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	Polyaromatic 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 <u>carbon regeneration unit</u>, 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:

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2.0 FEED STREAM DESCRIPTION
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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 in 40 CFR 63 Subpart EEE):
    - 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 (Cl_2) 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 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 Cl_2 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 an EPA Method 5 train with a smooth filter
- Stack gas CO and O_2 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 it's 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 it's 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 facility’s RCRA Waste Analysis Plan. 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. 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 ¾” 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. Feed characterization and analysis procedures and frequencies are described in the facility's RCRA Waste Analysis Plan.

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 conditions which may result in stopping waste feeds (referred to as "administrative stop-feeds" or "safety stop-feeds") 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.

At the request of EPA, WCAI has developed a Startup Shutdown and Malfunction Plan (SSM Plan), which has been submitted as a stand-alone document. The SSM Plan describes procedures for routine and non-routine startup and shutdown of the RF system, and it describes potential malfunctions and procedures to minimize emissions during a malfunction event. The provisions of the SSM Plan will apply during the performance testing, and are incorporated into this document by reference.

Operating conditions specified in Tables 4-2, 7-1 and 7-2 apply any time there is waste in the system, whether or not waste is being fed, except during startup and shutdown.

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 <u>carbon regeneration unit</u>, 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 (Cl_2) 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 Cl_2 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 an EPA Method 5 train with a smooth filter
- Stack gas CO and O_2 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 it's 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 slurrying 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 an aqueous solution of lead nitrate, while chromium will be added as an aqueous solution of chromium (III) nitrate. 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 fluctuation 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 Cl_2

The stack gas will be sampled for particulate, HCl, and Cl_2 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 Cl_2. 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-C_1 - C_7 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 O_2 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 (including spiking). 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 6 to 8 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. EPA will be notified if any of the RCRA interim status emission standards are exceeded during the preliminary testing.

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. Westates will inform EPA in writing if there are proposed changes to operating conditions for the CPT, based on information collected during the preliminary testing.

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.

Westates will inform EPA in writing if there are proposed changes to operating conditions for the CPT, based on information collected during the preliminary testing.

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 Cl_2 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 Cl_2 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/Cl_2 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.

$$HCl + NaOH \rightarrow NaCl + H_2O$$

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 it’s 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 aqueous solutions of metal salts. Lead will be fed as an aqueous solution of lead nitrate. Chromium will be fed as an aqueous solution of chromium (III) nitrate.

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. For all parameters having a limit based on rolling averages, the test report will contain both 1-minute average data and rolling average data.

Process Parameter	Units
Spent carbon feed rate	lb/h
Afterburner temperature	^{o}F
Hearth temperature (each hearth)	^{o}F
Venturi scrubber pressure differential	in. w.c.
Quench/Venturi scrubber liquid flow rate	gpm

Process Parameter	Units
Packed bed scrubber pH	pH
Packed bed scrubber liquid flow rate	gpm
Packed bed scrubber pressure differential	in. w.c.
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, of 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.

Table 5-1 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-lter 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 5-1 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 5-1 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 organcs 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	EPA Method 5 train with amooth filter media	As required	Collect particle size distribution samples on smooth filter media	Scanning electron microscope
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 5-1 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 <u>Test Methods for Evaluating Solid Waste</u>, Third Edition, November 1986, and Updates.
"EPA Method" refers to <u>New Source Performance Standards</u>, 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 5-3. 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 5-3. 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) & solvent exchanged to hexane or isooctane	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 5-3. 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 5-3. 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	Scanning electron microscope
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 5-3. 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 A-2. Spiking Material Sampling Procedure

Sample name: POHC spiking material
Metals spiking material

Sampler: Process sampling team

Locations: Sample tap on spiking system feed line

Equipment: 250-mL amber glass bottles with Teflon-lined lids

Gloves, eye protection, respiratory protection if needed.

Frequency: Once during test when used

Procedure Summary: Metals Spiking Material Sample Collection: The sample tap is purged by allowing a small amount of liquid to flow into the waste container. A 250 mL 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.

POHC Spiking Material: The sampling tap is purged by allowing a small amount of liquid to flow into the waste container. One 250-mL glass bottle is then filled from the sample tap. The sample bottle is labeled with the sample name, date, run number, and time. The sample number and time collected are recorded on the sample collection sheet.

All samples: 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.

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: 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.

The sample in the 4-liter bottle is subsequently transferred to three 1-liter bottles, as follows:

40 mL VOA vials – Volatile organics
1L – Semivolatile organics
1L – Metals
1L – Archive.

The 4-liter bottle and any excess samples are discarded as appropriate for the material.

Record the sampling time on sample collection sheet and approximate volume of final samples.

