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**Plan**

# IWTU System Performance Test Plan

Idaho Cleanup Project

CH2M ◆ WG Idaho, LLC is the Idaho Cleanup Project contractor for the U.S. Department of Energy

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# ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| ACFM | actual cubic feet per minute |
| AWFCO | automatic waste feed cutoff |
| CEM | continuous emission monitor |
| CEMS | continuous emission monitoring system |
| cfm | cubic feet per minute |
| CFR | Code of Federal Regulations |
| CO | carbon monoxide |
| $CO_2$ | carbon dioxide |
| CRO | control room operator |
| CRR | Carbon Reduction Reformer |
| CWI | CH2M-WG Idaho, LLC |
| DEQ | Idaho Department of Environmental Quality |
| DMR | Denitration Mineralization Reformer |
| DOE | U.S. Department of Energy |
| dscf | dry standard cubic foot |
| dscm | dry standard cubic meter |
| EDF | Engineering Design File |
| EL | emissions limit |
| EPA | U.S. Environmental Protection Agency |
| ft/sec | feet per second |
| GC | gas chromatograph |
| gpm | gallons per minute |
| gr/dscf | grains per dry standard cubic foot |
| $H_2$ | hydrogen |
| HEPA | high-efficiency particulate air (filter) |

| | |
|---|---|
| HVAC | heating, ventilating, and air conditioning |
| HWMA | Hazardous Waste Management Act |
| ID | internal diameter or identification (depending on context) |
| INL | Idaho National Laboratory |
| in. w.c. | inches of water column |
| INTEC | Idaho Nuclear Technology and Engineering Center |
| IWTU | Integrated Waste Treatment Unit |
| MACT | maximum achievable control technology |
| MS | mass spectrometer |
| ηg | nanogram |
| NFM | nuclear facility manager |
| $NO_x$ | nitrogen oxides |
| NWCF | New Waste Calcining Facility |
| $O_2$ | oxygen |
| OGC | off-gas cooler |
| OGF | off-gas filter |
| PAH | polynuclear aromatic hydrocarbon |
| PCDD/PCDF | polychlorinated dibenzo-p-dioxins and polychlorinated dibenzo furans |
| PGF | process gas filter |
| PLN | plan |
| ppmv | parts per million by volume |
| ppmw | parts per million by weight |
| PTC | Permit to Construct |
| QA | quality assurance |
| QAPjP | Quality Assurance Project Plan |
| QC | quality control |

| | |
|---|---|
| RCRA | Resource Conservation and Recovery Act |
| SBW | sodium-bearing waste |
| scfm | standard cubic feet per minute |
| SGV | superficial gas velocity |
| SPT | System Performance Test |
| SRE | system removal efficiency |
| SVOC | semivolatile organic compound |
| TCDD | tetrachloro dibenzo-p-dioxin |
| TIC | tentatively identified compound |
| µg | microgram |
| VOC | volatile organic compound |
| WFT | waste feed tank |

# 1. INTRODUCTION

## 1.1 Facility and Background Information

The Idaho Nuclear Technology and Engineering Center (INTEC), located at the Idaho National Laboratory (INL), is a Department of Energy (DOE) facility that was previously used to reprocess nuclear fuel. INTEC currently has in storage approximately 840,700 gallons of sodium-bearing liquid radioactive (mixed) waste. This waste is comprised of acid decontamination solutions that contain high levels of sodium. This waste also contains toxic metals and trace concentrations of organics that are regulated under the Hazardous Waste Management Act (HWMA)/Resource Conservation and Recovery Act (RCRA).

Based upon an agreement with the Idaho Department of Environmental Quality (DEQ), DOE is preparing to treat this waste. CH2M-WG Idaho, LLC (CWI) has been contracted by DOE to build and operate the Integrated Waste Treatment Unit (IWTU) to process this waste. The IWTU has been constructed at INTEC and will use a patented steam reforming technology. This technology was selected because it will immobilize the radionuclides, sodium, potassium, sulfate, chlorides, fluorides, and nonvolatile heavy metals into a stable granular solid product, while destroying nitric acid, nitrates, and organic compounds present in the waste. The process converts nitrates and nitrites directly to nitrogen gas. Organic material in the waste is converted to carbon dioxide and water vapor. The off-gas (mainly carbon dioxide, water, nitrogen, and oxygen) from the process is filtered through sintered metal filters, high-efficiency particulate air (HEPA) filters, and sulfur-impregnated granular-activated carbon. The filtered off-gas then will be vented to the atmosphere through a stack.

The IWTU facility is defined as a miscellaneous treatment unit under the HWMA/RCRA Subpart X regulations, and has received a HWMA/RCRA Part B Permit. The IWTU also has a Permit to Construct and is included in the INL Title V air permit. At the time the Air Permit to Construct was submitted, the DEQ Air Program determined that Maximum Achievable Control Technology (MACT) EEE was not applicable to the IWTU. Both the Air and HWMA/RCRA permits were developed to follow this approach and the HWMA/RCRA permit was reviewed and concurred with by EPA at the time of its issuance. Although compliance with MACT is not required, these regulations were considered during the development of this plan. As a miscellaneous unit, the purpose of the test is to demonstrate that the emissions from the unit do not pose an unacceptable risk to human health and the environment. This will be completed by comparing the measured test emissions with the acceptable risk concentrations in the risk assessment plans.

Because the IWTU is newly constructed, it is necessary to provide an orderly and systematic startup and testing of all components. The testing performed in support of IWTU startup is described in PLN-2020, "IWTU Commissioning, Transition, and Readiness Plan." The scope of PLN-2020 includes vendor testing, construction acceptance testing, component testing, system operability testing, and integrated testing. The IWTU Startup Plan (PLN-3350) provides a brief description of the approach for the System Performance Test (SPT) described in this test plan. The scope of PLN-2020 also includes the system turnover process used to transfer control of each system to other organizations when construction is completed and when testing is completed. The anticipated testing schedule is as follows:

- Component testing began in September 2009
- Construction completed June 2011
- Hot nitrogen integrated testing completed March 2012
- Integrated testing with water feed in March–April 2012

- SPT in May 2012
- Submission of SPT report in August 2012, but no later than 90 days following completion of the SPT.

## 1.2 Test Plan Purpose and Organization

As stated in Section 1.1, the IWTU is classified under the HWMA/RCRA regulations as a miscellaneous treatment unit. A miscellaneous treatment unit must be located, designed, constructed, operated, maintained, and closed in a manner that will ensure protection of human health and the environment. Permits for miscellaneous treatment units contain such terms and provisions as necessary to protect human health and the environment, and include those requirements of other HWMA/RCRA-regulated treatment units that are appropriate for the miscellaneous unit being permitted.

Because the IWTU is a thermal treatment process, the standards applied to other hazardous waste thermal treatment processes have been identified as the most appropriate targets. Specifically, the standards applicable to new sources, 40 Code of Federal Regulations (CFR) 63.1219(b), "Emission Limits for New Sources," will be used, where appropriate, to evaluate the performance of the IWTU, along with an evaluation of the potential risk posed by the IWTU emissions.

This test plan has been prepared to serve as a SPT plan for the IWTU. The objective of the SPT is to gather emissions data that can be used in evaluating the IWTU against the identified performance targets, and for validation of the existing IWTU Risk Assessments. The SPT will also document process operating conditions that can be incorporated into the permit to ensure that continuing operations conform to the conditions under which the SPT was conducted.

Successful treatment is defined as demonstration that the steam reforming process performs as designed to convert liquid waste to a solid granular product within the limits set by the approved permit, and that the emissions observed during the test validate the risk assessments which ensure that the IWTU operations do not pose a threat to human health or the environment.

The purpose of this SPT plan is to identify and document the necessary process details; sampling, analytical, and quality assurance/quality control (QA/QC) procedures; and anticipated HWMA/RCRA permit operating conditions necessary to demonstrate compliance with the applicable permit conditions. This SPT plan has been organized into the following major sections:

1. Introduction
2. Feed Stream Description
3. Engineering Description
4. Test Design and Protocol
5. Sampling, Analysis, and Monitoring Procedures
6. Test Schedule
7. Operating Limit Objectives
8. Test Report.

Appendix A contains sampling procedures to be used during the test. Appendix B contains specific analytical method descriptions. Appendix C contains the Quality Assurance Project Plan (QAPjP) for the test activities. Appendix D contains EDF-9483, "IWTU System Performance Test: Risk Evaluation for

Organic Compounds Emissions," describing how the emissions data will be used in risk evaluation. Appendix E contains the locations of emission sampling points. Appendix F contains supplemental sampling information.

## 1.3 Waste Feed and Treatment Process Overview

Liquid mixed waste has been generated over the last five decades at INTEC, formerly called the Idaho Chemical Processing Plant, as a result of normal operations and facility deactivation and decommissioning activities. From December 1963 until June 2000, the Waste Calcining Facility and the New Waste Calcining Facility (NWCF) processed the high-level liquid waste generated from reprocessing nuclear fuel into a granular, solid form. As of April 2011, approximately 840,700 gallons of sodium-bearing waste (SBW) remained in Tank Farm tanks at INTEC.

The IWTU is designed to treat liquid SBW to produce a solid treatment product for disposal. The IWTU will utilize steam reforming technology, which includes a dual fluidized-bed process that uses superheated steam, carbon, and other additives to convert the SBW into a solid, granular treatment product that is packaged into canisters suitable for disposal. The process is named the IWTU because two fluidized-bed steam reformers, the Denitration Mineralization Reformer (DMR) and the Carbon Reduction Reformer (CRR), are integrated into a single treatment process with a common air pollution control system. The DMR, CRR, and material transfer and loadout systems utilize filters that are integral to the processing system used to capture and package the solid treatment product; these filters are not part of the air pollution control system. The IWTU air pollution control system includes the process HEPA filter system (which is located downstream from the DMR and CRR), the building ventilation HEPA filter system, and a granular-activated carbon mercury adsorption system.

## 1.4 Test Objectives and Approach

This SPT plan has been prepared to provide system performance testing of the IWTU system, with the objective of gathering emissions data that can be used to evaluate the IWTU against the identified performance targets, and for validation of the existing IWTU Risk Assessments. The SPT will also document process operating conditions that can be incorporated into the HWMA/RCRA permit to ensure that continuing operations conform to the conditions under which the SPT was conducted. During the SPT, the IWTU will be operated at "bounding" conditions, which have been selected to maximize emissions.

It may not be possible to maximize all conditions at one time. Therefore, two test conditions have been incorporated into the test protocol. The IWTU will process mixed waste during the SPT. Conducting the SPT on mixed waste will establish operating conditions that will be included in the HWMA/RCRA permit. More specifically, the objectives of the SPT are as follows:

1. Demonstrate compliance with the operating conditions found in Module VI of the HWMA/RCRA Part B Permit, and determine actual emissions rates of carbon monoxide, particulate, hydrocarbon, hydrogen chloride, chlorine, mercury, semivolatile metals, low volatility metals, and dioxins and furans
2. Establish permit operating limits for waste feed rate, process operating conditions, gas flow rate, and off-gas treatment system components
3. Gather emissions information for validation of the existing IWTU Risk Assessments
4. Demonstrate nitrogen oxide emissions performance for air permit compliance.

## 1.5 Test Protocol Summary

The proposed SPT is comprised of two test conditions, each comprised of four replicate sampling runs. (Note that three runs are required by the regulations. The fourth run is being added as a contingency.) During the testing, the IWTU system will be operated at maximum waste feed rate, maximum process gas flow rate, minimum DMR and CRR temperature, minimum DMR off-gas hydrogen concentration, minimum CRR off-gas oxygen concentration, and conditions for the off-gas treatment components (as described in Section 4.2).

A more detailed description of the test operating conditions and objectives is provided in Section 4.3 of this plan.

## 1.6 Development of Permit Limits

Operating limits will be developed and included in the HWMA/RCRA permit to ensure that the IWTU complies with the applicable environmental performance standards at all times. These operating parameters as well as suggested limits and a list of anticipated process operational parameters are fully described in Section 7 of this plan.

