2 per analytical batch	2	2
Metals by Method 29	Method 29 front half: filter, and nitric acid probe and front half filter holder rinses	3	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	PDS ^d	One per test	1	4
				CVAA (SW-846 Method 7471)	PDS ^d	One per test	1	7
					Duplicate	Every sample	3	
	Method 29 10%HNO ₃ /5%H ₂ O ₂ impinger contents and rinses	3	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	PDS ^d	One per test	1	4
				CVAA (SW-846 Method 7471)	PDS ^d	One per test	1	7
					Duplicate	Every sample	3	
	Method 29 initially empty contents and rinses	3	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	PDS ^d	One per test	1	7
					Duplicate	Every sample	3	
	Method 29 4%KMnO ₄ /10%H ₂ SO ₄ impinger contents and rinses	3	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	PDS ^d	One per test	1	7
					Duplicate	Every sample	3	
	Method 29 4%KMnO ₄ /10%H ₂ SO ₄ 8N HCl rinses	3	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	PDS ^d	One per test	1	7
					Duplicate	Every sample	3	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Metals by Method 29 (cont'd)	Method 29 blank train front half: filter, and nitric acid probe and front half filter holder rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	MS ^d	One for test program	1	1
				CVAA (SW-846 Method 7471)	MS ^d	One for test program	1	1
				ICP (SW-846 Method 6010 or 6020)	MSD ^d	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD ^d	One for test program	1	1
	Method 29 blank train back half composite: 10%HNO ₃ /5%H ₂ O ₂ impinger contents and rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	MS ^d	One for test program	1	1
				CVAA (SW-846 Method 7471)	MS ^d	One for test program	1	1
				ICP (SW-846 Method 6010 or 6020)	MSD ^d	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD ^d	One for test program	1	1
	Method 29 blank train initially empty contents and rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	MS ^d	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD ^d	One for test program	1	1
	Method 29 blank train 4%KMnO ₄ /10%H ₂ SO ₄ impinger contents and rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	MS ^d	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD ^d	One for test program	1	1
	Method 29 blank train 4%KMnO ₄ /10%H ₂ SO ₄ impinger 8N HCl rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	MS ^d	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD ^d	One for test program	1	1

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Metlas by Method 29 (cont'd)	Method 29 filter reagent blank	1	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	Reagent Blank	One for test program	1	1
				CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	#REF!
	Method 29 HNO ₃ reagent blank	1	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	Reagent Blank	One for test program	1	1
				CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	Method 29 10%HNO ₃ /5%H ₂ O ₂ reagent blank	1	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	Reagent Blank	One for test program	1	1
				CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	4%KMnO ₄ /10%H ₂ SO ₄ reagent blank	1	Digestion (SW-846 Method 3051)	CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	8N HCl reagent blank	1	Digestion (SW-846 Method 3051)	CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	Analytical system QC	NA		ICP (SW-846 Methods 3051, 6010 or 6020) and CVAA (SW-846 Method 7471)	LCS	one per batch/ matrix specific	1 or more	3 or more
					Serial dilution	one per batch/ matrix specific	1 or more	
Method blank					one per batch/ matrix specific	1 or more		

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (two separate trains collected and analyze. The first for PCB & PAH. The second for SVOC & OCP	Method 0010 front half composite: filter and probe rinses	3	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	Semivolatile surrogate spikes	Every sample	3	3
					Semivolatile internal standard surrogate spikes	Every sample	3	
				GC for OCP (SW-846 Method 8081)	Semivolatile surrogate spikes	Every sample	3	3
					Semivolatile internal standard surrogate spikes	Every sample	3	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH isotope dilution internal standard spike	Every sample	3	3
					PAH recovery standard spike	Every sample	3	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB isotope dilution internal standard spike	Every sample	3	3
					PCB recovery standard spike	Every sample	3	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b	
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd) (two separate trains collected and analyze.) The first for PCB & PAH. The second for SVOC & OCP	Method 0010 back half composite: XAD-2 resin, condenser rinses	3	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	¹³ C-labeled pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	3	3	
					Semivolatile surrogate spikes	Every sample	3		
					Semivolatile internal standard surrogate spikes	Every sample	3		
					GC for OCP (SW-846 Method 8081)	¹³ C-labeled pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	3	3
						organochlorinated pesticide surrogate spikes	Every sample	3	
						organochlorinated pesticide standard surrogate spikes	Every sample	3	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	3	3	
					PAH isotope dilution internal standard spike	Every sample	3		
					PAH recovery standard spike	Every sample	3		
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	3	3	
					PCB isotope dilution internal standard spike	Every sample	3		
					PCB recovery standard spike	Every sample	3		

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd)	Method 0010 condensate impinger	3	Separatory funnel acid/base extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	Semivolatile surrogate spikes	Every sample	3	3
					Semivolatile internal standard surrogate spikes	Every sample	3	
				GC for OCP (SW-846 Method 8081)	organochlorinated pesticide surrogate spikes	Every sample	3	3
					organochlorinated pesticide internal standard surrogate	Every sample	3	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH isotope dilution internal standard spike	Every sample	3	3
					PAH recovery standard spike	Every sample	3	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB isotope dilution internal standard spike	Every sample	3	3
					PCB recovery standard spike	Every sample	3	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd)	Method 0010 blank train front half composite: filter and probe rinses	1	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	Semivolatile surrogate spikes	Every sample	1	1
					Semivolatile internal standard surrogate spikes	Every sample	1	
				GC for OCP (SW-846 Method 8081)	organochlorinated surrogate spikes	Every sample	1	1
					organichlorinated pesticide internal standard surrogate spikes	Every sample	1	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH isotope dilution internal standard spike	Every sample	1	1
					PAH recovery standard spike	Every sample	1	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB isotope dilution internal standard spike	Every sample	1	1
					PCB recovery standard spike	Every sample	1	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b	
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd)	Method 0010 blank train back half composite: XAD-2 resin, condenser rinses	1	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	¹³ C-labeled pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	1	1	
					Semivolatile surrogate spikes	Every sample	1		
					Semivolatile internal standard surrogate spikes	Every sample	1		
					GC for OCP (SW-846 Method 8081)	¹³ C-labeled pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	0	0
						Semivolatile surrogate spikes	Every sample	0	
						Semivolatile internal standard surrogate spikes	Every sample	0	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	1	1	
					PAH isotope dilution internal standard spike	Every sample	1		
					PAH recovery standard spike	Every sample	1		
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	1	1	
					PCB isotope dilution internal standard spike	Every sample	1		
					PCB recovery standard spike	Every sample	1		

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b			
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd)	Spiked resin blanks (Matrix spikes)	2	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	Semivolatile surrogate spikes	Every sample	2	2			
					Semivolatile internal standard surrogate spikes	Every sample	2				
				GC for OCP (SW-846 Method 8081)	OCP surrogate spikes	Every sample	2	2			
					OCP internal standard surrogate spikes (optional)	Every sample	2				
				HRGC/HRMS for PAHs (CARB Method 429)	PAH isotope dilution internal standard spike	Every sample	2	2			
					PAH recovery standard spike	Every sample	2				
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB isotope dilution internal standard spike	Every sample	2	2			
					PCB recovery standard spike	Every sample	2				
				Analytical system QC	NA	NA	GC/MS for SVOCs (SW-846 Method 8270)	LCS	1 per analytical batch	2 or more	4 or more
								Method Blank	1 per analytical batch	2 or more	
GC for OCP (SW-846 Method 8081)	LCS	1 per analytical batch	2 or more				4 or more				
	Method Blank	1 per analytical batch	2 or more								
HRGC/HRMS for PAHs (CARB Method 429)	LCS	1 per analytical batch	2 or more				4 or more				
	Method Blank	1 per analytical batch	2 or more								
HRGC/HRMS for PCBs (EPA Draft Method 1668A)	LCS	1 per analytical batch	2 or more				4 or more				
	Method Blank	1 per analytical batch	2 or more								