The sample bottles and vials are labeled with sample number, date, sample name, and test-run number.

Sample coordinator accepts custody of samples and records numbers and collection data in field logbook.

Samples are placed on ice in shipping container that is stored in the sample holding area separate from the container supply area.

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-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 (≥50°C) soapy (Alconox or equivalent) water, rinsing it three times each with hot water, distilled/deionized water, and drying in an oven for 2 hours at 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, acetone, 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 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°F ± 25°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 ± 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 acetone/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

three times with acetone/methylene chloride (1:1 v/v). The condensate knockout impinger (Impinger 1) is rinsed with acetone/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 acetone/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.

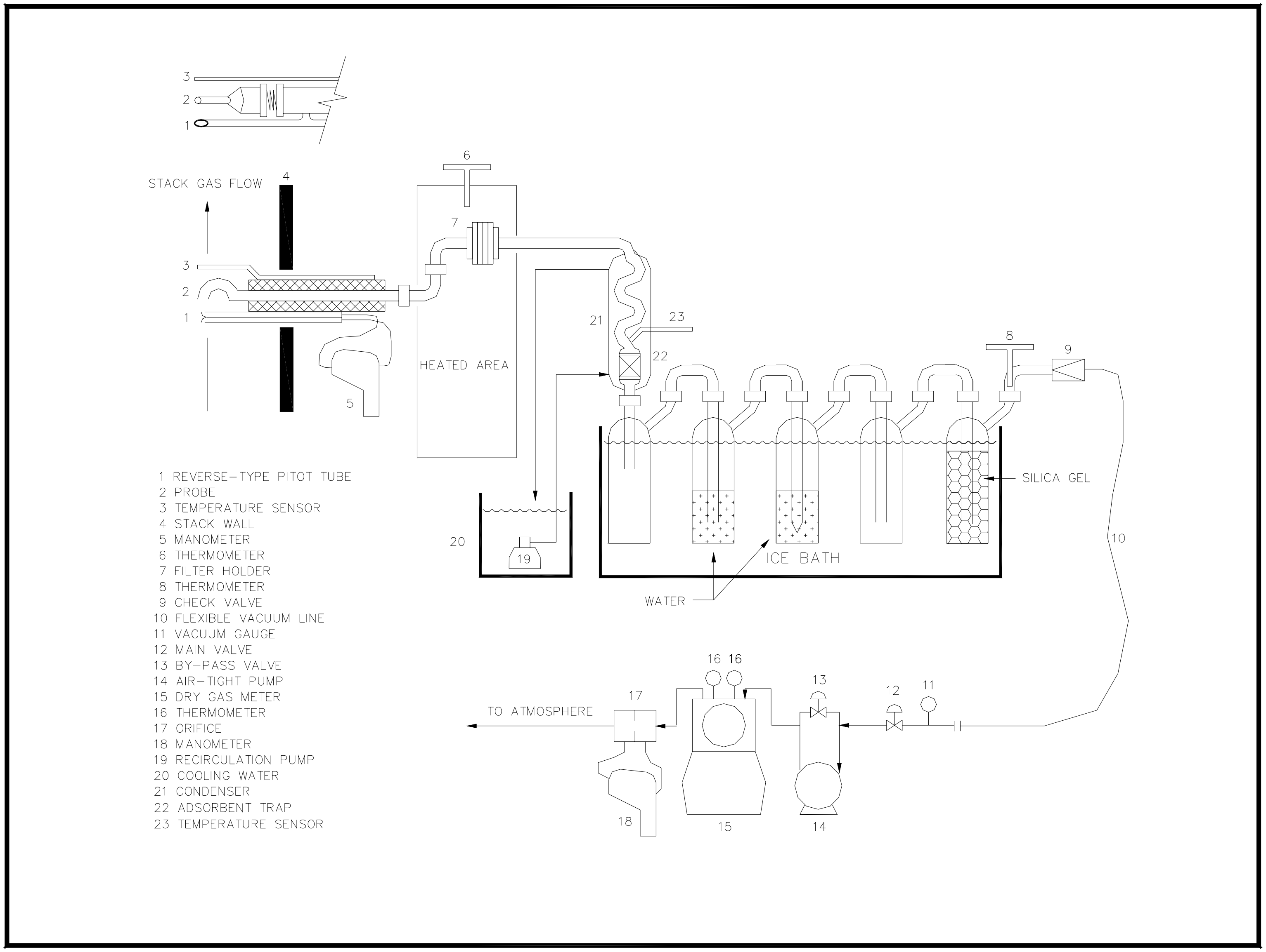

Figure 1. Method 0010 Sample Train for Semivolatile Organics

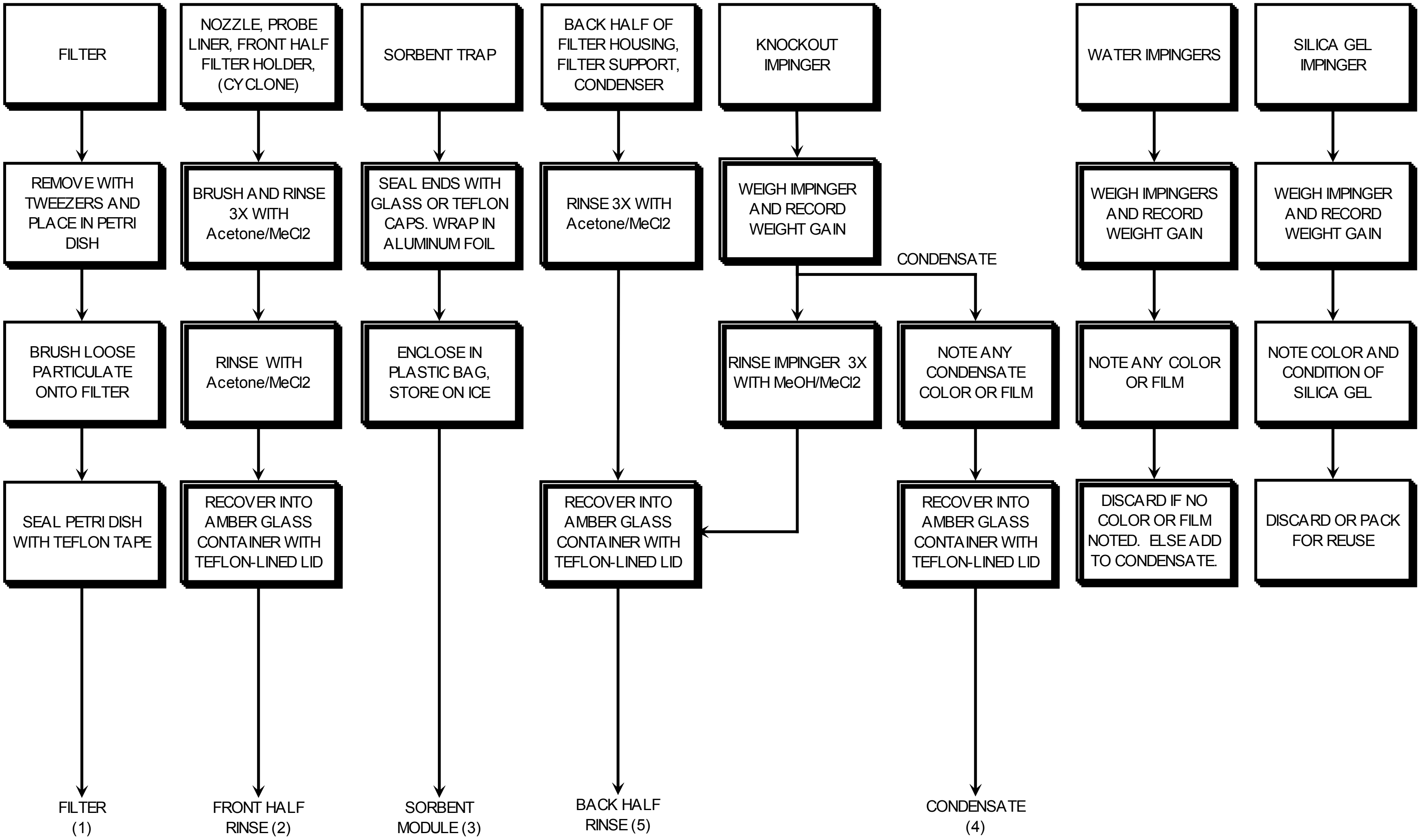

Figure 2. Method 0010 Semivolatile Organics Sample Recovery

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 (≥50°C) soapy (Alconox or equivalent) water, rinsing it three times each with hot water, distilled/deionized water, and drying in an oven for 2 hours at 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, acetone, 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 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°F ± 25°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 ± 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 acetone/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

three times with acetone/methylene chloride (1:1 v/v). The condensate knockout impinger (Impinger 1) is rinsed with acetone/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 acetone/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.