## 1.7 System Performance Test Team Roles and Responsibilities

Successful completion of the SPT will require strong communication and coordination between all participants. To ensure success, an SPT team will be established that provides overall coordination and oversight of the test activities. This team will be comprised of CWI Environmental, IWTU Operations, DEQ, DOE, Sampling Contractor, and consultant personnel. The roles and responsibilities for each of these entities are as follows:

- SPT Team Lead – CWI Environmental will have overall responsibility for safe completion of the testing, review of the results, and submittal of the final test reports and HWMA/RCRA permit modification requests to the DEQ. The SPT Team Lead ensures effective communication and interface occurs across all disciplines.
- IWTU Operations – The IWTU nuclear facility manager (NFM) directs actual safety and operation of the facility. Requested changes to the operational parameters must be discussed and approved by the NFM who will then direct operations personnel to make the desired changes. The NFM will be the primary interface between the SPT team and operating personnel.
- IWTU Test Engineer – The IWTU Test Engineer ensures test parameters are reached and maintained and that required samples and data are collected in accordance with IWTU-TI-103, "IWTU System Performance Test."
- DEQ Observer – The DEQ Observer provides regulatory oversight of the test to ensure the testing is completed in a manner that supports the final regulatory approval of the SPT Report. Proposed deviations to the test conditions or upset conditions will be discussed and approved by the DEQ Observer prior to continued testing.
- DOE Observer – The DOE Observer provides department oversight and consultation.
- Test Manager – The Test Manager (CWI contractor) coordinates the field activities of the Sampling Contractor, provides consultation to the team, and maintains the test log including documenting sampling activities and issue description and resolution. The Test Manager also reviews sample analyses results, and prepares and submits the draft final test report to CWI SPT Team Lead.

- Technical Advisor – The Technical Advisor (CWI contractor) provides technical support and recommendations.
- Sampling Contractor Lead – The Sampling Contractor Lead provides sampling oversight and direction to the sampling team to ensure the safe completion of sampling activities and sample recovery. The Sampling Contractor Lead also coordinates shipment of samples to the laboratory.

A fundamental tenet of safe operation of a nuclear facility is to maintain Conduct of Operations principles, especially as they pertain to the IWTU Control Room. Permission will be required from the Control Room Operator (CRO) prior to entering the control room. Only business related conversations may be held in the control room and personnel need to recognize that even business discussions can be distracting. Upon completion of the required work, personnel shall immediately leave the area. Discussions with operating personnel shall be limited to the alternate operator on duty. No discussions shall be held with the primary operator at the controls. The area immediately surrounding the control station has been clearly identified and is marked At-the Controls. No personnel other than the operators on duty can enter the At-the Controls. No food or drink is allowed in the At the Controls.

To ensure that the SPT Team is cognizant of these Conduct of Operations requirements and able to successfully monitor the test, a dedicated computer work station will be set up in close proximity to the IWTU control room. Test personnel will have access to this station throughout the test. This system will allow the test team to monitor the operating conditions of the process, including access to process and utility instrumentation. It will have a read-only interface with the Distributed Control System to allow the user to monitor data.

## 1.8 Reference Documents

Reference documents used in developing this plan include the following:

- Permit No.: P-2008.0199, *Permit to Construct (PTC) for Integrated Waste Treatment Unit (IWTU) at Idaho National Laboratory (INL), Idaho Nuclear Technology and Engineering Center (INTEC)*, Issued May 3, 2007, Modified August 31, 2009
- Permit No.: TI-2009.0114, *Idaho National Laboratory Site, Tier I Operating Permit (Facility ID Nos. 023-00001, 011-00022),* Issued June 28, 2005, Modified October 30, 2009
- American Society for Testing and Materials, *Annual Book of ASTM Standards*, latest annual edition
- 40 CFR 60, "Standards of Performance for New Stationary Sources, Appendix A, "Test Methods and Procedures," U.S. Environmental Protection Agency
- SW-846, *Test Methods for Evaluating Solid Wastes Physical/Chemical Methods*, Third Edition, U.S. Environmental Protection Agency, 1986 and updates
- EPA530-R-01-001, *Risk Burn Guidance for Hazardous Waste Combustion Facilities*, U.S. Environmental Protection Agency, July 2001
- PER-111, "Partial Permit for HWMA Storage and Treatment for the Liquid Waste Management System at the Idaho Nuclear Technology and Engineering Center on the Idaho National Laboratory," September 13, 2011.

Waste Characterization and Process Information Sources:

- Barnes, C. M., Millet C. B., and Johnson, V. J., *Feed Composition for the Sodium Bearing Waste Treatment Process,* INEEL/EXT-2000-01378, Rev. 4, Idaho National Engineering and Environmental Laboratory, Bechtel BWXT Idaho, LLC, June 2004
- EDF-8471, "VES-WM-187, VES-WM-188, AND VES-WM-189 Tank Characterization," Rev. 0, July 30, 2008
- SDD-227, "IWTU - Sodium Bearing Waste Steam Reforming Unit," Rev. 3, April 9, 2009
- SDD-230, "IWTU – Process Exhaust System," Rev. 4, April 1, 2009.

# 2. FEED STREAM DESCRIPTION

The IWTU is designed to treat acidic liquid waste resulting from normal operations and facility deactivation and decommissioning activities. The process is also fed several reactants and fluidized bed materials.

The mixed waste will be delivered from INTEC Tank Farm tanks and transferred to the IWTU feed tank. From the IWTU feed tank, the waste will be pumped into the steam reforming process.

The mixed waste from the tank farm and the waste product produced from this waste will not be sampled for the SPT. The feed has been characterized in INEEL/EXT-2000-01378, Revision 4, Feed Composition for the Sodium Bearing Waste Treatment Process, provided in the HWMA/RCRA Part B Permit for the IWTU. Using this process knowledge and the process controls the composition of the waste product will be known and will not require further characterization. Sampling of these two streams is further complicated by the high radiation levels associated with the samples. Obtaining a laboratory to provide the analytical services, transporting the samples to the laboratory and back to the INL for disposal is not practicable due to the high radiation levels. In addition, analysis of the samples would not be accurate due to the dilution of the samples to get the radiation levels down to a point where the samples can be handled.

## 2.1 Feed Stream Characteristics

### 2.1.1 Waste Feed Streams

The liquid mixed wastes consist of radioactive aqueous solutions with high concentrations of nitric acid, nitrates, alkali metals, aluminum, undissolved solids, and a wide variety of other inorganic oxides, referred to as "sodium-bearing waste" (SBW). This waste also contains toxic metals and trace concentrations of organics that are regulated under HWMA/RCRA. Detailed information concerning the waste material is included in the HWMA/RCRA permit. A summary of the maximum concentrations of major constituents in the waste streams (based on the most recent analysis) is provided in Table 2-1.

Table 2-1. Sodium-bearing waste maximum constituent concentrations.[a]

| Constituent/Tank | VES-187 (mol/L) | VES-188 (mol/L) | VES-189 (mol/L) | Maximum (mol/L) |
|---|---|---|---|---|
| Hydrogen | 1.66E+00 | 3.10E+00 | 2.67E+00 | 3.10E+00 |
| Aluminum | 5.72E-01 | 7.47E-01 | 6.91E-01 | 7.47E-01 |
| Antimony | 5.34E-06 | 8.64E-06 | 1.89E-06 | 8.64E-06 |

Table 2-1. (continued).

| Constituent/Tank | VES-187 (mol/L) | VES-188 (mol/L) | VES-189 (mol/L) | Maximum (mol/L) |
|---|---|---|---|---|
| Arsenic | 3.79E-06 | 3.92E-06 | 3.93E-06 | 3.93E-06 |
| Barium | 6.54E-05 | 1.06E-04 | 5.90E-05 | 1.06E-04 |
| Beryllium | 8.58E-06 | 1.75E-05 | 1.68E-05 | 1.75E-05 |
| Boron | 9.76E-03 | 2.10E-02 | 1.95E-02 | 2.10E-02 |
| Cadmium | 7.84E-04 | 3.15E-03 | 3.59E-03 | 3.59E-03 |
| Calcium | 4.47E-02 | 7.31E-02 | 7.15E-02 | 7.31E-02 |
| Cesium | 1.10E-05 | 3.91E-05 | 2.68E-05 | 3.91E-05 |
| Chlorine | 2.12E-02 | 1.70E-02 | 2.62E-02 | 2.62E-02 |
| Chromium | 3.36E-03 | 6.10E-03 | 5.65E-03 | 6.10E-03 |
| Copper | 2.50E-09 | 8.64E-04 | 9.54E-04 | 9.54E-04 |
| Fluorine | 3.91E-03 | 7.65E-03 | 7.81E-03 | 7.81E-03 |
| Iron | 2.01E-02 | 2.74E-02 | 2.68E-02 | 2.74E-02 |
| Lead | 1.12E-03 | 1.12E-03 | 1.15E-03 | 1.15E-03 |
| Magnesium | 2.49E-02 | 2.78E-02 | 2.21E-02 | 2.78E-02 |
| Manganese | 1.42E-02 | 1.68E-02 | 1.94E-02 | 1.94E-02 |
| Mercury | 1.74E-03 | 6.33E-03 | 6.33E-03 | 6.33E-03 |
| Nickel | 2.14E-03 | 2.88E-03 | 2.37E-03 | 2.88E-03 |
| Nitrate | 5.32E+00 | 7.56E+00 | 7.10E+00 | 7.56E+00 |
| Phosphate | 8.07E-03 | 7.49E-04 | 2.35E-03 | 8.07E-03 |
| Potassium | 1.68E-01 | 2.08E-01 | 2.39E-01 | 2.39E-01 |
| Selenium | NM[b] | 4.74E-06 | 9.79E-07 | 4.74E-06 |
| Silicon | 3.44E-04 | 4.13E-04 | 4.04E-04 | 4.13E-04 |
| Silver | 1.77E-06 | 4.40E-06 | 2.89E-06 | 4.40E-06 |
| Sodium | 1.71E+00 | 1.74E+00 | 1.93E+00 | 1.93E+00 |
| Sulfate | 4.29E-02 | 5.83E-02 | 5.85E-02 | 5.85E-02 |
| Vanadium | 1.55E-05 | 2.75E-05 | 2.55E-05 | 2.75E-05 |
| Zinc | 1.00E-03 | 1.08E-03 | 1.15E-03 | 1.15E-03 |
| Zirconium | 2.67E-05 | 2.58E-03 | 3.59E-04 | 2.58E-03 |
| **Radionuclides** | **Ci/liter** | **Ci/liter** | **Ci/liter** | **Maximum Ci/liter** |
| Cesium (Cs) | 3.04E-02 | 6.96E-02 | 5.01E-02 | 6.96E-02 |
| Strontium (Sr) | 2.38E-02 | 5.24E-02 | 3.88E-02 | 5.24E-02 |
| Yttrium (Y) | 2.38E-02 | 5.24E-02 | 3.88E-02 | 5.24E-02 |

Table 2-1. (continued).

| Constituent/Tank | VES-187 (mol/L) | VES-188 (mol/L) | VES-189 (mol/L) | Maximum (mol/L) |
|---|---|---|---|---|
| Barium (Ba) | 2.87E-02 | 6.58E-02 | 4.74E-02 | 6.58E-02 |

a. Concentration data are reported in EDF-8471 with the following exceptions:

1. Cesium, copper, and magnesium were not measured in EDF-8471, so the results are from estimates reported in interoffice memo MIL-05-05 from Clark Millet to Scott Roesener, July 11, 2005.
2. Reported nitrate concentration is based on charge balance of all measured species and not on the measured concentration of nitrate.

b. NM = not measured.

### 2.1.2 Other Feed Streams

A variety of materials are added to the IWTU as reactants, fluidizing media, and fluidized bed material. These materials include air, water, steam, nitrogen, oxygen, coal, carbon/coke, alumina, and aluminum hydroxide.

## 2.2 Feed Stream Management

SBW from the INTEC Tank Farm is transferred to the three existing Blend and Hold Tanks in the NWCF and then transferred in batches to the IWTU waste feed tank (WFT). A transfer/isolation valve in the line between the NWCF and IWTU establishes the boundary between the two facilities. The waste feed pump can empty the WFT through the transfer line back to the NWCF Blend and Hold Tanks. The WFT is utilized on the front of the IWTU to provide retention time during the periods that waste is not being transferred from the NWCF. The feed pump normally recirculates the solution in the feed tank at 30 gpm. A side stream is sent to the DMR as the waste feed. The liquid waste feed is introduced as a controlled spray mist into the DMR.