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Total semivolatile and nonvolatile organics by M0010 EPA TOE Guidance	Semivolatile front half composite: filter and solvent probe rinses	3	Soxhlet extraction (SW-846 Method 3542)	GC/FID analysis of one-half of pooled extracts (SW-846 Method 8015)	Duplicate	One per test	1	4
	Semivolatile back half composite: XAD-2 resin and condensor rinses	3						
	Semivolatile condensate inpinger and rinses	3		Gravimetric (Grav) analysis of one-half of pooled extracts (TOE Guidance)	Duplicate	Every sample	3	6
	Semivolatile blank train front half composite: filter and solvent probe rinses	1	Soxhlet extraction (SW-846 Method 3542)	GC/FID analysis of one-half of pooled extracts (SW-846 Method 8015)	Blank train	One per test	1	1
	Semivolatile blank train back half composite: XAD-2 resin and condensor rinses	1		Gravimetric (Grav) analysis of one-half of pooled extracts (TOE Guidance)				
	Methanol/ methylene chloride reagent blank	1	NA	GC/FID (SW-846 Method 8015)	Reagent blank	One per test	1	1
				Gravimetric (Grav) (TOE Guidance)				
	Filter blank	1	Soxhlet extraction (SW-846 Method 3542)	GC/FID (SW-846 Method 8015)	Reagent blank	One per test	1	1
				Gravimetric (Grav) (TOE Guidance)				
	XAD-2 resin blanks	2	Soxhlet extraction (SW-846 Method 3542)	GC/FID (SW-846 Method 8015)	Reagent blank	One per test	2	2
Gravimetric (Grav) (TOE Guidance)				One per test				

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Total volatile organics by M0040	Tedlar Bags	6	NA	GC/FID (Modified SW-846 Method 0040)	Duplicate	Every sample	6	18
	Field Blank Bag	3	NA	GC/FID (Modified SW-846 Method 0040)	Field blank	One per run	3	
	Condensate	3	NA	Purge and trap GC/FID (SW-846 Methods 0040 and 5030)	Duplicate	Every sample	3	8
	Condensate blank	1	NA	Purge and trap GC/FID (SW-846 Methods 0040 and 5030)	Field blank	One per run	1	
	Analytical system QC	NA	NA	GC/FID (Modified SW-846 Method 0040)	Zero gas	One per run	3	9
					Known gas	Two per run	6	
Purge and trap GC/FID (SW-846 Methods 0040 and 5030)					Method blank (water)	One per analytical batch	1 or more	1 or more
HCl by Method 26A	Method 26A H ₂ SO ₄ impingers	9		Ion chromatography (SW-846 Method 9057)	Duplicate	Every sample	9	22
					MS/MSD analyzed in duplicate ^d	1 per batch (assuming all samples batched together)	4	
	Method 26A H ₂ SO ₄ reagent blank	1		Ion chromatography (SW-846 Method 9057)	Duplicate	Every sample	1	2
Cl ₂ Method 26A	Method 26A NaOH impingers	9		Ion chromatography (SW-846 Method 9057)	Duplicate	Every sample	9	22
					MS/MSD analyzed in duplicate ^d	1 per batch (assuming all samples batched together)	4	
	Method 26A NaOH reagent blank	1		Ion chromatography (SW-846 Method 9057)	Duplicate	Every sample	1	2

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples ^a	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency ^a	QC Analyses	Total Analyses ^b
Cl ⁻ ion chromatography	Analytical system QC			Ion chromatography (SW-846 Method 9057)	LCS/LCSD	1 per batch following initial calibration (separate calibration for each matrix)	4	8
					Method Blank	One per batch/ matrix specific - analyzed in duplicate	4	
Particle size distribution by CARB 501	Cascade Impactor Filter	3		Gravimetric	Blank Filter	Once per test	1	4
Particulate by Method 26A	Method 26A particulate filter	9		Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	9	9
	Method 26A probe and filter holder acetone	9		Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	9	9
	Method 26A acetone reagent blank	1		Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	1	1

^a Each test condition is comprised of three replicate sampling runs. There is only one test condition planned for this test program. Refer to QAPP Tables 6-1 and 9-1.

^b Total laboratory analyses includes field sample analyses and laboratory QC analyses

^c Surrogate spikes are applied to all samples. Refer to Table 5-2 for the surrogate compounds.

^d MS = Matrix spike
MS = Matrix spike duplicate
PDS = Post digestion spikes

^e Refer to Table 5-2 for the matrix spike compounds.

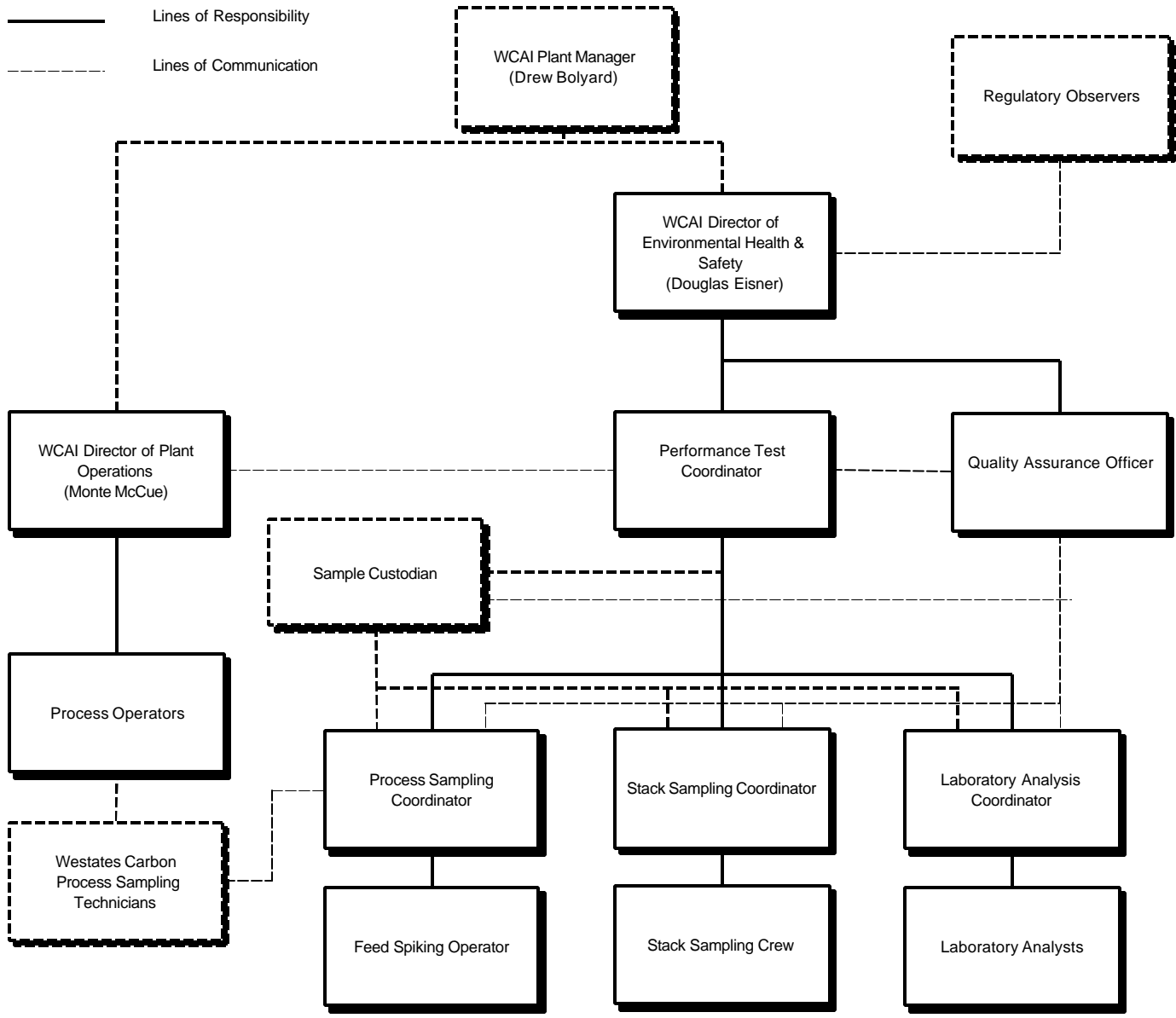


Figure 4-1. Performance Test Project Organization and Responsibility

Figure 7-1. Example of Chain of Custody Form

Custody Record
Focus Environmental, Inc.

Client: _____

Description: _____

Note: This form is to be accompanied by a "Request for Analysis" which specifies the preparation and analysis to be performed for each sample.