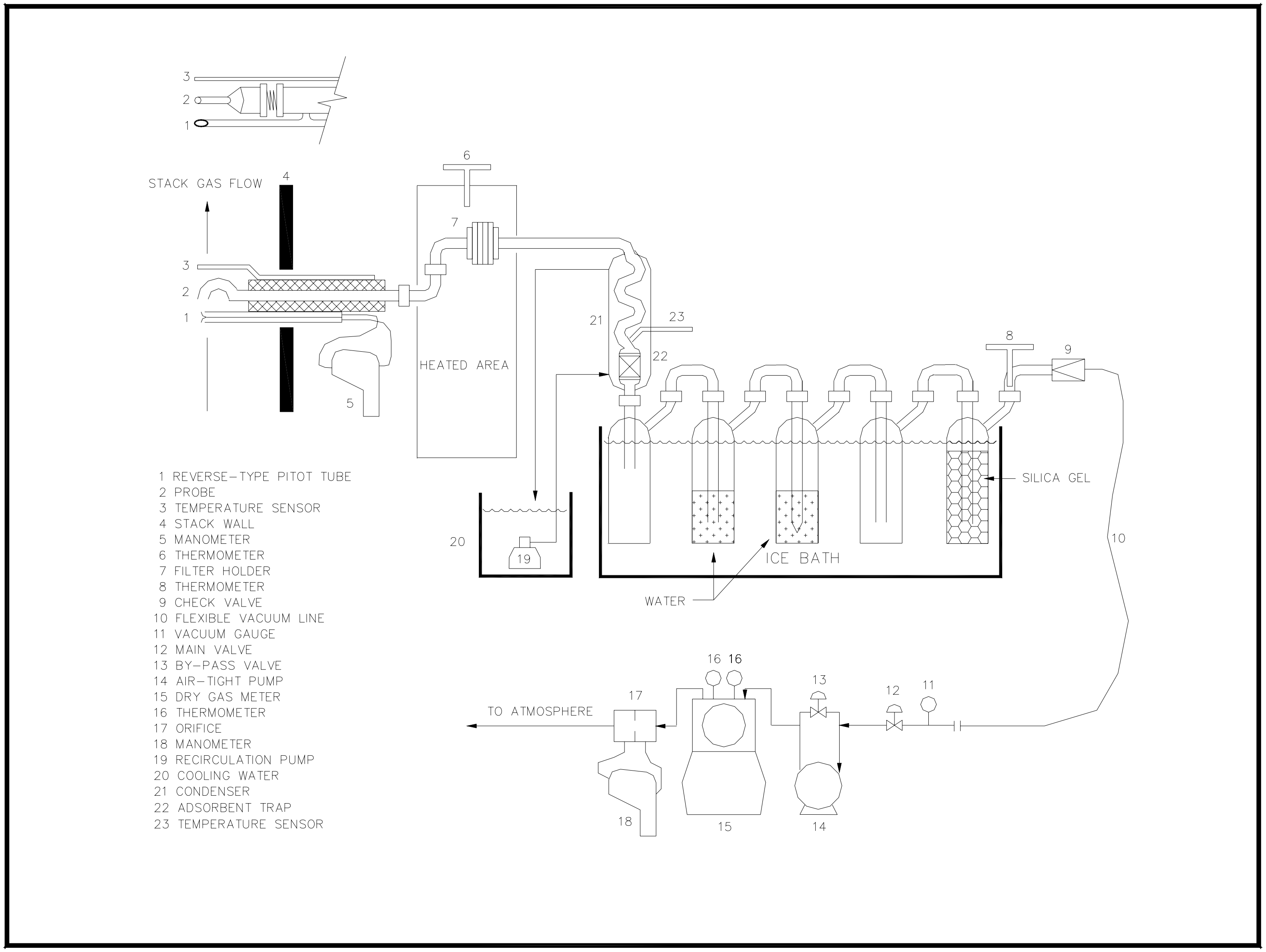

Figure 1. Method 0010 Sample Train for Semivolatile Organics

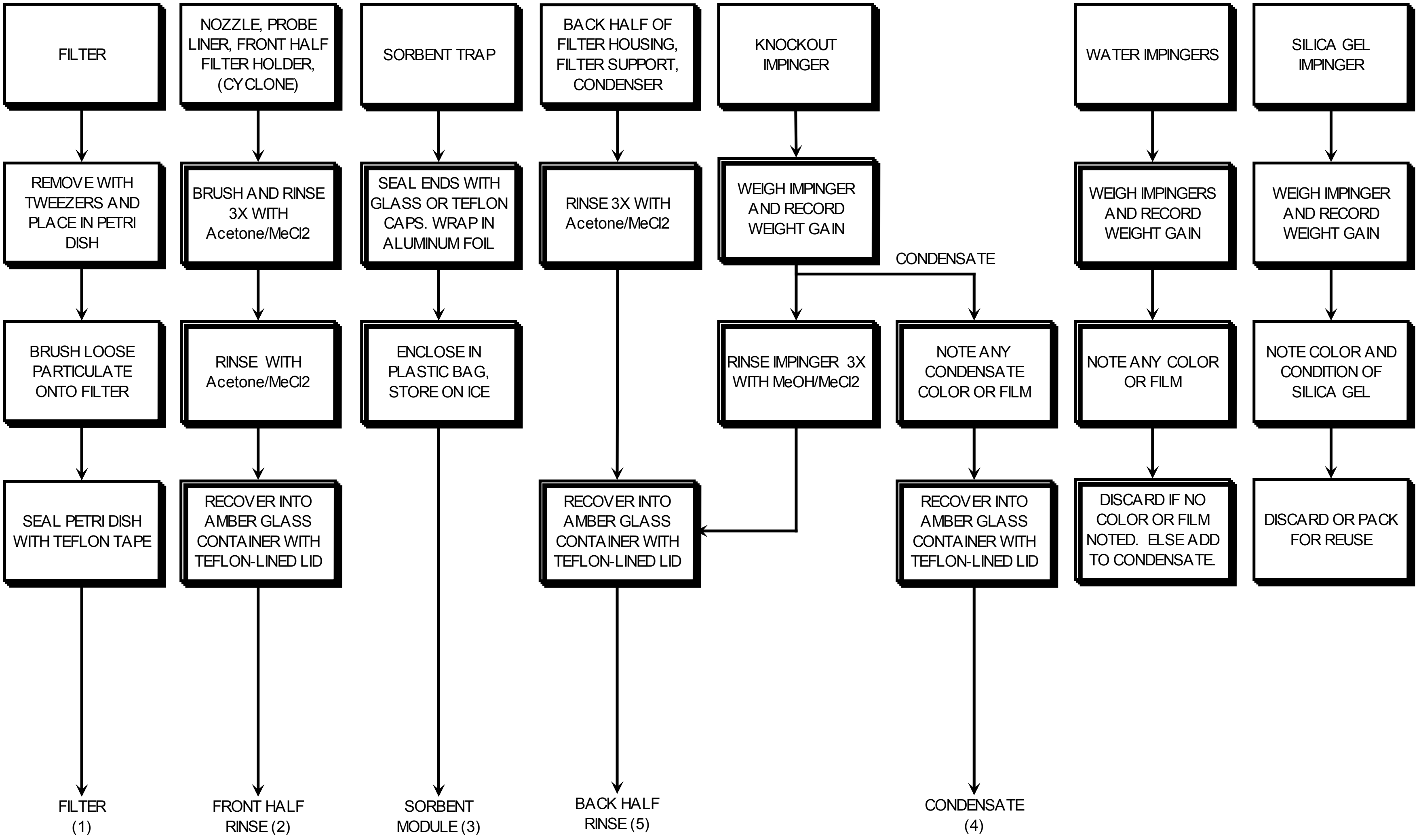

Figure 2. Method 0010 Semivolatile Organics Sample Recovery

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

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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 (≥50°C) soapy (Alconox or equivalent) water, rinsing it three times each with hot water, distilled/deionized water, and drying in an oven for 2 hours at 400 °C. After cooling, the glassware is rinsed 3 times each with pesticide grade methylene chloride and pesticide grade toluene. When glassware is dry, all openings are covered with cleaned aluminum foil or clean glass plugs until sample train assembly.

The 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, acetone, 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.)

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 emisson 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°F ± 25°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 ± 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 acetone/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 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.

- 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 acetone/methylene chloride (1:1 v/v). The condensate knockout impinger (Impinger 1) is rinsed with acetone/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 acetone/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.