The WFT is a vertical interim/day storage tank designed to store SBW feed, which is hazardous and radioactive (mixed). The tank is sized for 8 hours of IWTU operation at a normal processing rate of 3.1 gpm to reduce the impact of interruption of SBW supply from the NWCF or from the Tank Farm Facility. It is a 2,192-gallon vessel with a working volume of 1,500 gallons. The WFT is constructed of 304L SS (stainless steel) to eliminate the possibility of corrosion, provide protection/shielding from radiation, and provide a surface for decontamination, as needed. The WFT has an overall height of 115 1/8 in. and shell thickness of 7/8 in., with an inside diameter of 84 in. It is constructed with dished heads. The tank has design conditions from 10 to 79°C, with a normal operating temperature of 16 to 52°C. Maximum design pressure is 50 psig, but the tank normally operates at ambient pressure (12.38 psia). The WFT is overfill protected and vented through a vent separator where liquid is routed to the 4-pack sump and gas is routed to the 4-pack cell. Lugs on the WFT are bolted to the WFT support frame, which is anchored to the floor of the process cell. The WFT is provided with an external waste feed pump and a waste feed mixing jet.

Superheated steam is injected at the bottom of the DMR into a sparge ring, and steam enriched with oxygen is injected just above the sparge ring through four fluidizing gas distributors. The sparge ring is fed with steam only. This maintains fluidization at the DMR wall and maintains an oxygen-free environment in the bottom of the DMR.

Granular coal is used in the DMR as a fuel/reactant and is fed by gravity to the DMR from the coal airlock. Alumina is fed to the DMR as startup bed material. A common air lock is utilized to isolate the DMR atmosphere from the solids feed system. One solids injection location is provided for all additives.

A flow of oxygen and air is introduced into the lower, middle, and upper section of the CRR bed, where the oxygen converts the CRR bed and freeboard into an oxidizing environment.

Petroleum coke and a mixture of air and oxygen are added to the CRR to maintain the desired temperature. Aluminum hydroxide is used in the CRR as a reactant. Alumina is fed to the CRR as startup bed material. A common air lock is utilized to isolate the CRR atmosphere from the solids feed system. One solids injection location is provided for all additives.

# 3. ENGINEERING DESCRIPTION

## 3.1 Facility Process Overview

The IWTU treats mixed wastes. Mixed wastes at INTEC consist of radioactive aqueous solutions with high concentrations of nitric acid, nitrates, alkali metals, aluminum, undissolved solids, and a wide variety of other inorganic oxides. The IWTU destroys nitric acid, nitrates, and organic materials present in the SBW, and produces a dry, solid, particulate carbonate product. The steam reforming process has been proven to efficiently treat and solidify radionuclides, sodium, potassium, sulfate, chlorides, fluorides, and nonvolatile heavy metals into a dry solid particulate matrix. In addition, the process converts nitric acid and nitrates directly to nitrogen gas in the reformer. Organic material in the SBW is converted to carbon dioxide and water vapor in the steam reformers by a combination of steam reforming and oxidation reactions.

The IWTU consists of several subsystems and components. The waste transfer and feed subsystem transfers mixed waste from the Tank Farm Facility into the WFT within the IWTU facility and supplies it to the steam reforming process equipment for treatment. The DMR reduces nitrates and nitric acid into nitrogen and produces a dry solid product. The process gas filter (PGF) removes entrained particulates from the gas stream as the gas is transferred from the DMR to the CRR. The CRR reduces nitrogen oxide and oxidizes hydrogen, carbon monoxide, and organics in the process gas stream. The off-gas cooler (OGC) cools the process gas exiting the CRR, and the off-gas filter (OGF) removes particulate out of the gas exiting the OGC.

The process exhaust system receives the effluent of the OGF, and filters, monitors, and then exhausts process gases. The process exhaust system is composed of off-gas blowers, HEPA filters, granular-activated carbon beds used as a mercury adsorber, exhaust blowers, a mixing box, and a stack.

### 3.1.1 Principles of Operation

The principal process functions of the SBW Reforming Unit are:

- Conversion of metals to stable solids
- Treatment of hazardous metals
- Nitrate reduction to nitrogen gas
- Capture of radioactive constituents in a carbonate product
- Destruction of HWMA/RCRA organics.

### 3.1.2 Conversion of Metals to Stable Solids

The alkali metals present in the SBW will form mainly carbonates, the other inorganic constituents will form metal oxides and spinels with the iron in the SBW, and aluminates will form from the aluminum in the SBW. The carbonates are made from the reaction of carbon dioxide, from the steam reforming reactions in the DMR, with the alkali oxides from the SBW. The combined SBW inorganic minerals are largely carbonates and will have melting points in the 750 to 850°C range. Therefore, in order to reliably convert SBW constituents into carbonates, the DMR will be operated at no higher than 680°C in the fluidized bed to prevent the carbonates and other salts from partially melting in the DMR.

### 3.1.3 Treatment of Hazardous Metals

The strong reducing environment in the DMR is expected to convert certain hazardous metals to nonhazardous valence states [for example, Cr(VI) to Cr(III)]. Mercury present in the waste feed will not be bound-up in the solid product, but will be volatilized and converted to elemental mercury in the reformers. The gases from the DMR and the CRR will be treated in mercury adsorbers using a sulfur-impregnated granular-activated carbon bed prior to exhausting to the atmosphere.

### 3.1.4 Nitrate Reduction to Nitrogen Gas

The steam reforming process technology reduces nitrates and nitric acid to elemental nitrogen to the following extent:

- Residual nitrate content in reformed solid waste product is expected to be less than 10 parts per million by weight (ppmw)
- Greater than 97% of nitrates and nitric acid in waste feed to nitrogen gas
- Total nitrogen oxide in off-gas from the reformers is expected to be less than 1,500 ppmw.

Several specific features and reactions have been incorporated into the steam reforming process to achieve the referenced results:

- Fluidized bed material with high surface area and high heat transfer capability.
- High-energy generation in the DMR to evaporate and superheat the water in the waste feed.
- Strongly reducing condition that is provided by the carbonaceous and metal-based reactants in the DMR. Superheated steam that generates carbon monoxide and hydrogen inside the bed from the reaction of steam with the carbon reductant(s) ensures that all oxygen fed to maintain temperature is consumed, thus maintaining the reducing atmosphere.

The incoming waste feed to the DMR is atomized into small droplets that react with the fluidized mineral particles and are instantly dried. The large active surface of dried nitrates readily reacts with the hot carbon reductant particles, carbon monoxide and hydrogen gases, and the reduced metal and metal oxide particles in the fluidized bed. The presence of excess carbon and metal oxides from the SBW results in the reduction of nitrates to only trace levels of residual nitrates in the solid reformed product and the reduction of nitrogen oxide. A list of several of the reaction mechanisms whereby nitrates are converted to nitrogen gas follows:

$2NaNO_3 + C \rightarrow Na_2CO_3 + NO + NO_2$
$NO_2 + C \rightarrow NO + CO$
$NO_2 + H_2 \rightarrow NO + H_2O$
$NO_2 + CO \rightarrow NO + CO_2$
$2NO + 2C \rightarrow N_2 + 2CO$

$2NO + 2CO \rightarrow N_2 + 2CO_2$
$2NO + 2H_2 \rightarrow N_2 + 2H_2O$.

### 3.1.5 Destruction of HWMA/RCRA Organics

The steam reforming process destroys organics in the waste, including HWMA/RCRA organic constituents. Specifically, the process destroys HWMA/RCRA organic waste codes F001, F002, and F005 that are present in trace concentrations in the SBW waste stream. This destruction occurs through oxidation. The organics are initially volatilized and steam reformed into carbon dioxide ($CO_2$), carbon monoxide (CO), hydrogen ($H_2$), and a small quantity of light hydrocarbons (generally less than 1,000 parts per million by volume [ppmv], with methane being the main constituent). These reduction and reforming reactions occur in the DMR and lower portion of the CRR. Generally, the ratio of carbon dioxide to carbon monoxide is 50:1 from the DMR. The light hydrocarbons and the carbon monoxide and hydrogen gases generated from the steam reformation of carbon and organic materials are further oxidized to carbon dioxide and water vapor by adding oxygen in the CRR.

The following typical reactions occur:

DMR:
$CxHy + xH_2O \rightarrow xCO + (x+y/2)\ H_2$
$CxHy + 2(x-y/4)H_2 \rightarrow xCH_4$
$CO + H_2O \rightarrow CO_2 + H_2$

CRR:
$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$
$2CO + O_2 \rightarrow 2\ CO_2$
$2H_2 + O_2 \rightarrow 2\ H_2O$.

## 3.2 IWTU System Process Description

The mixed waste feed is introduced as a controlled spray mist into the DMR. Atomization of the waste feed is provided by a combination flow of air and nitrogen. The feed from the waste feed tank is pumped into the DMR through three waste feed nozzles. To start the feed to the DMR, atomization gas to each nozzle is confirmed on FH-160004, 005, and 006 then FCV-SRC-131-020 is opened. Three flow control valves, FCV-SRC-140-017, -018, and -019 control the waste feed to the three waste feed nozzles at 0.75 to 1.55 gpm each, with a not-to-exceed total feed rate of 3.5 gpm. Feed pressure in each of the nozzles is monitored and alarmed to provide indication of a plug in the nozzle or a line break. The waste enters the waste feed nozzles and is atomized into the heated fluidized bed with a controlled flow of nitrogen and instrument air. Each waste feed line is provided with a pressure transmitter which provides high and low pressure alarms. If a high pressure alarm occurs, it is an indication that the associated waste feed nozzle may be plugging with solids. The waste feed nozzles are provided with purge nitrogen connections to allow the line to be pressurized with nitrogen to clear any built up solids in the nozzle, should this occur. If a low pressure alarm occurs, the waste feed to the DMR will be stopped by closing the DMR feed valves. The waste feed pump will remain in tank recirculation mode. The three nozzles can be purged/flushed with high pressure nitrogen, water, and/or nitric acid solution in addition to the regular nitrogen purge described above. The atomizing instrument air and nitrogen flow to the waste feed nozzles are continuously monitored. If a low-low flow alarm occurs, the corresponding waste feed isolation valve and corresponding atomizing gas supply valve will be closed, but nitrogen atomizing flow will be maintained. The DMR contains a fluidized bed of product particles. The bed particles are fluidized by introduction of superheated steam. In the DMR, liquids are evaporated, trace organics are broken down into short chain hydrocarbons, nitric acid and nitrates are partially destroyed, and the inorganic chemicals

in the waste feed are converted to a solid waste product. Coal is added to the bed to supply the heat required for the reactions and to produce a reducing atmosphere. The DMR operates at about 620 to 660°C. The gases (steam, nitrogen, carbon dioxide, hydrogen, methane, carbon monoxide, mercury, and nitrogen oxide) are then passed through a sintered metal PGF and sent to the CRR. During developmental testing at Hazen Research Center, it was determined that high calcium content in the coke, used as an additive to the process, resulted in bed agglomeration and poor operations. Consequently, there was some concern that calcium content in the bed should also be monitored and controlled. However, by replacing the coke that was initially used with coal, the bed agglomeration problem from the high calcium content was eliminated. During the SPT and all further operation, this same type of coal will always be used. As such, it will not be necessary to monitor coke calcium content.

The CRR contains inert bed material. In the lower part of the fluidized bed, any nitrogen oxide gases from the first reformer are further reduced, and in the upper part of the fluidized bed, residual organics, carbon monoxide, and hydrogen are oxidized to carbon dioxide and water vapor. The CRR is refractory lined and operates at about 900 to 1,000°C. Carbon and oxygen/air are added to this bed to supply the required heat for the reactions and to provide the required oxygen to oxidize residual organics, carbon monoxide, and hydrogen to carbon dioxide and water vapor. The gases (steam, nitrogen, carbon dioxide, oxygen, and mercury) from the process are cooled by a water spray cooler and then filtered through the sintered metal OGF. Once out of the OGF, the process gas travels in a duct that exits the process cell and enters the process exhaust system.