Location: _____

Project No.: _____

Sample ID	Description	Container	No.	Grab/Comp	Date	Time	Remarks
1-1-M29-FIL	Stack Gas M29 Filter (Container 1)	Glass Petri Dish		Comp.	01/01/01	01:00	

Sampler: (Signature)	Date/Time 	Received by: (Signature)
Relinquished by: (Signature)	Date/Time 	Received by: (Signature)
Relinquished by: (Signature)	Date/Time 	Received by: (Signature)

Figure 7-2. Example Request For Analysis Form

REQUEST FOR ANALYSIS

Focus Environmental, Inc.

Client: _____

Description: _____

Location: _____

Project No.: _____

Sample Identification

1-1-M29-FIL

Test No.: 1

Description:

Run No.: 1

Stack Gas M29 Filter (Container 1)

Lab: Analytical Laboratory

Container: Glass Petri Dish

Number of Containers: 1

Requested Preparation/Analyses:

Metals – Mercury – Liquids CVAAS (SW846-7470)

Metals – Multiple (Specify) ICP (SW846-6010)

Preservation

None required

Preparation Method:

N/A

Acid Digestion – Stack Gas Multi-Metals

Hold Time

180 days/28 days for Hg

Special Instructions

Analyze for metals: Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Mn, Hg, Ni, P, Se, Ag, Tl, V, Zn.

Figure 7-3. Example Field Sampling Record

Westates,Carbon – Arizona, Inc. Parker, Arizona

Project No. 010111

Test # _____ Run # _____ Date & Time of Collection _____

Frequency _____

Sampler(s) _____

Sample Type (circle one): Composite Aliquot Grab

Sample Description (if grab sample was taken before during or after test):

Sample Source:

Composite Sampling Time Log

1)_____ 2)_____ 3)_____ 4)_____ 5)_____ 6)_____

7)_____ 8)_____ 9)_____ 10)_____ 11)_____ 12)_____

13)_____ 14)_____ 15)_____ 16)_____ 17)_____ 18)_____

19)_____ 20)_____ 21)_____ 22)_____ 23)_____ 24)_____

25)_____ 26)_____ 27)_____ 28)_____ 29)_____ 30)_____

Volume of each Grab _____ Total Number of Grabs _____

Final pH of Sample _____ Final Volume of Sample _____

Sample Container (circle one):

1 L Boston Round 1 L Wide-Mouth 500 mL Wide-Mouth

40 mL VOA Vial 20 mL VOA Vial 1 gallon. Amber Wheaton Jug

120 mL Wide-Mouth 2.5 L Jug Other: _____

Comments:

Samplers Signature/Date: _____

Figure 7-4. Example Sample Label Format

CLIENT:	Test No.: 1
PROJECT:	Run No.: 1
SAMPLER:	LAB:
SAMPLE ID.: <u>1-1-M29-FIL</u>	
DATE: <u>01/01/01</u>	
DESCRIPTION:	
Stack Gas M29 Filter (Container 1)	
Container ___ of ___	<i>Focus Environmental, Inc.</i>

Figure 7-5. Planned Sample Identification Codes

T1 R1 -XXX YY-Z

Special Code (as required)
A, B, etc. Component A, B, etc.
DUP Field Duplicate
FB Field Blank
RB Reagent Blank
TB Trip Blank
ARC Archive
AUD Audit

Sample Type

Solid/Liquid Samples
SVOC Semivolatile Organics
VOC Volatile Organics
MET Metals
PROP Physical/Chemical Properties(Density, Ash, etc)
ARCH Archive

Stack Gas M0030 Train (VOST)

TEN Tenax Resin Tube
CHA Tenax Resin/Charcoal Tube
CON Condensate

Stack Gas M26A train (Particulate & HCl/Cl2)

FIL Filter
FHR Front Half Acetone Rinse
KOA Knockout & Acid Impinger and Rinse
ALK Alkaline Impinger and rinse
H2SO4 H2SO4 (blank)
NaOH NaOH (blank)
DIW Deionized Water (blank)
ACE Acetone (blank)

Stack Gas M0010A Train (SVOA & OCP)

FIL Filter
FHR Front Half Methanol/Methylene Chloride Rinse
XAD XAD-2 Resin Trap
CON Condensate
BHR Back Half Methanol/Methylene Chloride Rinse
MECL2 Methylene Chloride Rinse Reagent (blank)
MEOH Methanol Rinse Reagent (blank)

Stack Gas PSD

PSD Cascade impactor filters

Sample Location

M26A Method 26A Train
0023A Method 0023A Train
0010A Method 0010-SV Train (SVOA & OCP)
0010B Method 0010-SV Train (PCB & PAH)
0010C Method 0010-TOE Train
0030 Method 0030 Train
0040 Method 0040 Train
00061 Method 0061 train
M29 Method 29 train
SAC Spent Activated Carbon
MUW Makeup water
CAS Caustic
APC Scrubber blowdown
POTW POTW discharge water

Run Number

R0 Applies to Entire Test Condition
R1, R2, etc. Run 1, Run 2, etc.

Test Condition Number

T0 Applies to Entire Test Program
T1, T2, etc. Test 1, Test 2, etc.

Stack Gas M0023A Train (D/F)

FIL Filter
RIN Front Half & Back Half Acetone & Methylene Chloride Rinses
TRIN Front Half & Back Half Toluene Rinses
XAD XAD-2 Resin Trap
CON Condensate (for SVOCs only)
ACE Acetone Reagent (blank)
MECL2 Methylene Chloride Reagent (blank)
TOL Toluene Reagent (blank)

Stack Gas M0010C Train (Total SV & NV Organics)

FIL Filter
FHR Front Half Methanol/Methylene Chloride Rinse
XAD XAD-2 Resin Trap
CON Condensate
BHR Back Half Methanol/Methylene Chloride Rinse
MECL2 Methylene Chloride Rinse Reagent (blank)
MEOH Methanol Rinse Reagent (blank)

Stack Gas M0040 Train

BAG Gas Bag
CON Condensate

Stack gas M29 train (Metals)

FIL Filter
PRO Probe Rinse
APL Acidified Peroxide Impinger Liquid
IEL Initially Empty Impinger Liquid
PPL Potassium Permanganate Impinger Liquid
RIN HCl Rinse of Permanganate Impingers
NIT Nitric Acid Solution Reagent (blank)
H2O2 Nitric Acid/Hydrogen Peroxide Reagent (blank)
KMNO4 Potassium Permanganate Reagent (blank)
HCL Hydrochloric Acid Reagent (blank)

Stack Gas M0061 Train

LIQ Impinger Solution and Rinses
KOH Potassium Hydroxide Reagent (blank)
WAT Water Reagent (blank)
HNO3R Nitric Acid Rinse
HNO3 Nitric Acid Reagent (blank)
FIL Filter & Residue

Stack Gas M0010B Train (PCB & PAH)

FIL Filter
FHR Front Half Methanol/Methylene Chloride Rinse
XAD XAD-2 Resin Trap
CON Condensate
BHR Back Half Methanol/Methylene Chloride Rinse
MECL2 Methylene Chloride Rinse Reagent (blank)
MEOH Methanol Rinse Reagent (blank)

Other

X Not Specified, or Multi-Purpose

Figure 11-1. Example Performance Test Report Outline

- 1.0 EXECUTIVE SUMMARY
- 2.0 TEST PROGRAM SUMMARY
 - 2.1 Engineering Description
 - 2.1.1 General Description
 - 2.1.2 Residence Time Determination
 - 2.1.3 Burner Description
 - 2.1.4 Waste Feed Systems
 - 2.1.5 Auxiliary Fuel System
 - 2.1.6 Air Pollution Control System
 - 2.1.7 Process Monitoring System (CMS)
 - 2.1.8 Continuous Emissions Monitoring System (CEMS)
 - 2.1.9 Automatic Waste Feed Cutoff System
 - 2.2 Summary of Test Plan and Objectives
 - 2.3 Test Implementation Summary
 - 2.3.1 Deviations from the Test Plan
- 3.0 PROCESS OPERATIONS
 - 3.1 Process Operating Conditions
 - 3.2 Feed Material Characteristics
 - 3.3 Feed Material Spiking
 - 3.4 Effluent Characteristics
- 4.0 COMPLIANCE RESULTS
 - 4.1 POHC Destruction and Removal Efficiency
 - 4.2 Particulate Emissions
 - 4.3 Hydrogen Chloride and Chlorine Emissions
 - 4.4 Metals Emissions
 - 4.5 Stack Gas Oxygen, Carbon Monoxide, and Total Hydrocarbons
 - 4.6 Dioxin and Furan Emissions
- 5.0 QUALITY ASSURANCE/QUALITY CONTROL RESULTS
 - 5.1 QA/QC Activities and Implementation
 - 5.1.1 QA Surveillance
 - 5.1.2 Sample Collection
 - 5.1.3 Sample Analysis
 - 5.1.4 Process Instrumentation
 - 5.1.5 Stack Sampling Equipment
 - 5.1.6 Laboratory Analytical Instrumentation
 - 5.2 Audits and Data Validation
 - 5.3 Calculations