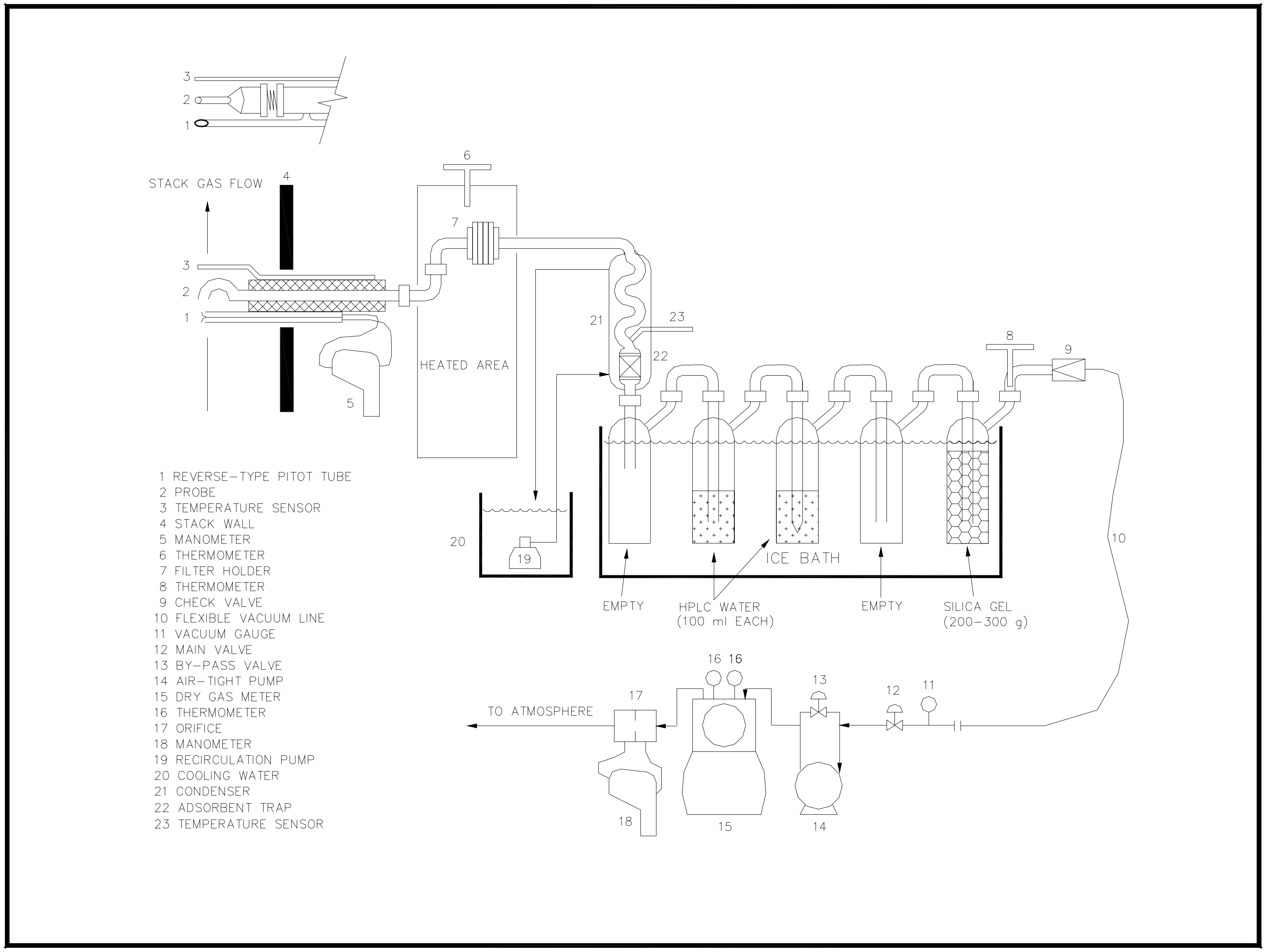

Figure 1. Method 0010 Sample Train for Semivolatile and Nonvolatile Organics

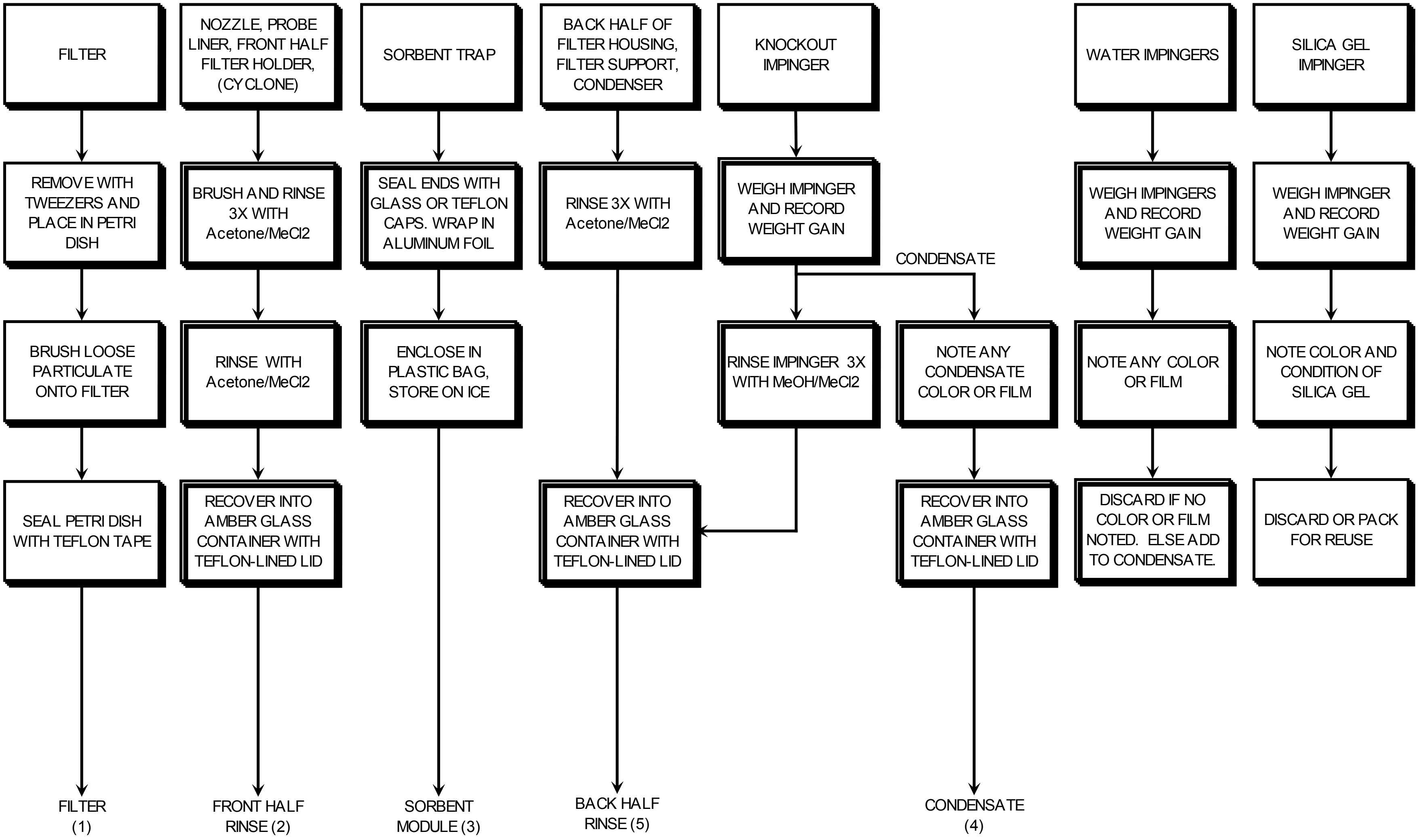

Figure 2. Method 0010 Semivolatile and Nonvolatile Organics Sample Recovery

Table A-15. Particle Size Distribution Sampling Procedure Using Method 5 and Scanning Electron Microscope Analysis

Sample name: Stack gas particle size distribution (PSD)

Sampler: Stack sampling team

Locations: System exhaust duct

Equipment: EPA Method 5 sampling train; amber glass sample jars with Teflon-lined lids; petri dish with tared particulate filter; balance; glass graduated cylinder.

Frequency: Continuous during a test run; three runs to complete test. A maximum of 10 dry standard cubic feet of sample will be collected in order to avoid overloading the filter and interfering with the scanning electron microscope (SEM) imaging process for particle size distribution determination. Typically, only 24 to 30 minutes total sampling time is required.

Procedures: Stack gases will be sampled isokinetically to collect particulate matter on a filter. The particulate weight will be determined gravimetrically and the particle size distribution will be determined by scanning electron microscope.

Sampling point locations are determined in accordance with EPA Method 1. An initial traverse is made with a pitot tube at each sample point following EPA Method 2 to establish stack gas velocity profile, temperature, and flow rate, and to check for cyclonic flow (cyclonic flow will be checked only on the first day of testing). EPA