The elapsed time from entry of waste to the DMR to offgas exit from the OGF is approximately 48 seconds. Superficial gas velocity (SGV) will be measured and maximized (1.3 ft/sec) during the SPT as an indicator of minimum gas residence time. The SGV is the velocity of gas in the DMR at the fluidizing gas distributor. The SGV is calculated by the DCS using the ideal gas law to convert the measured flow at the flow element upstream of the superheater to the actual flow rate in the DMR. The SGV is important because at too low a flow, the bed would defluidize and reactions would be greatly hindered. If the flow is too high, then it would blow the fluidized bed material over the top of the DMR and into the PGF. The permit has both high (1.5 ft/sec) and low (0.3 ft/sec) alarms and waste feed cutoffs. SGV flow of 1.0 ft/sec is anticipated during normal operations.

The process exhaust system receives cooled and filtered off-gas from the steam reforming unit. The process off-gas stream then flows through a bank of HEPA filters and a granular-activated carbon mercury adsorber. A continuous emissions monitor system (CEMS) is provided on the process exhaust to monitor for carbon monoxide and oxygen. Filtered air from the facility heating, ventilating, and air conditioning (HVAC) system is combined with the process exhaust in the air mixing box. A radiation monitoring system is provided for radiological pollutants (radioactive particulate and iodine-129). The off-gas is discharged through the facility exhaust stack.

Figure 3-1 illustrates the IWTU steam reforming process. The process exhaust system is shown in Figure 3-2. More detailed descriptions of the IWTU components follow.

### 3.2.1 Denitration Mineralization Reformer

The DMR is constructed of Haynes 556. The DMR is a pressure vessel designed for temperatures between -29 and 700°C, with a normal operating temperature of 640°C. The DMR has an internal design pressure of 14 psig, with an operating pressure of 2 to 8 psig. The DMR has a capacity of 3,040 gallons. The DMR tan-tan length is 18 ft 9 in., with an upper section inner diameter (ID) of 5 ft 8 in. and seam-seam length of 10 ft 1 in., a transition of 1 ft 6 in., and a lower section of 4 ft 0 in. ID and seam-tan length of 7 ft 0 in. The DMR is equipped with three cyclones constructed of Haynes 556, which are located in the upper elevation of the DMR. The cyclones keep the fluidized bed particles from flowing through to the SBW PGFs by returning the particles to the fluidized bed.

The bottom of the DMR is equipped with an auger/grinder, which is constructed of Inconel 625 and located above the sump of the DMR under the fluidized bed. The auger/grinder consists of two rotating paddles with small augers on each side to rotate the solid material toward the paddles. The paddles rotate with the motor shaft to grind the material over a mesh screen, which will allow particles up to 3/8 in. to flow through the screen to a collection pot. The solids produced from the grinder are transferred to the product receiver cooler.

It has been determined that given the feed rates of the SPT operational conditions, the bed material will need to be transferred up to two times during any sampling activity. Therefore, bed transfers are considered a normal part of operation of the IWTU, and SPT sampling activities will continue during bed transfers.

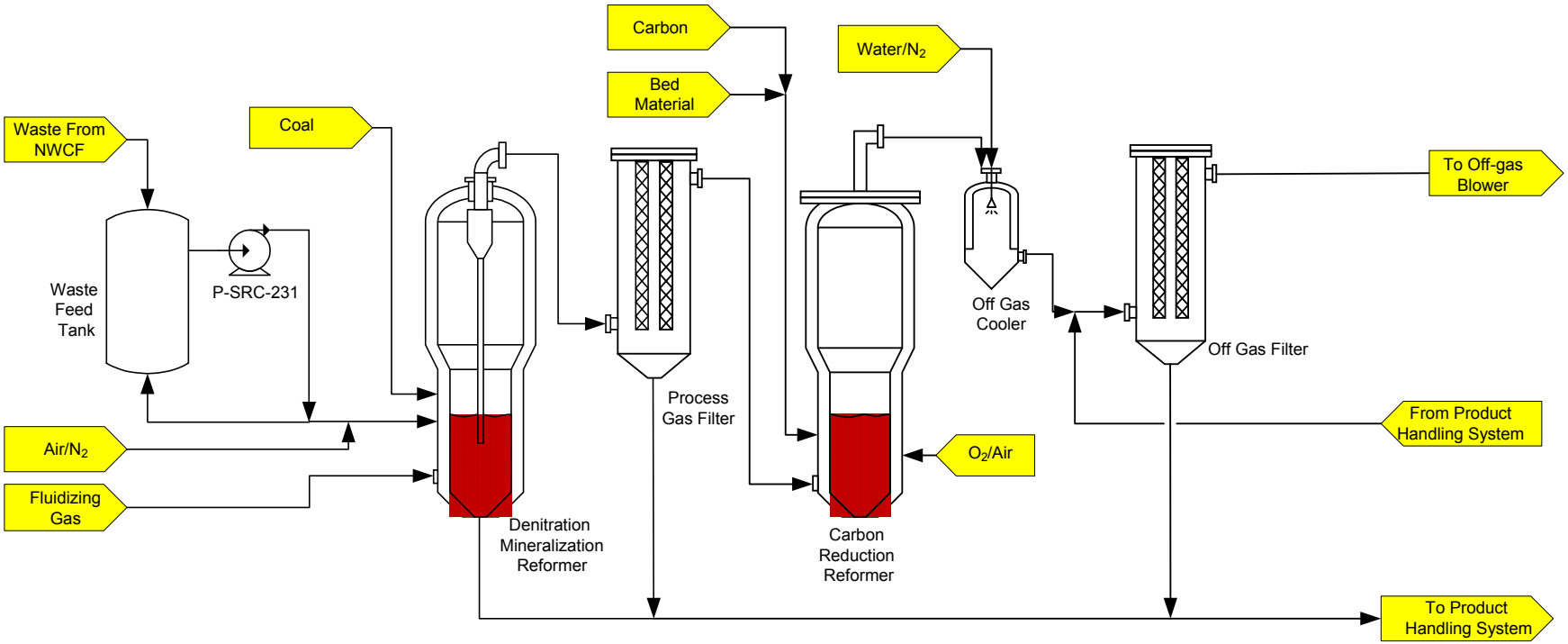


Figure 3-1. IWTU steam reforming components simplified process flow diagram.

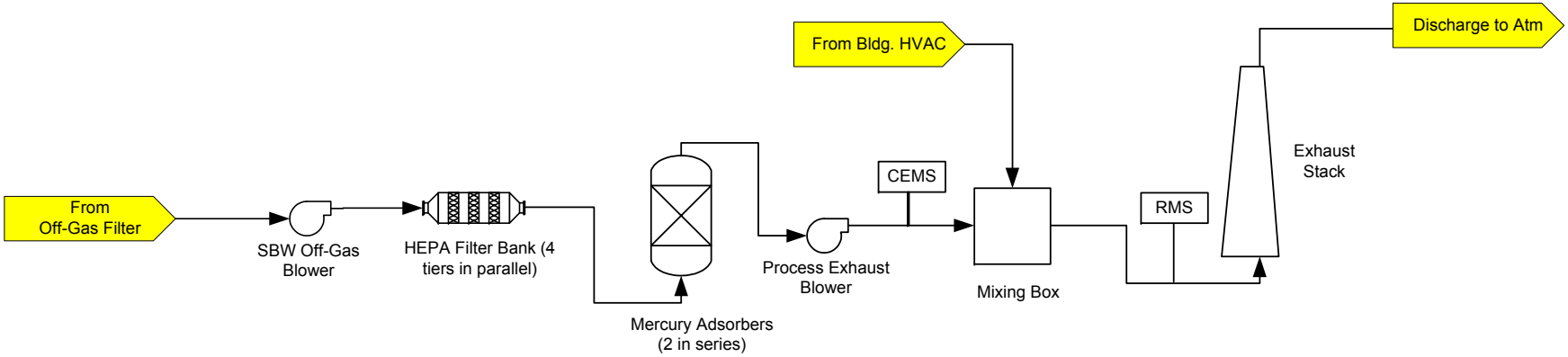


Figure 3-2**.** IWTU process exhaust components simplified process flow diagram.

### 3.2.2 Sodium-Bearing Waste Process Gas Filter

The PGF is 105 in. ID with 8 in. of insulation, a dished head, and contains sintered powdered metal filter elements. The PGF is constructed of Haynes 556 material. It is designed with a maximum temperature of 680°C and a maximum pressure between 0 and 13 psig. The PGF operates between 610 and 660°C, with a normal operating pressure from 2 to 8 psig. The PGF has a capacity of 5,716 gallons. The design flow rate for the PGF is 1,829 standard cubic feet per minute (scfm), with a normal flow rate of 1,460 scfm, and the PGF removes 99% of particles 2.0 microns or larger. The PGF includes a tubesheet comprised of bundles containing multiple filter elements that filter the process gas. Each bundle can be remotely removed and replaced if filter-changeout is needed. The inlet pressure and outlet pressure of the PGF are monitored, and the differential pressure across the filter is calculated. To prevent reaching a high differential pressure across the filter, a timer on the nitrogen system initiates a pulse of nitrogen to clean the filter bundles. Nitrogen is pulsed sequentially through the filter bundles and the removed filter cake particulates drop by gravity to the bottom cone of the filter vessel for collection. If the differential pressure across the filter remains elevated after pulsing with nitrogen, additional pulsing may be completed. When necessary, the process is shutdown and the filter bundles are removed and replaced.

### 3.2.3 Carbon Reduction Reformer

The CRR has a capacity of 6,686 gallons. The CRR is designed for temperatures between -29 and 1,160°C, with an internal design pressure of 10 psig. The normal operating temperature is between 900 and 1,000°C, with an internal pressure of –1.6 psig. The CRR is manufactured from SA-516-70 carbon steel plate, and is refractory lined. The CRR has a tan-tan length of 18 ft 4-3/4 in. (not including sump section), with an upper section inner diameter of 8 ft 0 in. The refractory is approximately 10 in. thick within the entire vessel; therefore, the upper inner diameter of the vessel is 6 ft 4 in. with the refractory. The upper portion of the vessel has a seam-seam length of 9 ft 11 in., a transition of 2 ft 3-3/4 in., and a lower section inside diameter of 6 ft 2 in. (4 ft 6 in. with refractory), and seam-tan length of 5 ft 10 in. The length from the tangent line (at 6 ft 2 in. diameter) to the bottom of the skirt support is 6 ft 3 in. The upper portion of the CRR and the entire crossover duct is surrounded by a stainless steel insulated shroud that is offset for the outer surface of the CRR.

### 3.2.4 Sodium-Bearing Waste Off-Gas Cooler

The SBW OGC has a capacity of 4,358 gallons. The SBW OGC is a vertically suspended insulated vessel located adjacent to the CRR. The vessel insulation covers the top head and the cylindrical shell of the vessel. The inlet nozzle is refractory lined. The OGC is designed for off-gas temperatures of 16 to 1,000°C with a design pressure of –10 psig, with normal operating temperatures between 140 to 925°C and an operating pressure of –1.5 psig. Demineralized water enters the OGC at approximately 5.0 gpm, which cools the off-gas to approximately 160–200°C. The vessel is constructed of AL6XN for the upper shell and Hastelloy C-276 for the lower cone, with a Haynes 556 inner liner through the entire vessel. The OGC has an ID of 8 ft, with a liner ID of 7 ft 7 in., and tan-tan height of 10 ft, with a dished top head and a conical bottom. The inside of the upper shell is insulation lined and is 3 in. thick, with 3-to-7-13/16-in.-thick insulation around the head and 7-1/2-in. refractory on the inlet gas nozzle. Insulation is also provided for the exterior of the vessel, which reduces the amount of heat transferred into the CRR cell.

### 3.2.5 Sodium-Bearing Waste Off-Gas Filter

The OGF has a capacity of 6,869 gallons. The SBW OGF is a sintered pleated fiber metal, pulse-jet type filter that separates solids larger than 2 microns from the cooled CRR off-gas. The filter is operated continuously at approximately –2.5 psig, and 140 to 200°C, with design parameters of –5 to 7 psig and 110 to 250°C. The vessel height is 18 ft 3-3/8 in. from the bottom seam to the face of the head flange, with a dished head and a cone-shaped bottom. The vessel diameter is 114 in. The OGF is constructed of AL6XN, and is provided with 4 in. of insulation. The OGF is designed to remove more than 99% of 2.0 micron solids at a normal flow rate of 1,460 scfm in the off-gas stream.