Figure 11-1. Example Performance Test Report Outline

- 5.4 Conclusions
- 6.0 ANTICIPATED PERMIT OPERATING CONDITIONS
 - 6.1 Development of Operating Limits
 - 6.2 Specific Control Parameters
- 7.0 RECOMMENDED EMISSIONS DATA FOR USE IN RISK ASSESSMENT
 - 7.1 Metals
 - 7.2 Hydrogen Chloride and Chlorine
 - 7.3 Particle Size Distribution
 - 7.4 Speciated Volatile Organics
 - 7.5 Total Volatile Organics
 - 7.6 Speciated Semivolatile Organics
 - 7.7 Total Semivolatile and Nonvolatile Organics
 - 7.8 Dioxins and Furans
 - 7.9 Speciated PAHs
 - 7.10 Speciated Organochlorine Pesticides
 - 7.11 Speciated PCBs

APPENDICES

- A. Process Operating Data
- B. Test Manager's Log
- C. Spiking Report and Certificate of Analysis for Spiking Material
- D. Process Instrument Calibration Data
- E. Continuous Emissions Monitoring Data
- F. Sampling Report
- G. List of Samples
- H. Analytical Report
- I. Calculations
- J. Documentation to Support Metals Extrapolation
- K. Data Validation Report
- L. Corrective Action Requests

Figure 12-1. Example Equipment Inspection Record Form

Client: _____

Project No. _____

Date/Time of inspection: _____

Equipment Inspected:

1) _____

Condition: Good See Problems Section Below

2) _____

Condition: Good See Problems Section Below

3) _____

Condition: Good See Problems Section Below

4) _____

Condition: Good See Problems Section Below

5) _____

Condition: Good See Problems Section Below

Problems Noted:

Action Taken

Inspector's Signature: _____

Figure 14-1. Example Corrective Action Request Form

Number:

Date:

File Name: Westates Carbon – Arizona, Inc. Performance Demonstration Test

REQUEST

To:

You are hereby requested to take corrective actions indicated below and as otherwise determined by you (A) to resolve the noted condition and (B) to prevent it from recurring. Your written response is to be returned to the project Quality Assurance Officer or other responsible manager by.

Condition:

Reference Documents:

Recommended Corrective Actions:

Originator

Date

Approval

Date

Approval

Date

RESPONSE

Cause of Condition:

Resolution:

Prevention:

Affected Documents:

Signature

Date

QA Follow-up on
Corrective Action Verified

Date

**ATTACHMENT D
CALCULATIONS**

POHC AND ORGANIC SURROGATE SPIKING RATES

VOLATILE POHC SPIKING DATASHEET

Evaluates POHC spiking rate needed for desired DRE demonstration, and accounts for PICs/background

Client: **Westates Carbon - Arizona, Inc.**
 Facility: **Parker, AZ RF-2**
 Case: **Performance Demo Test**

Proj. No.: **010111**
 By: **ARE**
 Date: **27-Jan-03**
 Worksheet: **MCB-POHC.WK4**

Input Data

POHC Name	Monochlorobenzene
POHC Molecular Weight	112.5
Dry Stack Gas Flow Rate (dscfm)	3878
DRE Requirement (%)	99.99
Expected DRE (%)	99.9999
Lower Quantitation Limit (ng/tube set)	10
Upper Quantitation Limit (ng/tube set)	1000
Sample Volume (l, dry std./tube set)	20
Actual POHC Spiking (lb/hr)	35
Analytical Safety Factor	10
POHC Background Concentration in Stack Gas (ng/l)	0
POHC Contamination in Sample Train (ng/tube set)	0

Minimum Emissions and Spiking Required to Demonstrate Regulatory DRE

% DRE (Regulatory Limit)	99.99
Stack Emission Rate Required for Detection (lb/hr)	7.26E-06
Min. Stack Conc. to Demonstrate DRE (ng/l, dry std.)	5.00E-01
Min. Stack Conc. to Demonstrate DRE (ppbvds)	0.107
Min. Spiking Rate to Demonstrate DRE (lb/hr)	0.0726
Spiking Rate With Analytical Safety Factor Applied (lb/hr)	0.726

Minimum Emissions and Spiking Required to Demonstrate Expected DRE

% DRE (Expected Performance)	99.9999
Stack Emission Rate Required for Detection (lb/hr)	7.26E-06
Min. Stack Conc. to Demonstrate DRE (ng/l, dry std.)	5.00E-01
Min. Stack Conc. to Demonstrate DRE (ppbvds)	0.107
Min. Spiking Rate to Demonstrate DRE (lb/hr)	7.2625
Spiking Rate With Analytical Safety Factor Applied (lb/hr)	72.625

Potential Emissions and DRE Demonstration Capability at Actual Spiking Rate

Spiking Rate (lb/hr)	35
POHC Emission at Regulatory DRE (lb/hr)	3.50E-03
POHC in Stack Gas at Regulatory DRE (ng/l, dry std.)	2.41E+02
POHC in Stack Gas at Regulatory DRE (ppbvds)	51.5315
POHC Emissions at Expected DRE (lb/hr)	3.50E-05
POHC in Stack Gas at Expected DRE (ng/l, dry std.)	2.41E+00
Train Loading at Regulatory DRE (ng/tube set)	4819.3
Train Loading at Expected DRE (ng/tube set)	48.2
Max. DRE Demonstrated at Spiking Rate (%)	99.99997925

Impact of Sample/Combustion Gas PICs and Background POHC Concentration

Emissions at Regulatory DRE from Actual Spiking (lb/hr)	3.50E-03
Emissions Equivalent from PICs, Background (lb/hr)	0.00E+00
Apparent Total Emissions w/PICs & Background (lb/hr)	3.50E-03
Apparent DRE at Reg. DRE w/PICs & Background (%)	99.99
Added Sample Train Loading from PICs/Background (ng)	0.00
DRE Required to Counter PICs/Background (%)	99.99

Notes:

Actual spiking rate determined based on need for chlorine feed rate rather than POHC detection.

VOLATILE POHC SPIKING DATASHEET

Evaluates POHC spiking rate needed for desired DRE demonstration, and accounts for PICs/background

Client: **Westates Carbon - Arizona, Inc.**
 Facility: **Parker, AZ RF-2**
 Case: **Performance Demo Test**

Proj. No.: **010111**
 By: **ARE**
 Date: **27-Jan-03**
 Worksheet: **Perc-POHC.WK4**

Input Data

POHC Name	Tetrachloroethene
POHC Molecular Weight	116
Dry Stack Gas Flow Rate (dscfm)	3878
DRE Requirement (%)	99.99
Expected DRE (%)	99.9999
Lower Quantitation Limit (ng/tube set)	10
Upper Quantitation Limit (ng/tube set)	1000
Sample Volume (l, dry std./tube set)	20
Actual POHC Spiking (lb/hr)	35
Analytical Safety Factor	10
POHC Background Concentration in Stack Gas (ng/l)	0
POHC Contamination in Sample Train (ng/tube set)	0

Minimum Emissions and Spiking Required to Demonstrate Regulatory DRE

% DRE (Regulatory Limit)	99.99
Stack Emission Rate Required for Detection (lb/hr)	7.26E-06
Min. Stack Conc. to Demonstrate DRE (ng/l, dry std.)	5.00E-01
Min. Stack Conc. to Demonstrate DRE (ppbvds)	0.104
Min. Spiking Rate to Demonstrate DRE (lb/hr)	0.0726
Spiking Rate With Analytical Safety Factor Applied (lb/hr)	0.726

Minimum Emissions and Spiking Required to Demonstrate Expected DRE

% DRE (Expected Performance)	99.9999
Stack Emission Rate Required for Detection (lb/hr)	7.26E-06
Min. Stack Conc. to Demonstrate DRE (ng/l, dry std.)	5.00E-01
Min. Stack Conc. to Demonstrate DRE (ppbvds)	0.104
Min. Spiking Rate to Demonstrate DRE (lb/hr)	7.2625
Spiking Rate With Analytical Safety Factor Applied (lb/hr)	72.625