Method 3, extended for the inclusion of hydrogen in the system exhaust, will be used to determine system exhaust dry molecular weight. EPA Method 4 will be followed to determine the gas moisture content. EPA Method 5 procedures are followed for pretest and post-test leak checks, isokinetic sampling rate, filter changeouts (if needed), and data recording.

The Method 5 train utilizes a heated probe and particulate filter, two water-containing impingers, an empty impinger to catch any liquid carryover from the water-containing impingers, and a silica gel impinger. All four impingers are placed in an ice bath.

Field assembly of the sampling train will take place in a clean area. Train components will be handled so that exposure to ambient conditions will be minimized.

A clean and inspected filter will be placed in the filter holder. Impingers 1 and 2 will each contain 100 ml of water. Impinger 3 is initially empty and will serve to collect any liquid carryover from impingers 1 and 2. Impinger 4 will contain 200 to 300 g of indicating silica gel.

During sampling operations, gas is pulled through the probe nozzle and a borosilicate or quartz glass-lined and heated probe. Particulate matter is removed from the gas stream by means of a filter, housed in a glass filter holder maintained at 248 ± 25 °F. A chilled impinger train is used to remove water from the gas. A dry gas meter is used to measure the sample gas flow.

The sample train will be operated according to EPA Method 5 procedures, and an isokinetic sampling rate will be maintained within ±10% of 100%.