### 3.2.6 Sodium-Bearing Waste Off-Gas Blowers

The SBW off-gas blowers provide the vacuum in the SBW treatment system. Both blowers will be sized at 100% capacity and will be normally operating one at a time with an installed spare. Each blower is equipped with a variable-speed motor designed to generate a static differential pressure of approximately –70 in. water column (w.c.) at approximately 6,000 actual cubic feet per minute (ACFM).

### 3.2.7 Process HEPA Filters

The process HEPA filter unit consists of four filter tiers, stacked vertically. The off-gas flow is split equally between the tiers and flows through each one while kept at a constant temperature. All off-gas piping and the process HEPA filters are insulated to maintain temperature in the off-gas system above 120°C. The HEPA filter housings will be manufactured of AL6XN material or equivalent. The filter housings are contained behind a shielding wall.

### 3.2.8 Mercury Adsorbers

The mercury adsorber unit is comprised of two beds that remove vapor phase mercury present in the off-gas. The mercury beds adsorbent material is sulfur-impregnated granular-activated carbon. The off-gas enters the two mercury adsorbers, which are positioned vertically, and flows in series, in an upward direction, through each granular-activated carbon bed. The treated off-gas exits the mercury adsorbers and enters the process exhaust blowers. The mercury adsorbers can be bypassed if needed by opening the two bypass valves and closing the inlet valve. However, during operations, the mercury adsorbers are only bypassed in a process upset situation. During the SPT, a test of the automatic waste feed cutoff (AWFCO) will be initiated that will bypass the mercury adsorbers and waste feed cutoff. After the AWFCO test, the adsorbers will be returned to service. The adsorbers will only be bypassed during system upset requiring activation of the AWFCO or the AWFCO test.

The mercury adsorbers are bypassed by the rapid shutdown system under the following conditions:

- There will be a time delay of 300 seconds before the mercury adsorbers are bypassed to ensure that all the mercury in the system has passed through the mercury adsorbers prior to being bypassed. The following are on a time delay after a rapid shutdown system trip:
    - High-high DMR pressure
    - Low-low DMR fluidizing gas flow
    - Low-low oxygen concentration in the CRR off-gas outlet
    - High-high oxygen concentration in the CRR off-gas outlet
    - Seismic switch

- Low-low INTEC-supplied compressed air pressure
- Low-low SBW off-gas blower motors current
- Low-low process exhaust blower motors current
- Loss of pressure in OGC surge water tank
- Low-low liquid level in OGC surge water tank
- Manual shutdown switch.

- The following are the rapid shutdown system trips that are concerned with personnel safety and protecting equipment and require immediate bypass of the mercury adsorbers with no time delay:
  - Low-low temperature in the CRR
  - High-high off-gas temperature after the OGC
  - High-high carbon monoxide at outlet of mercury adsorber
  - High-high temperature in outlet process HEPA filter/inlet to mercury adsorbers
  - Loss of nitrogen
  - Low-low DMR fluidizing gas pressure
  - Loss of electrical power
  - Pressure relief valves release DMR and PGF
  - High-high-high DMR pressure
  - Low-low DMR pressure
  - High-high-high CRR pressure
  - High-high alarm on the steam/$N_2$ to the fluidizing gas superheater

- Pressure relief valve-activated OGC outlet
- Pressure relief valve-activated HEPA filter inlet.

The above trips require immediate bypass of the mercury adsorbers to allow purging with nitrogen to prevent condensation or support fire suppression. The rest of the IWTU would be purged and cooled slowly to prevent thermal shock and possible failure of the equipment.

The two bypass valves have a nitrogen purge between them to prevent off-gas from leaking past the valves. When the mercury adsorbers are bypassed, a nitrogen purge of the adsorbers is activated. The mercury adsorbers are designed and manufactured with AL6XN material or equivalent. The mercury adsorbers are safety significant for the confinement of mercury.

Each mercury adsorber vessel is approximately 12 ft in diameter and 15.5 ft in height and has a minimum bed depth of 7.5 ft. The design flow for each of the adsorbers is 5,430 ACFM normal and 6,788 ACFM maximum. The mercury adsorbers are designed with 48-ft-per-minute face velocity and an 8.1-second gas residence time at normal flow conditions. At maximum design flow conditions, the face velocity increases to 60 ft per minute, with 6.5 seconds of residence time in each bed.

### 3.2.9 Process Exhaust Blowers

The process exhaust blowers discharge the treated off-gas to the atmosphere through the air mixing box and stack while being monitored via the off-gas monitoring systems (CEMS and radiation monitoring system). Each blower is designed for 8,334 ACFM, with a design static differential pressure of 2.65 psi. The normal operating inlet volume is 5,890 ACFM, with a suction pressure of 10.60 psia and discharge to atmosphere via the facility stack (pressure of 12.60 psia). One blower will operate at 100% capacity at any one time. The installed spare can easily be placed on-line in the situation that the operating blower must be shut down for maintenance or in an emergency situation. The blowers operate under normal operating temperature of 135°C and design maximum inlet temperature of 160°C.

### 3.2.10 Air Mixing Box

The air mixing box is designed to blend the process exhaust and building ventilation exhaust streams, then exhausts to the stack. The air mixing box is 8 ft 8 in. × 8 ft 8 in. × 16 ft and includes a mixing section. The air mixing box provides the volume needed to contain the air exhausted from the two systems and supplies the greatest level of mixing in the shortest distance. The air mixing box is constructed of carbon steel and operates at atmospheric pressure and a normal operating temperature (combined process exhaust and building HVAC exhaust) of 63°C, with a design maximum temperature of 175°C.

### 3.2.11 Stack

The gas discharge from the SBW process exhausts to the atmosphere through the stack, STK-SRE-140. The stack is 96 in. in diameter at the base, 60 in. in diameter at the discharge point, and 120 ft high. A gravity drain sump is provided at the base of the stack to collect condensate that may form in the stack. The stack and internal support elements are constructed of carbon steel.

## 3.3 Process Monitoring, Control, and Operation

The IWTU system is equipped with instrumentation to monitor and control process flows, temperatures, and pressures, and to transmit signals to the distributed control system (DCS). The DCS can control valves, motors, pumps, and fans, as well as alarms and initiate waste feed cutoff interlocks if process conditions deviate from established limits.

Table 3-1 summarizes the major compliance-related process instrumentation associated with the IWTU system and monitored on the DCS.

### 3.3.1 Process Off-gas Continuous Monitoring

The discharge gas from the process exhaust blowers is continuously monitored with a CEMS. The CEMS consists of gas analyzers, gas sampling system, and monitors (temperature, pressure, and flow) that are integrated with a data acquisition system. The CEMS unit includes redundant monitors that measure carbon monoxide and oxygen. (Note that the oxygen monitor is used to correct the carbon monoxide value to 7% oxygen.) Additionally, the CEMS measures the exhaust gas temperature, pressure, and flow rate for maintaining adequate sampling conditions. Certification and performance test ports are located 90 degrees apart in the same plane, in the process off-gas line.

Table 3-1. IWTU compliance monitoring instrumentation.

| Parameter | Monitoring Device | Units | Instrument ID | Manufacturer | Span | Accuracy | Cal. Interval |
|---|---|---|---|---|---|---|---|
| Waste feed rate | Flow meter | gal/min | FC-C-140-1[a]<br>FC-C-140-2[a]<br>FC-C-140-3[a] | Yokogawa | 0–3<br>0–3<br>0–3 | 0.1% of measured value | Annual |
| DMR bed temperature (7 of 13 instruments must be in service to operate) | Thermocouple | °C | TC-C-140-9[b] | Yokogawa | 0–800 | ±0.1% of full scale | Annual |
| CRR bed temperature (3 of 6 instruments must be in service to operate) | Thermocouple | °C | TC-C-160-4[b] | Yokogawa | 0–1,200 | ±0.1% of full scale | Annual |
| DMR pressure[c] | Pressure transmitter | psig | PIT-SRC-140-001[d] | Yokogawa | 0–15 | ±0.4% of full scale | Annual |
| CRR pressure[c] | Pressure transmitter | In. w.c. | PIT-SRC-160-023[d] | Yokogawa | -100–40 | ± 0.4% of full scale | Annual |
| DMR hydrogen concentration | Gas analyzer | vol%, dry | AY-C-153-1A, B, C[e] | Yokagowa/ Measurementation | 0–10% | ±1.0% of full scale | Annual |
| CRR oxygen concentration[c] | Gas analyzer | vol%, wet | AC-C-760-001 A, B, C[f] | Teledyne | 0–12% | ±1.0% of measured value | Annual |
| Off-gas cooler temperature[c] | Thermocouple | °C | TIT-SRC-160-001 A/B[g] | Yokogawa | 0–250 | ±0.1% of full scale | Annual |
| DMR superficial gas velocity | Fluidizing gas | Ft/sec | FY-B-365-1[h] | Distributed Control System Calculation | NA | NA | NA |
| HEPA filter pressure differential | Pressure transmitter | In. w.c. | See Note i | Yokogawa | 0–10 | ±0.4% of full scale | Annual |
| Process off-gas carbon monoxide | Gas analyzer | ppmv | AY-SRH-240-007A & 7B & 7C | ThermoScientific/ Centek | 0–200 | ±1.0% of full scale | Annual |
| Process off-gas oxygen | Gas analyzer | vol%, dry | A-H-941-2A & B[j] | AMETEK/Centek | 0–25% | ±0.75% of reading | Annual |

a. Instantaneous total waste feed through each nozzle.
b. This is an average of the values for the thermocouples that are functioning properly. If a thermocouple is malfunctioning, its value is dropped out of the average. There are 13 thermocouples on the DMR and six on the CRR. If less than seven of the DMR thermocouples are functioning properly, an alarm is activated and a normal shutdown is performed. If less than four of the CRR thermocouples are functioning properly, an automatic shutdown is performed.
c. One instrument must be in service to operate, although they can be temporarily taken out of service to calibrate or purge/clean without shutting down the process.
d. The DMR and CRR free board pressure are measured.
e. Three hydrogen sensors are used to calculate the DMR average hydrogen content on a dry basis. If all three fail and if at least one cannot be made functional in a specified amount of time, a normal shutdown will be performed.
f. Three oxygen sensors are used to calculate the CRR average oxygen content. If all three fail and if at least one cannot be made functional in a specified amount of time, a normal shutdown will be performed.
g. There are two temperatures. A is designated the primary, and B is the secondary.
h. Sum velocity of all fluidizing gas streams.
i. There are eight delta pressures across the process HEPA filters, PDIT-SRH-140-005A, 5B, 5C, 5D, 6A, 6B, 6C, 6D.
j. Average of valid $O_2$ sensors. Valid sensors are automatically selected. Operator may manually exclude a sensor from calculation.

During the SPT only, the process off-gas will be monitored to determine the total hydrocarbon concentration. The process off-gas also will be sampled or monitored for carbon dioxide and oxygen concentration so that the gas molecular weight can be determined for use in calculating the gas flow rate and the isokinetic conditions for the sampling trains. Moisture content determination is included with each isokinetic sampling train run.

### 3.3.2 Automatic Waste Feed Cutoffs

The IWTU includes an AWFCO system. If any one of several conditions is not met, the waste feed is cut off. Waste feed cutoff events are described in Table 7-2. A test of the AWFCO system will be included in the SPT (see Section 5.6).

### 3.3.3 Startup and Shutdown Procedures

Detailed startup and shutdown procedures are being developed and will be maintained as part of the system operating instructions.

***3.3.3.1 Rapidly Stopping Waste Feeds.*** The IWTU is controlled by a process control computer. Alarms and waste feed cutoff interlock set points are included, which will automatically stop waste feeds before any limits are reached. If any of these preprogrammed operating set points are reached, the computer will take automatic action to immediately stop waste feed. The same action to cease waste feed can be activated from the control room by operating personnel.

***3.3.3.2 Shutting Down the System.*** Shutdowns may occur for two reasons:

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

Critical automation equipment or instrumentation failures will result in automatic stoppage of waste feed and partial or complete system shutdown, depending on the severity of the failure or malfunction. Operations personnel are able to initiate a system shutdown manually from the control room. Standard operating procedures are maintained, including those for normal shutdown of the system.