Potential Emissions and DRE Demonstration Capability at Actual Spiking Rate

Spiking Rate (lb/hr)	35
POHC Emission at Regulatory DRE (lb/hr)	3.50E-03
POHC in Stack Gas at Regulatory DRE (ng/l, dry std.)	2.41E+02
POHC in Stack Gas at Regulatory DRE (ppbvds)	49.9767
POHC Emissions at Expected DRE (lb/hr)	3.50E-05
POHC in Stack Gas at Expected DRE (ng/l, dry std.)	2.41E+00
Train Loading at Regulatory DRE (ng/tube set)	4819.3
Train Loading at Expected DRE (ng/tube set)	48.2
Max. DRE Demonstrated at Spiking Rate (%)	99.99997925

Impact of Sample/Combustion Gas PICs and Background POHC Concentration

Emissions at Regulatory DRE from Actual Spiking (lb/hr)	3.50E-03
Emissions Equivalent from PICs, Background (lb/hr)	0.00E+00
Apparent Total Emissions w/PICs & Background (lb/hr)	3.50E-03
Apparent DRE at Reg. DRE w/PICs & Background (%)	99.99
Added Sample Train Loading from PICs/Background (ng)	0.00
DRE Required to Counter PICs/Background (%)	99.99

Notes:

Actual spiking rate determined based on need for chlorine feed rate rather than POHC detection.

METAL SPIKING RATES

Constituent Removal Performance and Maximum Feed Calculations

CASE: Max Flow RF-2

Metal	SRE (%)	Emission Limit (ug/dscm @7% O2)	Emission Rate at Limit (lb/hr)	Maximum Metal Feed Rate (lb/hr)
Mercury	0.00	130	1.89E-03	1.89E-03
SVM (Cd + Pb)	99.66	240	3.49E-03	1.03E+00
LVM (As + Be + Cr)	99.88	97	1.41E-03	1.17E+00
HCl/Cl2	SRE (%)	Emission Limit (ppmv @7% O2)	Emission Rate at Limit (lb/hr)	Maximum Chloride Feed Rate (lb/hr)
Total chlorine/chloride	99.00	77	1.70E+00	1.65E+02

Basis:

Stack Gas Flow Rate (dscfm)

3878

Stack Gas Oxygen Concentration (vol%, dry)

7

Metal SRE values are averages from previous testing.

Chlorine/chloride SRE is based on minimum engineering specifications for HCl.

EXAMPLE PERFORMANCE TEST CALCULATIONS

EXAMPLE CALCULATIONS

This section presents example calculations to be used for each test determination. Wherever applicable, calculations from the appropriate regulations are used in making the test determinations. In other instances, calculations are based on generally accepted scientific and engineering principles.

A. CONVERSION FACTORS AND CONSTANTS

The following conversion factors and constants are used throughout the calculations:

Volume:

1 cubic meter = 35.31 cubic feet

1 cubic foot = 28.32 liters

Mass:

1 pound = 453.59 grams

1 pound = 7000.0 grains

1 kilogram = 2.2046 pounds

Length:

1 meter = 3.281 feet

Constants:

Standard conditions are 68°F, 29.92 in. Hg, when using English units.

Standard conditions are 20°C, 101.3 KPa (760 mm Hg), when using metric units.

Normal conditions are 0°C, 101.3 KPa (760 mm Hg), when using metric units.

1 lb-mol occupies 385 cubic feet at **standard conditions**.

1 kg-mol occupies 24.04 cubic meters at **standard conditions**.

1 kg-mol occupies 22.4 cubic meters at **normal conditions**.

B. CALCULATIONS

Constituent Input Rates:

Constituent input rates (metals, chlorine/chloride, ash, etc.) for each run are calculated by multiplying the feed rate of each input material times the constituent concentration for that material. The total constituent input rate is calculated by summing the constituent input rate from each input material. Depending upon the form of laboratory results (mass concentration, weight percent, or volumetric measurement) the appropriate formula shown below is used:

Mass Concentration Results:

$$\text{Constituent Input Rate}(\text{mass} / \text{time}) = \sum_i \left([C]_i \times \frac{1 \text{ kg}}{1 \times 10^6 \text{ mg}} \times \dot{m}_i \right)$$

where:

$[C]_i$ = Concentration of constituent C in material i (mg/kg)

\dot{m}_i = Mass feed rate of material i (mass/time)

Weight Percent Results:

$$\text{Constituent Input Rate}(\text{mass} / \text{time}) = \sum_i ([C]_i \times \dot{m}_i)$$

where:

$[C]_i$ = Concentration of constituent C in material i (wt %)

\dot{m}_i = Mass feed rate of material i (mass/time)

Aqueous Sample Mass/Volume Results:

$$\text{Constituent Input Rate}(\text{lb} / \text{hr}) = \sum_i \left([C]_i \times \frac{1 \text{ g}}{1 \times 10^6 \text{ } \mu\text{g}} \times \frac{1 \text{ lb}}{453.59 \text{ g}} \times \dot{m}_i \times \frac{3.785 \text{ l}}{\text{gal}} \times \frac{1 \text{ gal}}{8.33 \text{ lb}} \right)$$

where:

$[C]_i$ = Concentration of constituent C in material i ($\mu\text{g/l}$)

\dot{m}_i = Mass feed rate of material i (lb/hr)

Gaseous Sample Volumetric Results:

$$\text{Constituent Input Rate (lb / hr)} = \sum_i \left([C]_i \times Q_{sd} \times \frac{60 \text{ min}}{\text{hr}} \times MW_c \times \frac{1}{1 \times 10^6} \times \frac{1 \text{ lb - mol}}{385 \text{ dscf}} \right)$$

where:

$[C]_i$ = Concentration of constituent C in material *i* (ppmv, dry)

Q_{sd} = gaseous stream flow rate (dscfm)

MW_c = molecular weight of constituent C

Stack Gas Sampling:

The following equations are used for all stack gas sampling determinations. These calculations are given in the referenced sampling methods and in 40 CFR 60.

Stack Gas Velocity:

$$v_s = K_p \times C_p \times (\sqrt{\Delta p})_{avg} \times \sqrt{\frac{T_{s(avg)}}{P_s \times M_s}}$$

where:

v_s = Average stack gas velocity (m/sec) or (ft/sec)

$$K_p = \text{Pitot tube constant } 34.97 \frac{m}{\text{sec}} \left[\frac{(g / g - mol)(mm Hg)}{(^{\circ}K)(mm H_2O)} \right] \text{ or } 85.49 \frac{ft}{\text{sec}} \left[\frac{(lb / lb - mol)(in. Hg)}{(^{\circ}R)(in. H_2O)} \right]$$

C_p = Pitot tube coefficient, dimensionless

Δp = Velocity head of stack gas (mm H₂O) or (in. H₂O)

T_s = Absolute stack gas temperature (K) or (R)

P_s = Absolute stack gas pressure (mm Hg) or (in. Hg)

M_s = Molecular weight of stack gas, wet basis (g/g-mol) or (lb/lb-mol)

Stack Gas Molecular Weight:

$$M_d = 0.440 \times [CO_2] + 0.320 \times [O_2] + 0.280 \times [N_2 + CO]$$

where:

M_d = Molecular weight of stack gas, dry basis (g/g-mol) or (lb/lb-mol)

$[CO_2]$ = Concentration of carbon dioxide in stack gas (vol%, dry)

$[O_2]$ = Concentration of oxygen in stack gas (vol%, dry)

$[N_2 + CO]$ = Concentration of nitrogen plus carbon monoxide in stack gas (vol%, dry)

$$M_s = M_d \times (1 - B_{ws}) + 18.0 \times B_{ws}$$

where:

M_s = Molecular weight of stack gas, wet basis (g/g-mol) or (lb/lb-mol)

M_d = Molecular weight of stack gas, dry basis (g/g-mol) or (lb/lb-mol)

B_{ws} = Water vapor fraction in stack gas by volume, dimensionless

Stack Gas Volumetric Flow Rate:

$$Q_{sd} = 60 \times (1 - B_{ws}) \times v_s \times A \times \frac{T_{std}}{T_{s(avg)}} \times \frac{P_s}{P_{std}}$$

where:

Q_{sd} = Stack gas volumetric flow rate (dscm/min) or (dscfm)