After sampling, the probe will be removed from the duct and the nozzle will be covered. External particulate matter will be wiped off the probe. It will then be disconnected from the train and both ends capped. The probe and impinger assembly will be transported to the sample recovery area. The sampling train cleanup and recovery scheme is briefly described as follows:

Table A-15. Particle Size Distribution Sampling Procedure Using Method 5 and Scanning Electron Microscope Analysis
(continued)

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

Impinger Liquid -- The liquid contents of impingers 1, 2, and 3 are measured to the nearest milliliter or weighed to the nearest 0.5 g for determination of system exhaust moisture content. Any color or film observed is noted on the sample recovery sheet. The liquid is then discarded.

Silica Gel -- The silica gel contents of impinger 4 are weighed to the nearest 0.5 g. The color and condition of the silica gel is noted on the sample recovery sheet.

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

For particle size distribution determination, the particulate matter will be collected on a polycarbonate or acetate filter. This filter is a slight variation from the glass fiber filter specified in Method 5, however it will significantly enhance the quality of the SEM analysis by improving the smoothness of the background filter.

References: EPA Methods 1, 2, 3, 4, and 5, Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.

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

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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 according to SW-846 Method 5035. An aliquot of the solution is added to reagent water in a purge tube. Surrogates and internal standards are added to the solution then processed through purge and trap procedures and analyzed by GC/MS according to SW-846 Methods 5030 and 8260

Aqueous samples generally do not require methanol dispersion. Surrogates and internal standards will be added and 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-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:

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 though 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 acetone and methylene chloride adding the rinses to the same amber glass jar. Store this portion at 4^{o}C until the front half rinse extraction is completed.

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 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 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.

The combined filter extract, front half rinse extract, and rinses are then concentrated using a Kuderna-Danish concentrator. Pour the extracts to be condensed though 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^{o}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.

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 grater 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 condentsate 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-labled 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 mehtylene chloride extraction. Then make the solution basic (pH > 11 but < 14) and perform another mehtylene 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

	extraction is complete, the methylene chloride extract is poured though 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-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:

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 though 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 acetone 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.

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 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 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.

The combined filter extract, front half rinse extract, and rinses are then concentrated using a Kuderna-Danish concentrator. Pour the extracts to be condensed though 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.

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 grater 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 condentsate 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-labled 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 mehtylene chloride extraction. Then make the solution basic (pH > 11 but < 14) and perform another mehtylene 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 extraction is complete, the methylene chloride extract is poured though 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-24. Analysis of Particle Size Distribution from M5 Filter Samples Using SEM

Matrices:	Particulate filter

Procedures: During the test, stack gas will be sampled isokinetically to collect particulate matter on a 0.2μ polycarbonate filter according to the procedures given in EPA Method 5. A maximum of 20 ft^3 will be sampled since SEM requires a small amount of particulate on the filter to avoid a layering effect. The M5 particulate filter will be oven dried at 105°C (220°F) for 2 to 3 hours. After drying, the filter is subjected to scanning electron microscopy to determine the particle size distribution.

A primary electron beam is focused to a diameter of 20 to 60 Å and is magnetically scanned over the area of the specimen under investigation. Collision of the primary electron beam with the surface of the specimen produces low energy secondary electrons and back-scattered primary electrons. These are drawn to a positively charged scintillator, where the energy of the back-scattered and secondary electrons is converted to photons. The photon intensity is converted to an electrical signal whose output is proportional to the electron intensity. The photomultiplier signal is suitably amplified and used to modulate the intensity of a cathode ray tube (CRT). The CRT and the magnetic scanning coils are driven by the same scan generator in order to produce the image of the specimen on the screen of the CRT. Magnification is provided by varying the length of scan of the primary electron beam across the specimen while holding the scan length on the CRT constant. The procedure for magnification calibration is performed by varying the accelerating voltage from 2 kV to 20 kV at various magnifications and working distances.

The particle size distribution will be determined at the appropriate magnification. At least 100 particles will be counted for the range containing the most particles. The following "cuts" will be reported as the number of particles in each range:

Diameter Range
≤0.2 - 0.5 μm
>0.5 - 0.9 μm
>0.9 - 1.3 μm
>1.3 - 2.7 μm
>2.7 - 4.5 μm
>4.5 - 6.5 μm
>6.5 - 9.7 μm
>9.7 - 15 μm
>15 μ m

Any oblong shaped particles that fall on the borderline of these ranges will be counted as the smaller particle.

As stated above, the electron microscope image will be optically examined and the number of particles in each size range will be reported. In order to convert this "number distribution" into a "mass distribution" as needed for input to the risk assessment modeling, a unit density will be assigned to each particle and the resulting mass of material in each particle size range will be calculated.