***3.3.3.3 Controlling Emissions During Equipment Malfunctions.*** Process gases are directed through the emissions control equipment during normal operations. If an equipment malfunction affects system performance, waste feed may be automatically discontinued. Because all waste feed is injected into the DMR, stopping the waste feed eliminates the potential for uncontrolled emissions. To the greatest extent possible, the emissions control equipment will continue to operate while a malfunction is corrected. Waste feed may be resumed once the process operating conditions have been returned within the permit limits. If the malfunction cannot be corrected in a reasonable time or requires the unit to be taken off line, the system will be shut down in an orderly fashion according to operating procedures. Waste feed will not resume until the malfunction has been corrected and the system has been returned to operating conditions within the permitted limits.

# 4. TEST DESIGN AND PROTOCOL

## 4.1 Regulatory Requirements

This SPT is being conducted to demonstrate compliance with the applicable requirements of HWMA/RCRA regulations as they apply to miscellaneous treatment units under HWMA/RCRA Subpart X.

## 4.2 Test Objectives and Approach

This SPT plan has been prepared to describe the testing of the IWTU system that will be conducted to demonstrate compliance with the applicable environmental regulations and to gather data for use in validating the existing IWTU Risk Assessments. The objectives of the SPT are to gather emissions data, while operating at "bounding" conditions processing mixed waste. Conducting the SPT on mixed waste will establish operating conditions that will be included in the HWMA/RCRA permit. While 40 CFR 63, Subpart EEE ("National Emission Standards for Hazardous Air Pollutants from Hazardous Waste Combustors") does not strictly apply, these standards are believed to be the most appropriate for a thermal treatment device such as the IWTU, and have, thus, been established as performance targets. More specifically, the objectives of the SPT are as follows:

1. Evaluate emissions performance against the following targets:
   - Particulate emission concentration less than or equal to 0.0016 grains per dry standard cubic foot (gr/dscf), corrected to 7% oxygen
   - Emission concentration of dioxins and furans not exceeding 0.11 ηg/dscm, expressed as toxic equivalents of 2,3,7,8- tetrachloro dibenzo-p-dioxin (TCDD) toxicity equivalent (rated to 2,3,7,8-TCDD), corrected to 7% oxygen
   - Mercury emission concentration less than or equal to 8.1 μg/dscm, corrected to 7% oxygen
   - Emission concentration of semivolatile metals (cadmium and lead, combined) less than or equal to 10 μg/dscm, corrected to 7% oxygen
   - Emission concentration of low volatility metals (arsenic, beryllium, and chromium, combined) less than or equal to 23 μg/dscm, corrected to 7% oxygen
   - Carbon monoxide emissions concentration less than or equal to 100 ppmv, dry basis, hourly rolling average, corrected to 7% oxygen
   - Hydrocarbon emission concentration of less than or equal to 10 ppmv, as propane, dry basis, hourly rolling average, corrected to 7% oxygen.

   While the IWTU is not a combustion unit, an oxygen correction of emission concentrations has been included because the IWTU off-gas oxygen concentration is expected to vary in a manner similar to some combustion processes.
2. Establish permit operating limits:
   - Demonstrate maximum waste feed rate
   - Demonstrate maximum process gas flow rate
   - Establish appropriate operating limits for the process and off-gas treatment system components.

3. Gather information for evaluating risk:
   - Measure emissions of specific volatile and semivolatile organic emission products, plus tentatively identified volatile and semivolatile organics (tentatively identified compounds [TICs])
   - Measure emissions of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDF).
   - Measure emissions of representative volatile, semivolatile, and nonvolatile metals.
4. Demonstrate nitrogen oxide emissions performance for air permit compliance.

### 4.2.1 Bounding Conditions

With the multiple goals of evaluating emissions performance, establishing operating limits, and gathering risk evaluation data, it is important to ensure that the selected test conditions and sampling and analytical activities are appropriate for achieving those goals.

The applicable regulations specifically require establishing certain operating limits as a means of demonstrating continuing compliance with associated emission and performance standards. As such, it is appropriate to conduct the SPT under "bounding" conditions for those operating parameters that are associated with the emission standards being used as targets. The following sections briefly describe the test development approach used to ensure that bounding conditions are addressed.

***4.2.1.1 Organic Destruction/Removal.*** Organic destruction/removal occurs primarily in the high temperature environment of the DMR and CRR. Operating parameters related to organic destruction/removal are maximum waste feed rate, minimum temperature, and minimum gas residence time (maximum process gas flow rate). Thus, operating under conditions of maximum total waste feed rate, minimum DMR and CRR temperature, and maximum process gas flow rate would be considered as bounding for organic destruction.

Similar to establishing bounding conditions for organic destruction/removal, these same conditions are expected to be bounding for formation and potential emission of dioxins and furans and other thermal treatment by-products. As a miscellaneous unit, the purpose of the SPT is to demonstrate that the emissions from the unit do not pose an unacceptable risk to human health and the environment. While the mixed waste to be processed does carry the F001, F002 and F005 waste codes, the concentrations of organic material in the waste stream are extremely low. Spiking of the actual waste feed above the organic levels within the waste feed changes the nature of the waste feed and it provides waste solution that is not representative of the actual waste. Loading organic materials on the system may also influence thermal treatment by-product generation. Therefore, comparison of emissions with acceptable risk concentrations is a more appropriate evaluation for this unit.

In conversations with DEQ, it has been determined that adding organics to the waste, solely for the purpose of demonstrating destruction and removal, is not representative of the mixed wastes to be fed to the system under normal operations, and therefore is not desirable for this test. Thus, the approach to organic destruction and emissions control is to evaluate the emissions of organics during the test and to compare those emissions to established risk levels. EDF-9483 describing this approach is provided in Appendix D.

***4.2.1.2 Chlorine/Chloride.*** Potential chlorine emissions from the IWTU process have been reviewed and documented in RPT-354 Risk Assessment for Air Emissions from the Integrated Waste Treatment Unit and INTEC Liquid Waste Management System (April 2007) and in EDF-6495 Mass and Energy Balance for Sodium Bearing Waste Integrated Waste Treatment Unit – Modified to Support Emissions Permitting (February 2009). The results of the evaluation determined that if 100% of the chlorine in the liquid feed was emitted out the stack (no credit for the process or any emissions controls) that the worst case scenario led to a maximum emission rate of 1.28 lb/hr. This value was over the IDAPA 58.01.01 Emission Limit (EL) of 0.2 lb/hr so this was modeled (AERMOD) per State of Idaho Air Quality Modeling Guidance AQ-011, Rev 1. The resultant calculated ambient concentration was 4.63 E-01 $\mu g/m^3$ which is well below the acceptable ambient concentration of 150 $\mu g/m^3$. Based upon the information contained in these reports a worst case analysis (EDF-10146, September 2011) was also conducted to determine what the maximum chlorine emission would be if 100% solids were processed. Under this scenario (again with no credit for the process or any emissions controls) it was determined that the ambient concentration would be 3.86 $\mu g/m^3$.

Lastly, although the IWTU is not an incinerator, the maximum expected emission rate (9% chlorine in the waste is emitted) was compared to the chlorine performance standard in 40 CFR 264.343 and determined to be below the 4 lb/hr limit for both the normal liquid processing and the worst case processing of 100% solids.

***4.2.1.3 Risk Evaluation Data.*** Data are being gathered for use in a risk evaluation that will examine the long-term risks posed by emissions from the facility. As discussed in Section 4.2.1.1, EDF-9483 has been developed to describe the risk evaluation approach, and is presented in Appendix D. Collecting risk evaluation emissions data during the same bounding test conditions used for emissions performance evaluation will yield conservative emissions data and will thus avoid the potential for "second guessing" later. As such, the following elements have been incorporated into the test plan.

Mixed wastes will be used so that a typical emission profile will be present during the test. As discussed in Section 4.2.1.1, bounding conditions for organic destruction/removal, formation of thermal treatment by-products, and off-gas treatment component operations will be used during the test. These conditions, plus the maximum waste feed rate, during the test have been established such that potential emissions of organic compounds and metals are maximized.

## 4.3 Test Protocol

The proposed SPT is comprised of two test conditions, each with four replicate sampling runs. (Three sampling runs are required by the regulations. The fourth run has been added as a contingency.) During the test, the IWTU will be operated at maximum waste feed rate, maximum process gas flow rate, minimum DMR and CRR temperatures, and bounding conditions for the remainder of the process components. The two test conditions are identical, with the exception of the target DMR temperature. The first test condition will target a minimum DMR temperature that is expected to provide compliant performance with a very high degree of confidence. The second test condition will target a somewhat lower DMR temperature, which would provide more operating flexibility for the system, if appropriate performance is demonstrated in this condition.

Table 4-1 summarizes the operational and sampling protocol for the test.

Table 4-1. System Performance Test protocol summary.

| Process Parameter | Objective |
|---|---|
| Total waste feed rate | Maximum |
| DMR and CRR pressure | Normal |
| DMR bed temperature | Minimum in both test conditions, but the target in Condition 2 will be somewhat more aggressive (lower) than in Condition 1 |
| CRR bed temperature | Minimum |
| DMR superficial fluidizing gas velocity | Maximum |
| DMR off-gas hydrogen concentration | Minimum |
| CRR off-gas oxygen concentration | Minimum |
| Off-gas cooler outlet temperature | Maximum |
| Off-gas cooler outlet temperature | Minimum |
| HEPA filter inlet temperature | Normal |
| HEPA filter pressure differential | Normal |
| Mercury adsorber inlet temperature | Normal |
| **Sample** | **Description** |
| Gaseous emissions for particulate matter, hydrogen chloride, and chlorine | EPA Method 26A during each run |
| Gaseous emissions for speciated volatile organic compounds and TICs | SW-846 Method 0031 during each run |
| Gaseous emissions for speciated semivolatile organics and TICs | SW-846 Method 0010 during each run |
| Gaseous emissions for PCDD/PCDF | SW-846 Method 0023A during each run |
| Gaseous emissions for polynuclear aromatic hydrocarbons | SW-846 Method 0010 during each run |
| Gaseous emissions for multiple metals | EPA Method 29 during each run |
| Gaseous emissions for carbon monoxide and oxygen | Installed CEMS during each run |
| Gaseous emissions for total hydrocarbons, nitrogen oxides, carbon monoxide, and oxygen | Temporary CEMS during each run |

**NOTE:** *The temporary CEMS carbon monoxide and oxygen sample are the reference methods used to perform the relative accuracy test audit for the installed CEMS. These data are collected for use in determining volumetric flow calculations.*

## 4.4 Feed Characteristics

### 4.4.1 Description and Composition of Feed Streams

The SPT will be conducted on actual mixed waste. The concentration of radionuclides in the waste is less than 160 mg/liter. This small mass is insignificant from a chemical perspective, therefore, it would not change the operating parameters or the chemical composition of the off-gas and solid treatment product coming from the system.

### 4.4.2 Metals Content

Metals are classified by volatility class. Three classes are defined: (1) volatile metals (mercury), (2) semivolatile metals (cadmium and lead), and (3) low volatility metals (arsenic, beryllium, and chromium).

Feed rates during the SPT have been determined to represent upper bound conditions based on expected concentrations in the actual wastes.

## 4.5 Operating Conditions

Planned process operating conditions and feed rates are targeted such that the anticipated permit operating limits (Section 7) will be achieved. Table 4-2 presents the target process operating conditions for the test.

Table 4-2. System Performance Test target operating conditions.

| Parameter | Test 1 Target | Test 2 Target |
|---|---|---|
| Total waste feed rate (gal/min) | 3.5 | 3.5 |
| DMR pressure (psig) | <12 | <12 |
| CRR pressure (in. w.c.) | <0 | <0 |
| DMR off-gas temperature (°C) | 640 | 605 |
| CRR off-gas temperature (°C) | 875 | 875 |
| DMR superficial gas velocity (ft/sec) | 1.3 | 1.3 |
| DMR off-gas hydrogen concentration (vol%, dry) | 3.0 | 3.0 |
| CRR off-gas oxygen concentration (vol%, wet) | 2 | 2 |
| Off-gas cooler outlet temperature (°C) | <250 | <250 |
| HEPA filter inlet temperature (°C) | <250 | <250 |
| HEPA filter pressure differential (in. w.c.) | <5 | <5 |
| Mercury adsorber inlet temperature (°C) | <180 | <180 |
| Process off-gas carbon monoxide (ppmv, dry) | <100 | <100 |
| Process off-gas total hydrocarbons (ppmv as propane, dry) | <10 | <10 |

### 4.5.1 Preconditioning, Steady-State, and Interruptions in Sampling

The SPT will be conducted under stable conditions at the target process parameters to the extent possible. The steady-state conditions will be determined during startup testing and will be bounded by these tests. Because the testing is designed to stress the system, variations from the targets can be expected, both over time during a run and from run to run at the same condition. Targets may be modified during testing as dictated by process conditions, within the envelope of the SPT operating limits. DOE and DEQ will be notified and agreement obtained prior to any changes to operating targets during the SPT.