B_{ws} = Water vapor fraction in stack gas by volume, dimensionless

v_s = Average stack gas velocity (m/sec) or (ft/sec)

A = Cross-sectional area of stack (m²) or (ft²)

T_{std} = Standard absolute temperature (293 K) or (528 R)

T_s = Absolute stack temperature (K) or (R)

P_s = Absolute stack pressure (mm Hg) or (in. Hg)

P_{std} = Standard absolute pressure (760 mm Hg) or (29.92 in. Hg)

Stack Gas Moisture:

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}}$$

where:

B_{ws} = Water vapor fraction in stack gas by volume, dimensionless

$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions (scm) or (scf)

$V_{wsg(std)}$ = Volume of water vapor collected in silica gel, corrected to standard conditions (scm) or (scf)

$V_{m(std)}$ = Gas sample volume (dscm) or (dscf)

$$V_{wc(std)} = \frac{(V_f - V_i) \times r_w \times R \times T_{std}}{P_{std} \times M_w}$$

$$V_{wsg(std)} = \frac{(W_f - W_i) \times R \times T_{std}}{P_{std} \times M_w}$$

where:

V_f = Final volume of condenser water (ml)

V_i = Initial volume of condenser water (ml)

r_w = Density of water (0.9982 g/ml) or (0.002201 lb/ml)

W_f = Final weight of silica gel (g)

W_i = Initial weight of silica gel (g)

R = Ideal gas constant $0.06236 \frac{(mmHg)(m^3)}{(g-mol)(^\circ K)}$ or $21.85 \frac{(in.Hg)(ft^3)}{(lb-mol)(^\circ R)}$

T_{std} = Standard absolute temperature (293 K) or (528 R)

P_{std} = Standard absolute pressure (760 mm Hg) or (29.92 in. Hg)

M_w = Molecular weight of water (18.0 g/g-mol) or (18.0 lb/lb-mol)

Sample Gas Sample Volume:

$$V_{m(std)} = V_m \times Y \times \left(\frac{T_{std}}{T_m} \right) \times \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}} \right]$$

where:

$V_{m(std)}$ = Gas sample volume (dscm) or (dscf)

V_m = Dry gas volume measured at dry gas meter (dcm) or (dcf)

Y = Dry gas meter calibration factor, dimensionless

T_m = Absolute temperature at dry gas meter (K) or (R)

T_{std} = Standard absolute temperature (293 K) or (528 R)

P_{std} = Standard absolute pressure (760 mm Hg) or (29.92 in. Hg)

P_{bar} = Barometric pressure at the sampling site (mm Hg) or (in. Hg)

ΔH = Average pressure differential across the orifice meter (mm H₂O) or (in. H₂O)

Isokinetic Variation:

$$I(\%) = \frac{T_s \times V_{m(std)} \times P_{std}}{T_{std} \times v_s \times 60 \times q \times A_n \times P_s \times (1 - B_{ws})} \times 100\%$$

where:

T_{std} = Standard absolute temperature (293 K) or (528 R)

T_s = Absolute stack temperature (K) or (R)

P_s = Absolute stack pressure (mm Hg) or (in. Hg)

P_{std} = Standard absolute pressure (760 mm Hg) or (29.92 in. Hg)

$V_{m(std)}$ = Gas sample volume (dscm) or (dscf)

B_{ws} = Water vapor fraction in stack gas by volume, dimensionless

v_s = Average stack gas velocity (m/sec) or (ft/sec)

q = Total sampling time (min)

A_n = Cross-sectional area of sample nozzle (m²) or (ft²)

60 = sec/min.

Stack Gas Emission Concentration Corrections:

Numerous stack gas emission results are required to be corrected (or normalized) to a specified oxygen concentration in order to account for dilution effects. The following calculations are used to perform those corrections:

Correction to 7% oxygen:

$$C_{(corr)} = C_{(unc)} \times \frac{21-7}{21-Y}$$

where:

$C_{(corr)}$ = Corrected sample concentration.

$C_{(unc)}$ = Uncorrected sample concentration

Y = Stack gas oxygen concentration (vol %, dry).

Correction to 11% oxygen:

$$C_{(corr)} = C_{(unc)} \times \frac{21-11}{21-Y}$$

where:

$C_{(corr)}$ = Corrected sample concentration.

$C_{(unc)}$ = Uncorrected sample concentration

Y = Stack gas oxygen concentration (vol %, dry).

Particulate Matter:

Particulate emissions are determined in accordance with EPA Method 5 and the calculations presented in 40 CFR 60 Appendix A. The final emission rate and stack gas concentration equations are as follows:

$$E_p (\text{lb} / \text{hr}) = \frac{M_p}{V_{m(\text{std})}} \times Q_{sd} \times \frac{1 \text{ g}}{1000 \text{ mg}} \times \frac{1 \text{ lb}}{453.59 \text{ g}} \times \frac{60 \text{ min}}{\text{hr}}$$

where:

E_p = Stack gas emission rate of particulate matter (lb/hr)

M_p = Mass of particulate matter collected in the M5 sample (mg)

$V_{m(\text{std})}$ = Gas sample volume (dscf)

Q_{sd} = Stack gas volumetric flow rate (dscfm)

$$C_p (\text{gr} / \text{dscf}) = \frac{M_p}{V_{m(\text{std})}} \times \frac{1 \text{ g}}{1000 \text{ mg}} \times \frac{1 \text{ lb}}{453.59 \text{ g}} \times \frac{7000 \text{ gr}}{\text{lb}}$$

where:

C_p = Measured stack gas particulate matter concentration (gr/dscf)

M_p = Mass of particulate matter collected in the M5 sample (mg)

$V_{m(\text{std})}$ = Gas sample volume (dscf)

$$C_p (\text{mg} / \text{dscm}) = \frac{M_p}{V_{m(\text{std})}} \times \frac{35.31 \text{ dscf}}{\text{dscm}}$$

where:

C_p = Measured stack gas particulate matter concentration (mg/dscm)

M_p = Mass of particulate matter collected in the M5 sample (mg)

$V_{m(\text{std})}$ = Gas sample volume (dscf)

Note: Oxygen corrections are made using the formulas given at the beginning of this document.

Hydrogen Chloride and Chlorine:

Stack gas samples are collected in acidic and alkaline impingers to capture gaseous chlorides. The chloride captured in the acidic impingers is assumed to be associated with hydrogen chloride emissions. The chloride captured in the alkaline impingers is assumed to be associated with chlorine emissions. Overall control efficiency for any halogen is calculated by taking the ratio of the total stack gas emission rate for the halogen to the total input of the corresponding halogen.

Note: Example calculations are provided for hydrogen chloride and chlorine.

$$E_{HCl} (lb/hr) = \frac{[Cl^-]_a \times Q_{sd}}{V_{m(std)}} \times \frac{1g}{1000mg} \times \frac{1lb}{453.59g} \times \frac{60min}{hr} \times \frac{36.46lb HCl}{35.45lb Cl}$$

where:

E_{HCl} = Emission rate of hydrogen chloride (lb/hr)

$[Cl^-]_a$ = Chloride in acidic impingers (mg)

Q_{sd} = Stack gas volumetric flow rate (dscfm)

$V_{m(std)}$ = Gas sample volume (dscf)

$$E_{Cl_2} (lb/hr) = \frac{[Cl^-]_b \times Q_{sd}}{V_{m(std)}} \times \frac{1g}{1000mg} \times \frac{1lb}{453.59g} \times \frac{60min}{hr}$$

where:

E_{Cl_2} = Emission rate of chlorine (lb/hr)

$[Cl^-]_b$ = Chloride in alkaline impingers (mg)

Q_{sd} = Stack gas volumetric flow rate (dscfm)

$V_{m(std)}$ = Gas sample volume (dscf)

Total HCl and Cl₂ as HCl Equivalents:

$$C_{HCl(eq)} = \frac{\left([Cl^-]_a + [Cl^-]_b \right) \times \frac{36.46 \text{ mg HCl}}{35.45 \text{ mg Cl}}}{V_{m(std)}} \times \frac{35.31 \text{ ft}^3}{\text{m}^3} \times \frac{1 \text{ kg HCl}}{1 \times 10^6 \text{ mg HCl}} \times \frac{1 \text{ kg - mol HCl}}{36.46 \text{ kg HCl}} \times \frac{24.04 \text{ dscm}}{\text{kg - mol}} \times \left(1 \times 10^6 \text{ ppm} \right)$$

where:

$C_{HCl(eq)}$ = Concentration of HCl and Cl₂ as HCl equivalents (ppmv)

$[Cl^-]_a$ = Chloride in acidic impingers (mg)

$[Cl^-]_b$ = Chloride in alkaline impingers (mg)

$V_{m(std)}$ = Gas sample volume (dscf)

Note: Oxygen corrections are made using the formulas given at the beginning of this document.

Metals:

For each metal, the amount found in each sample component is added, and the total amount is used in all calculations:

$$E_{m(i)} \text{ (lb / hr)} = \frac{\sum M_{m(i)} \times Q_{sd}}{V_{m(std)}} \times \frac{1 \text{ g}}{1 \times 10^6 \text{ } \mu\text{g}} \times \frac{1 \text{ lb}}{453.59 \text{ g}} \times \frac{60 \text{ min}}{\text{hr}}$$

where:

$E_{m(i)}$ = Emission rate of metal i (lb/hr)

$M_{m(i)}$ = Mass of metal i collected in sampling train components (ug)

Q_{sd} = Stack gas volumetric flow rate (dscfm)

$V_{m(std)}$ = Gas sample volume (dscf)

$$C_{m(i)} \text{ (} \mu\text{g / dscm)} = \frac{\sum M_{m(i)}}{V_{m(std)}} \times \frac{35.31 \text{ dscf}}{\text{dscm}}$$

where:

$C_{m(i)}$ = Concentration of metal i (ug/dscm)

$M_{m(i)}$ = Mass of metal i collected in sampling train components (ug)

$V_{m(std)}$ = Gas sample volume (dscf)

Note: Oxygen corrections are made using the formulas given at the beginning of this document.

Volatile Organic Compound Emission Rates (Using Sorbent Tubes):

For each volatile organic compound, the amounts found in all sorbent tube sets used in a run are added together. The amount of gas sampled through all analyzed tube sets used in the run is also added together. Emission rates are calculated as follows:

$$E_{v(i)} (g / sec) = \frac{\sum M_{v(i)}}{\sum V_{v(std)}} \times \frac{1000 \text{ liters}}{m^3} \times Q_{sd} \times \frac{1 m^3}{35.31 ft^3} \times \frac{1 g}{1 \times 10^9 ng} \times \frac{1 \text{ min}}{60 \text{ sec}}$$

where:

$E_{v(i)}$ = Emission rate of volatile organic compound i (g/sec)

$M_{v(i)}$ = Mass of volatile organic compound i found in VOST train components (ng)

$V_{v(std)}$ = Sorbent tube set gas sample volume (liters, dry standard)

Q_{sd} = Stack gas volumetric flow rate (dscfm)

Emission Rates of Semi-Volatile Organic, PAH, Organochlorine Pesticide, PCB, and Carbonyl Compounds:

For each compound, the total amount found in the entire sample is divided by the gas sample volume, and multiplied by the stack gas volumetric flow rate to yield the compound emission rate. Note that the example given is for semivolatile compounds, as follows:

$$E_{sv(i)} (g / sec) = \frac{\sum M_{sv(i)}}{V_{m(std)}} \times Q_{sd} \times \frac{1 g}{1 \times 10^6 \text{ } \mu\text{g}} \times \frac{1 \text{ min}}{60 \text{ sec}}$$

where:

$E_{sv(i)}$ = Emission rate of semi-volatile compound i (g/sec)

$M_{sv(i)}$ = Mass of semi-volatile organic compound i found on sampling train components (μg)

$V_{m(std)}$ = Gas sample volume (dscf)

Q_{sd} = Stack gas volumetric flow rate (dscfm)

Destruction and Removal Efficiency:

Destruction and removal efficiency (DRE) for POHCs is calculated using the formula given in 40 CFR 264.343(a)(1).

$$DRE (\%) = \frac{(W_{in} - W_{out})}{W_{in}} \times 100\%$$

where:

W_{in} = Mass feed rate of POHC into the incinerator

W_{out} = Mass emission rate of POHC present in the exhaust emissions prior to release to the atmosphere

For preservation of significant figures, the equation is transformed to the following:

$$DRE (\%) = \left(1 - \frac{W_{out}}{W_{in}} \right) \times 100\%$$

PCDD/PCDF Emission Rate and Stack Gas Concentration:

Total emissions of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) are calculated by summing the emissions of the tetra- through octa- chlorinated PCDDs and PCDFs. Emission rates of the PCDDs and PCDFs are calculated by summing the total mass of the tetra- through octa- chlorinated PCDDs and PCDFs found in the entire stack gas sample, dividing by the gas sample volume, and multiplying by the stack gas flow rate. Stack gas PCDD/PCDF concentration is calculated by dividing the total mass of PCDDs and PCDFs in the stack gas sample by the gas sample volume. Values are corrected to an equivalent 7% oxygen, dry basis. Calculations used for PCDD/PCDF determinations are as follows:

$$E_{DF} (g / sec) = E_D + E_F$$

where:

E_{DF} = Total PCDD/PCDF emission rate (g/sec)

E_D = Total tetra- through octa- chlorinated PCDD emission rate (g/sec)

E_F = Total tetra- through octa- chlorinated PCDF emission rate (g/sec)

$$E_D \text{ (g/sec)} = \frac{\sum_{i=4}^8 M_{D(i)}}{V_{m(std)}} \times Q_{sd} \times \frac{1 \text{ g}}{1 \times 10^9 \text{ ng}} \times \frac{1 \text{ min}}{60 \text{ sec}}$$

$$E_F \text{ (g/sec)} = \frac{\sum_{i=4}^8 M_{F(i)}}{V_{m(std)}} \times Q_{sd} \times \frac{1 \text{ g}}{1 \times 10^9 \text{ ng}} \times \frac{1 \text{ min}}{60 \text{ sec}}$$

where:

$M_{D(i)}$ = Mass of CDD on sample in congener group i (ng)

$M_{F(i)}$ = Mass of CDF on sample in congener group i (ng)

i = Number of chlorine atoms in congener group (i.e., $i = 4$ represents tetra- CDD/CDF)

$V_{m(std)}$ = Gas sample volume (dscf)

Q_{sd} = Stack gas volumetric flow rate (dscfm)

$$C_{DF(corr)} = C_{D(corr)} + C_{F(corr)}$$

where:

$C_{DF(corr)}$ = Total stack gas concentration of PCDDs and PCDFs (ng/dscm, corrected to 7% O₂, dry)

$C_{D(corr)}$ = Stack gas concentration of tetra- through octa- chlorinated PCDDs (ng/dscm, corrected to 7% O₂, dry)

$C_{F(corr)}$ = Stack gas concentration of tetra- through octa- chlorinated PCDFs (ng/dscm, corrected to 7% O₂, dry)

$$C_{D(corr)} = \frac{\sum_{i=4}^8 M_{D(i)}}{V_{m(std)}} \times \frac{35.31 \text{ ft}^3}{\text{m}^3} \times \frac{14}{21-Y}$$

$$C_{F(corr)} = \frac{\sum_{i=4}^8 M_{F(i)}}{V_{m(std)}} \times \frac{35.31 \text{ ft}^3}{\text{m}^3} \times \frac{14}{21-Y}$$

where:

$M_{D(i)}$ = Mass of CDD on sample in congener group i (ng)

$M_{F(i)}$ = Mass of CDF on sample in congener group i (ng)

i = Number of chlorine atoms in congener group (i.e., $i = 4$ represents tetra- CDD/CDF)

$V_{m(std)}$ = Gas sample volume (dscf)

Y = Stack gas oxygen concentration (vol %, dry).

ATTACHMENT E
PROCESS ENGINEERING INFORMATION

SHUTDOWN PROCEDURES

NORMAL SHUTDOWN PROCEDURES

The following procedure is to be followed to cool down the reactivation unit to ambient temperature.

STEP NUMBER	TEMPERATURE SCHEDULE (DEGREES FAHRENHEIT)	HOLDING TIME HOURS	TOTAL HOURS
1.	Shut off feed to furnace. Let carbon clear from all hearths before starting to cool down the hearths and afterburner. The furnace retention time for carbon is 48 minutes. NOTE: Keep all APC equipment running as normal until 48 minutes after the furnace feed has been shut off.	N/A	N/A
2.	Begin to cool down the hearths and afterburner at a rate of 100°F per hour to 1400°F. DO NOT HURRY THE PROCESS AS REFRACTORY DAMAGE CAN BE DONE IF THE TEMPERATURE IS DECREASED TOO FAST.	100°F/Hour	4.00
3.	Decrease furnace and afterburner temperatures 50°F per hour to ambient temperature.	50°F/Hour	~28.00
TOTAL COOLING TIME		ESTIMATED	32.00

EMERGENCY SHUTDOWN

The following procedures are followed in the event it is necessary to shutdown the reactivation unit in an emergency situation.