The IWTU will be operated on mixed waste, and the process parameters will be established at the target values (as allowed by process conditions) prior to testing in order to establish steady process operations. Once this has been achieved (based on process operator judgment), the system will be "declared" to be at steady-state, and emissions sampling will begin. A guideline for determining the achievement of steady-state conditions will be when temperatures and flows are being controlled to within ±5% of the set point for at least 30 minutes. The actual acceptable process stability value will be discussed and agreed to by the SPT Team (see Section 1.7) prior to conducting the SPT.

If an AWFCO or other process problem occurs, or if an emissions sampling problem occurs, all or part of the sampling activities may be temporarily halted until the problem is resolved. If mixed waste feed is halted, all sampling will be stopped until the problem is corrected and the feed is restored. If the feed interruption is less than 15 minutes, sampling will resume immediately. If the interruption is between 15 and 30 minutes, a 15-minute preconditioning period will be required prior to restarting sampling. If the interruption is greater than 30 minutes, then the process operators will re-establish steady-state conditions as described in the previous paragraph before resuming sampling activities. Any deviation from these procedures will be discussed with DEQ personnel on-site prior to taking action. Following resolution of the problem, sampling will be resumed, and the run will be completed. In some cases, an extended period may be required to resolve the problem, or the test interruption may have compromised the integrity or quality of the samples. In these cases, the on-site DEQ representative(s) will be consulted, and a decision may be made to abort the run. Table 4-3 presents guidelines to be used in troubleshooting and in making decisions to restart or abort an interrupted run.

Table 4-3. System Performance Test troubleshooting guidance.

| Condition | Resolution Guidance |
|---|---|
| Process conditions cannot be maintained within the specifications of the test plan | Document which parameters need to be modified. If modified conditions will be more conservative (for example, minimum temperature higher than originally planned, waste feed rate less than originally planned), these modifications should be acceptable. The operator must understand, however, that certain permit limits will be based on actual test conditions. If modified conditions will be less conservative than specified in the approved test plan, specific DEQ approval will be needed. In all cases, the on-site DEQ observers should be notified of process operating changes. |

Table 4-3. (continued).

| Condition | Resolution Guidance |
|---|---|
| Run start/restart | Prior to starting a run, ensure that the process has attained the desired operating conditions and has remained at those conditions (within reasonable bounds of normal process variability) as defined in the declaration of steady-state (see Section 4.5.1). If a run is stopped due to process deviations, allow the process to operate at desired conditions for at least 15 minutes before restarting the run. If the interruption lasted between 15 to 30 minutes, allow the process to run at the desired conditions for 30 minutes prior to restarting the run. If the run is interrupted for more than 30 minutes, re-establish steady-state as described in Section 4.5.1 before restarting the run. In most cases, waste feed systems should be operated throughout process upsets. If waste feed systems have been interrupted, allow these systems to operate at test conditions for at least 30 minutes or for the time required to achieve steady-state prior to restarting the run. |
| Process conditions drift within or between runs | A certain amount of process variability is normal, and will occur due to variability in the waste feeds, ambient conditions, operator experience, and process control tolerances. The process should be operated as closely as possible to the target test conditions for each run, and all runs within a given test condition should be conducted at similar conditions so the results of each run within a test condition can be compared. Normal process variability within a run or between runs is site-specific and must be evaluated on a case-by-case basis. Process variability for temperatures and flows in this system are expected to be ±5% of the set point value. The actual acceptable process stability value will be judged by the SPT Team, and will be recorded as appropriate in the test log. |
| Minor process upset | If the process experiences short-term, minor process upsets that do not result in a waste feed cutoff, the sampling should continue through the event, and a notation should be made in the test manager's log. If the process conditions drift beyond what the process operations personnel deem to be normal, and cannot be quickly corrected, or if conditions between runs do not meet the process operations personnel's satisfaction, the testing should be halted until appropriate process conditions are established. An on-going run can normally be resumed after process conditions are corrected (see "Resumption of testing" for additional guidance). At the discretion of the facility operator, the run may be aborted and repeated at any time. |
| Major process upset | If the process deviates from the desired conditions to the extent that a waste feed cutoff occurs, all testing activities will be halted. Testing may be resumed after the interlock is cleared and the process is stabilized at the test conditions (see "Resumption of testing" for additional guidance). If two or more major process upsets occur within a run, consideration should be given to aborting and repeating the run. At the discretion of the facility operator, a run may be aborted and repeated at any time. |

Table 4-3. (continued).

| Condition | Resolution Guidance |
|---|---|
| Minor sampling system problem | If a minor problem occurs in an individual sampling system (for example, filter changeout, full impinger, power problem, temperature problem), the affected sampling system will be temporarily halted until the problem is corrected. All other sampling systems will continue as normal. The run may need to be extended to allow for collection of the appropriate sample volume in the affected sampling system. |
| Major sampling system problem | If a major problem occurs in an individual sampling system (for example, broken glassware, failure of control module), or if a problem impacts several sampling systems (for example, power outage) all sampling will be halted until the problem is diagnosed and an appropriate course of action is determined. If it appears that the problem can be corrected within approximately 60 minutes, all sampling will be suspended until the problem is corrected, and then sampling will resume. If it appears that more than 60 minutes will be needed to correct the problem, the entire sampling program may be suspended until the problem is corrected, or the unaffected systems may resume sampling, and the affected system will be resumed after the problem is corrected. It may be necessary to extend the run to allow for collection of the appropriate sample volume in the affected sampling system(s). (Note: It is not critical that all sampling is conducted concurrently as long as process operating conditions are consistent during the entire run.) |
| Temporary halt of sampling | If a temporary halt of sampling is required, the test manager will communicate to all parties that sampling is to be placed "on hold." Each sampler will note the event and the time on the sample collection sheet, and the test manager will note the event and the time in the test log. Emissions sampling trains will be stopped, but will be kept in place unless the halt exceeds (or is expected to exceed) about 15 minutes. If the temporary halt exceeds about 15 minutes, emissions sampling train probes will be removed from the duct, and the nozzle openings will be capped. Sampling system heating and cooling (as appropriate to each train) will be continued. Sample trains that have been removed from the stack will be leak-checked before resuming sampling. Process samples will be secured at the sampling location until the test is resumed. |

Table 4-3. (continued).

| Condition | Resolution Guidance |
|---|---|
| Resumption of testing after a temporary halt | Once the condition that caused a temporary halt to sampling has been resolved, the test manager (in consultation with the SPT Team) may either abort the run or may resume it using the following guidance and site-specific considerations. In all cases, the decision to abort or resume will be documented in the test log. If sampling is resumed, each sampler will note the time of sample resumption on the sample collection sheets, and the test manager will note the time in the test log.<br><br>In general, a run can be resumed if it can be completed within the same test period. The samples will not be held overnight and resumed the following day.<br><br>If the process downtime during a run exceeds 3 hours, the run should be aborted unless sound technical justification is provided and all parties agree.<br><br>If the reason for the sample interruption was a major process upset, or if the temporary halt exceeds 1 hour, the volatile organic sample resin tube set that was being collected at the time of the test interruption should be discarded, and the test resumed with a fresh tube set. Note that this does not apply to the resin in a semivolatile organic or PCDD/PCDF sampling train. |
| Disposition of samples from aborted run | A sample run is aborted because it is deemed in the field that the samples will not be suitable for their intended purpose. In general, samples from an aborted run are discarded, and the run is repeated in its entirety, using the original test run identification. There may be rare circumstances where the samples are recovered and archived for possible analysis. In this case, the samples from the aborted run are assigned their original sample identifications, and the repeat run is assigned a new run identification. |
| Sample lost during recovery operations | If, after completing a run, a sample is broken, spilled, contaminated, or otherwise lost, the SPT Team must decide on a corrective action to replace the sample. In this case, the additional fourth test run samples will be used for the replacement sample. For example, if a test run includes emissions sampling for organics, particulate, and metals, and the metals train is dropped and broken during removal from the sampling location, the additional run sample for the metals train will be used.<br><br>If a partial loss of sample occurs, the analytical laboratory will be consulted to determine if a smaller sample size is sufficient. In such instances, the recovered portion of the sample may be submitted for analysis with DEQ observer concurrence. |
| Sample cannot be collected as specified in the approved test plan | This situation can arise due to unique features and circumstances of the process or logistics complications. Appropriate modifications must be discussed and agreed among the SPT Team on a case-by-case basis. The test manager will describe the situation and resolution in the test log. |

Table 4-3. (continued).

| Condition | Resolution Guidance |
|---|---|
| Sampling train fails leak check at port change or at test completion (isokinetic samples), or at tube set completion (volatile organic samples) | Leak checks on isokinetic sampling trains should be conducted before the run, at each port change, and at run completion. Allowable leakage rate for isokinetic sampling trains is 0.02 cfm at a vacuum equivalent to or higher than the sampling condition. Volatile organic sampling trains are leak-checked before and after each tube set. Allowable leakage rate for M0031 is 0.02 liters per minute for sampling rates of 1 liter per minute, and 0.01 liters per minute for sampling at lower rates.<br><br>Leaks found before sampling will be corrected before sampling begins.<br><br>If a volatile organic sample fails the leak check at the completion of a tube set, the tube set will be discarded, and an additional set will be collected during the run as a replacement. The run time may need to be extended to allow for collection of the appropriate sample volume and number of tube sets in the affected sampling system(s).<br><br>If an isokinetic sampling train fails a leak check, the sampling team should isolate the train sections and determine where the leak occurred. In some cases, it also may be possible to determine when the leak problem occurred. If it can be determined that the leak occurred during the port change or after sampling was completed, the test will be considered valid, with concurrence of the DEQ observer. In other instances, if the leak is small (less than twice the allowable leakage rate) and occurred at a location inside the duct (probe nozzle assembly), keeping the sample may be justified, with concurrence from the DEQ observer. In-duct leakage can be confirmed by comparing the collected moisture content from the train in question to other trains from the same run or from other runs within the same test condition. If it is decided to keep the sample, the portion of the run during which the leak occurred should be corrected for the leak (see procedure in EPA Method 5) even though the leak appeared to have been inside the duct, and the isokinetic sampling rate should be confirmed with the sample volume correction. This procedure is conservative because it yields a smaller sample volume than was actually collected. Leaks larger than 0.02 cfm that are found to have been outside the duct will result in rejection of the sample unless sound technical justification is provided and all parties agree. |

# 5. SAMPLING, ANALYSIS, AND MONITORING PROCEDURES

The following paragraphs discuss the planned sampling and analyses. The sampling procedures to be used during the SPT are listed in Appendix A. Analytical methods are described in Appendix B.

## 5.1 Sampling and Sample Recovery Locations and Procedures

Sample collection locations, equipment, methods, and frequency are summarized in Table 5-1. The types of analyses planned for the SPT samples are provided in Table 5-2. Analytical methods and procedures to be used for each sample are summarized in Table 5-3. Sample recovery will occur in the unused Canister Loading Station cell (Room 114), the truck bay (Room 107), or a laboratory trailer that the sampling contractor may bring on site.

### 5.1.1 Waste Feed

The feed material will be mixed waste and its composition is known from previous characterization efforts. No additional sampling will be conducted on the mixed waste feed.

### 5.1.2 Process Effluent Streams

The only effluents from the IWTU are the solid granular product and the gaseous emissions. Sampling methods are presented in Appendix A and analytical methods are presented in Appendix B. As discussed in Section 2, the composition of the solid granular product will be known.