LOSS OF ELECTRICAL POWER

If the facility loses electrical power, the following critical equipment would not function:

1. Induced Draft Fan
2. Combustion Fans
3. Shaft Cooling Fan
4. Scrubber Water Recycle Pump
5. Continuous Emission Monitoring System
6. Plant Monitoring Equipment
7. Plant Compressed Air Supply.

The first priority is to start the auxiliary gas-powered shaft cooling fan. If the outlet temperature increases to over 500°F, damage to the shaft and/or arms can occur from warpage due to the high temperature. The fan must be started no more than 5 minutes after the loss of electrical power. Plant management is to be notified as soon as possible.

If the outage continues for any length of time, the afterburner will start to lose temperature. It is imperative, once power is regained, that the temperature in the afterburner is brought up to 1800°F prior to re-establishing spent carbon feed to the furnace. Procedures for normal start-up are to be followed for unit warmup. The warmup procedure is not to be hurried.

LOSS OF CITY WATER SUPPLY

The loss of the of the city water supply to the facility can cause extensive damage to the air pollution control equipment. This city water is also used to make up the scrubber recycle liquor and if operations persist without correcting the problem, the scrubber loop will eventually run dry resulting in increased particulate matter and acid gas emissions.

If the scrubber system starts to lose water, shut down the feed to the furnace. Proceed to shutdown the burners/combustion fans and the induced draft fan. Seal the furnace and attempt to rectify the problem.

If the facility loses city water, the booster pump should be checked immediately. The pump is located by the back-flow prevention valves near the main gate. If the pump is down, bypass the pump and use city water pressure for plant purposes. Notify plant management of the problem as soon as possible.

EXPLOSION IN FURNACE OR AFTERBURNER

If there is an explosion in the furnace or the afterburner, immediately shut down all equipment surrounding the furnace. This includes all burners, fans, and the dewatering screw. The natural gas is to be manually turned off immediately. A wrench is provided on the fence (in a red case) to turn the gas off outside the fence on the southwest side of the plant. Notify the emergency coordinator as soon as possible.

DRAWINGS AND SPECIFICATIONS

Specifications		
Company	Document Reference	Description
Combustion and Control Engineers	501-58 (2 pages)	Hot air blower
North American Mfg. Co.	501-1 (7 pages)	Hearth burners
North American Mfg. Co.	501-23 (4 pages)	Afterburner burners
Altech Systems Corp.	ASC303-03 (1 page)	CEMS diagram
Thermo Environmental Instruments Inc.	Model 48 CO Analyzer (4 pages)	CO analyzer
Ametek	Thermox (3 pages)	Oxygen analyzer

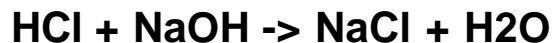
ATTACHMENT F
JUSTIFICATION FOR COMPLIANCE WITH CHLORINE FEED RATE
LIMITS BY USING TDS RESTRICTIONS

USFilter Westates Rationale for Complying with Chlorine Feed Rate

The purpose of this document is to demonstrate how compliance with the facility's existing wastewater discharge limit of delta 1400 mg/L for the rise in total dissolved solids (TDS) can be used to ensure compliance with the facility's proposed 12-hour rolling average limit for the feed rate of total chlorine and chloride.

During the RF-2 compliance tests for semi volatile metals (cadmium and lead), low volatility metals (arsenic, beryllium and chromium), hydrogen chloride and chlorine gas, US Filter/Westates Carbon plans to process spent carbon feed with a total chlorine and chloride loading of 5% by weight dry basis to establish a 12-hour rolling average limit of 78 lbs/hr for the feed rate of total chlorine and chloride for compliance with applicable requirements of 40 CFR 63, Subpart EEE.

Sodium hydroxide (NaOH) is added to the recirculated scrubber solutions for the venturi and packed bed scrubbers to neutralize and control the emissions of hydrogen chloride (HCl) from the carbon reactivation furnace. HCl is readily dissolved in water, where chloride ions react with sodium ions to form salt.



The recirculated scrubber solutions for the venturi and packed bed scrubbers of RF-2 are maintained at a pH of 5 to make sure a sufficient amount of sodium hydroxide is continuously added to the scrubber water for reaction with the hydrogen chloride. At this pH level, there is a negligible excess of HCl in the scrubber solutions (3.03E-6 pounds of HCl per gallon).

The amount of sodium chloride formed is dependent upon the chloride content and the feed rate of the waste material. At a chloride concentration of five percent by weight dry basis, the maximum wet feed rate of 2760 lbs/hr (1560 dry pounds per hour) equates to a total chlorine loading of $1560 \times 0.05 = 78$ lbs/hr. Multiplication by the ratio of molecular weights for salt (NaCl) and chlorine (Cl) yields $78 \text{ lbs/hr Cl} \times (23 + 35.45) / 35.45 = 128.6$ lbs/hr NaCl. Maintenance of this chlorine loading for twelve hours would result in a total 12-hour salt production of $12 \times 128.6 = 1,543$ lbs/hr NaCl.

The actual rise in dissolved solids is dependent upon the wastewater discharge rate in addition to the total amount of salt formed. Westates Carbon has an existing 144,000 gallon per day influent restriction for "city" water usage. Approximately 20,000 gallons per day, or about 14% of the influent water consumed by the plant is lost to evaporation in the scrubber system. Thus, the maximum wastewater discharge from the facility is approximately 124,000 gallons per day.

Discharge of the 1,543 lbs/hr of NaCl produced from 12 hours of operation at the proposed feed rate limit of 78 lbs/hr for total chlorine and chloride over a 24-hour period in the 124,000 gallons of wastewater would result as a rise in TDS of $1,543 \text{ lbs/hr} \times 453.6 \text{ g/lb} \times 1000 \text{ mg/g} / (124,000 \text{ gallons} \times 3.785 \text{ liters/gallon}) = 1491 \text{ mg/L}$. Note that this delta TDS level is slightly in excess of the 24-hour maximum delta TDS limit. This confirms that compliance with the 1400 mg/L delta TDS limit ensures that the 12-hour rolling average limit of 78 lbs/hr for the feed rate of total chlorine and chloride will not be exceeded.

The above presentation is oversimplified and the calculations conservative. Several additional factors contribute to an increase in the delta TDS level of the facility's wastewater discharge without a corresponding increase in the total feed rate of chlorine.

Evaporation of approximately 20,000 gallons per day of water in the scrubber system concentrates dissolved solids in the influent water by a factor of 16%. For "city" water with a TDS of 500 mg/L this represents a delta TDS of 80 mg/L, whereas starting with a "city" water TDS of 1000 mg/L, evaporation increases the TDS in the scrubber blow down by 160 mg/L.

Absorption of carbon dioxide and sulfur oxides in the scrubber water, even when processing non-chlorinated feed materials, results in an acidic scrubber solution which requires the addition of sodium hydroxide for neutralization. This further increases the delta TDS into the range of 200 to 500 mg/L.

A portion of the wastes processed by the facility contain halogens other than chlorine. These materials, such as fluorine, bromine and iodine, are typically emitted as acid gases from RF-2 and absorbed in the scrubber solution. Similar to hydrogen chloride, they also react with the sodium hydroxide to form salts and increase the TDS in the scrubber blow down. Maintaining compliance with the delta 1400 TDS 24-hour discharge limit while feeding wastes that contain other halogens reduces the total amount of chlorine and chlorides that can be fed to RF-2.

Other discharges from the facility, such as blow down from the boilers and process water cooling tower, although quite high in TDS are so small in quantity that they contribute less than one percent of the total delta TDS discharged by the facility and can be ignored for the purposes of discussion herein.

Thus, compliance with the facility's wastewater discharge limit of delta 1400 mg/L for the rise in total dissolved solids (TDS) ensures compliance with the facility's proposed hazardous waste 12-hour rolling average feed rate limitation of 78 lbs/hr for total chlorine and chloride.

ATTACHMENT G
DATA IN LIEU OF TESTING