### 5.1.3 Gaseous Emissions

***5.1.3.1 Semivolatile Organics and Polynuclear Aromatic Hydrocarbons.*** The gaseous emissions will be sampled using an SW-846 Method 0010 sampling train to measure the emissions of semivolatile organics and polynuclear aromatic hydrocarbons (PAHs). The extract from this train will be analyzed using SW-846 Method 8270 to determine the emissions of target semivolatile organic compounds (SVOCs), target PAHs, and semivolatile TICs. The sampling method is described in Appendix A.

The target SVOC analytes for the Method 0010 samples are presented in Table 5-4. The target polynuclear aromatic hydrocarbon analytes are presented in Table 5-5. In addition to the SVOC target analytes, analyses will identify nontarget analyte peaks (TICs) based on the nearest internal standard and analytical instrument library.

The sampling train preparation and analytical methods are described in Appendix B.

Table 5-1. Sample collection locations, equipment, and methods.

| Location[a] | Sample Name/ Number | Access | Equipment | Sample Size | General Procedure/Frequency | Ref. Method[b] |
|---|---|---|---|---|---|---|
| Process off-gas | M26A | Fifth set of ports in horizontal section of duct | EPA Method 26A particulate matter, hydrogen chloride and chlorine sampling train | As recommended in method guidance[c,d] | Collect integrated sample for particulate matter, hydrogen chloride, chlorine, and moisture during each run. Measure gas velocity, pressure, and temperature. Collect bag samples, or use continuous emission monitor (CEM) for molecular weight determination. | EPA Methods 1-5 and 26A |
| Process off-gas | M29 | First set of ports downstream of process exhaust blower in slanted portion of duct | EPA Method 29 multiple metals sampling train | Minimum 120 minutes[c,d] | Collect integrated sample for metals and moisture during each run. Measure gas velocity, pressure, and temperature. Collect bag samples, or use CEM for molecular weight determination. | EPA Methods 1-4 and 29 |
| Process off-gas | M0023A | Second set of ports downstream of process exhaust blower in slanted portion of duct | EPA Method 0023A sampling train | Minimum 3 dry standard cubic meters[c,d] (~4-hour run) | Collect integrated sample for PCDD/PCDF and moisture during each run. Measure gas velocity, pressure, and temperature. Collect bag samples, or use CEM for molecular weight determination. | EPA Methods 1-4 and 0023A |

Table 5-1. (continued).

| Location[a] | Sample Name/ Number | Access | Equipment | Sample Size | General Procedure/Frequency | Ref. Method[b] |
|---|---|---|---|---|---|---|
| Process off-gas | M0010 | Third set of ports downstream of process exhaust blowers in slanted portion of duct | EPA Method 0010 sampling train | Minimum 3 dry standard cubic meters[c,d] (~4-hour run) | Collect integrated sample for target SVOCs and SVOC TICs during each run of the test. | SW-846 Method 0010 |
| Process off-gas | M0031 | First set of ports downstream of process exhaust blower in slanted portion of duct | EPA Method 0031 sampling train | Minimum 120 minutes[c,d] | Collect integrated sample for VOCs and VOC TICs during each run of the test. | SW-846 Method 0031 |
| Process off-gas | Temporary CEMS | First vertical port in horizontal section of duct | Temporary CEMS total hydrocarbons, nitrogen oxide | Continuous | Continuously monitor total hydrocarbon and nitrogen oxide emissions during each run. | 40 CFR 60, Appendix B, Specification 2, 8A, EPA Method 25A and 7E |
| Process off-gas | CEMS | Second vertical port in horizontal section of duct | Installed CEMS carbon monoxide, oxygen | Continuous | Continuously monitor carbon monoxide emissions and oxygen concentration during each run. | 40 CFR 60 Appendix B, Specification 4B |

Table 5-1. (continued).

| Location[a] | Sample Name/ Number | Access | Equipment | Sample Size | General Procedure/Frequency | Ref. Method[b] |
|---|---|---|---|---|---|---|

a. Relative location in the process.
b. SW-846 = *Test Methods for Evaluating Solid Waste*, Third Edition, November 1986 and updates. EPA Method = *Test Methods and Procedures*, Appendix A, 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 gas molecular weight (EPA Method 3)
- Performing gas velocity, pressure, and temperature profile measurement for each sampling location (EPA Method 2)
- Determining the moisture content of the gas for each sampling train sample (EPA Method 4)

Table 5-2. Analyses planned for System Performance Test samples.

| Sample Name | Analyses | Rationale |
|---|---|---|
| Gaseous emissions M0010 | Target SVOCs, SVOC TICs, PAHs, oxygen, carbon dioxide, temperature, flow rate | Gather emissions data |
| Gaseous emissions M0023A | PCDD/PCDFs, oxygen, carbon dioxide, temperature, flow rate | Demonstrate performance and gather emissions data |
| Gaseous emissions M0031 | Target volatile organics and volatile organic TICs | Gather emissions data |
| Gaseous emissions M26A | Particulate, hydrogen chloride, chlorine, moisture, oxygen, carbon dioxide, temperature, flow rate | Demonstrate performance and gather emissions data |
| Gaseous emissions M29 | Multiple metals, moisture, oxygen, carbon dioxide, temperature, flow rate | Demonstrate performance and gather emissions data |
| Process off-gas CEMS | Carbon monoxide and oxygen (using installed CEMS). Total hydrocarbons, nitrogen oxides, carbon monoxide, and oxygen (using temporary CEMS) | Evaluate performance and gather emissions data |

Table 5-3. Summary of System Performance Test analytical procedures and methods.

| Sample Name | Analysis | Samples per Run | Total Field Samples[a] | Preparation Method[b] | Analytical Method[b] |
|---|---|---|---|---|---|
| Gaseous emissions (Method 0031) | Volatile organic compounds (VOCs) (tenax & anasorb tubes) | Footnote c | Footnote c | Thermal desorption, trap (SW846-5041A) | Gas chromatography/mass spectrometry (GC/MS) (SW846-5041A/8260B) |
| | VOCs (Cond.) | 1 | 8 | Purge and trap | GC/MS (SW846-8260B) |
| Gaseous emissions (Method 0010) | SVOCs, PAHs | 1 | 8 | Solvent extraction (SW846-3542) | GC/MS (SW846-8270C) |
| | Moisture | 1 | 8 | NA | Gravimetric (EPA Method 4) |
| | Temperature | 1 | 8 | NA | Thermocouple (EPA Method 2) |
| | Velocity | NA | NA | NA | Pitot tube (EPA Method 2) |
| | Oxygen, carbon dioxide | Footnote d | Footnote d | NA | Orsat or CEM (EPA Method 3) |
| Gaseous emissions (Method 0023A) | PCDD/PDCFs | 1 | 8 | Solvent extraction (SW846-0023A) | GC/MS (SW846-8290) |
| | Moisture | 1 | 8 | NA | Gravimetric (EPA Method 4) |
| | Temperature | 1 | 8 | NA | Thermocouple (EPA Method 2) |
| | Velocity | NA | NA | NA | Pitot tube (EPA Method 2) |
| | Oxygen, carbon dioxide | Footnote d | Footnote d | NA | Orsat or CEM (EPA Method 3) |
| Gaseous emissions (Method 26A) | Particulate matter | 1 | 8 | Desiccation (EPA Method 5) | Gravimetric (EPA Method 5) |
| | Hydrogen chloride/chlorine | 2 | 16 | NA | ICP (SW846-9057) |
| | Moisture | 1 | 8 | NA | Gravimetric (EPA Method 4) |
| | Temperature | 1 | 8 | NA | Thermocouple (EPA Method 2) |
| | Velocity | NA | NA | NA | Pitot tube (EPA Method 2) |

Table 5-3. (continued).

| Sample Name | Analysis | Samples per Run | Total Field Samples[a] | Preparation Method[b] | Analytical Method[b] |
|---|---|---|---|---|---|
| | Oxygen, carbon dioxide | Footnote d | Footnote d | NA | Orsat or CEM (EPA Method 3) |
| Gaseous emissions (Method 29) | Metals | 1 | 8 | Acid digestion (SW846-3015 & 3050)<br>CVAA (SW846-7470) | ICP (SW846-6010B)<br>CVAA (SW846-7470) |
| | Moisture | 1 | 8 | NA | Gravimetric (EPA Method 4) |
| | Temperature | 1 | 8 | NA | Thermocouple (EPA Method 2) |
| | Velocity | NA | NA | NA | Pitot tube (EPA Method 2) |
| | Oxygen, carbon dioxide | Footnote d | Footnote d | NA | Orsat or CEM (EPA Method 3) |
| Process off-gas temporary CEM | Total hydrocarbons, nitrogen oxides, carbon monoxide, oxygen | Footnote e | Footnote e | NA | Extractive analyzers, 40 CFR 60, Appendix B, Specification 2, 8A, EPA Methods 25A, 7E |
| Process off-gas installed CEMs | Carbon monoxide, oxygen | Footnote e | Footnote e | NA | Extractive analyzers, 40 CFR 60, Appendix B, Specification 4B |

a. Two test conditions will be conducted, each comprised of four runs. Sample count does not include QA/QC samples.

b. "SW846" refers to *Test Methods for Evaluating Solid Waste*, Third Edition, November 1986, and updates.
"EPA Methods" refers to "New Source Performance Standards, Test Methods and Procedures," App. A, 40 CFR 60.
"EPA Continuous Source Monitoring Manual," Revision 6, January 1996.

c. During each sampling run, four sets of sampling method for volatile organic compounds tubes (eight samples) will be collected, but only three sets (six samples) will be analyzed. The extra tube set provides a contingency in case of breakage or other event that could require analysis of the extra tube set. The first tube in each tube set will be analyzed separately from the last tube to assess breakthrough.

d. One set of gas bag samples collected during each traverse for Orsat analysis or CEM.

e. CEMS sampling and analysis is continuous during each run.

Table 5-4. Target semivolatile organic analytes.

| Semivolatile | CAS[a] Number |
|---|---|
| Acetophenone | 98-86-2 |
| Aniline | 62-53-3 |
| Benzaldehyde | 100-52-7 |
| Benzidine | 92-87-5 |
| Benzoic acid | 65-85-0 |
| Benzyl alcohol | 100-51-6 |
| bis(2-Chloroethoxy) methane | 111-91-1 |
| bis(2-Chloroethyl) ether | 111-44-4 |
| bis(2-ethylhexyl) phthalate | 117-81-7 |
| 4-Bromophenyl-phenylether | 101-55-3 |
| Butyl benzyl phthalate | 85-68-7 |
| Carbazole | 132-32-1 |
| 4-Chloro-3-methylphenol | 59-50-7 |
| 4-Chloroaniline | 106-47-8 |
| 2-Chlorophenol | 95-57-8 |
| 4-Chlorophenyl-phenylether | 7005-72-3 |
| Dibenzofuran | 132-64-9 |
| 1,2-Dichlorobenzene | 95-50-1 |
| 1,3-Dichlorobenzene | 541-73-1 |
| 1,4-Dichlorobenzene | 106-46-7 |
| 3,3'-Dichlorobenzidine | 91-94-1 |
| 2,4-Dichlorophenol | 120-83-2 |
| Diethyl phthalate | 84-66-2 |
| Dimethyl phthalate | 131-11-3 |
| 2,4-Dimethylphenol | 105-67-9 |
| Di-n-butyl phthalate | 84-74-2 |
| 4,6-Dinitro-2-methylphenol | 534-52-1 |
| 1,3-Dinitrobenzene | 99-65-0 |
| 2,4-Dinitrophenol | 51-28-5 |
| 2,4-Dinitrotoluene | 121-14-2 |
| 2,6-Dinitrotoluene | 606-20-2 |
| Di-n-octylphthalate | 117-84-0 |
| Diphenylamine | 122-39-4 |
| 1,2-Diphenylhydrazine | 122-66-7 |

Table 5-4. (continued)

| Semivolatile | CAS[a] Number |
|---|---|
| Hexachlorobenzene | 118-74-1 |
| Hexachlorobutadiene | 87-68-3 |
| Hexachlorocyclopentadiene | 77-47-4 |
| Hexachloroethane | 118-74-1 |
| Isophorone | 78-59-1 |
| 2-Methylphenol (o-Cresol) | 95-48-7 |
| 3 & 4-Methylphenol (m- & p-Cresol) | 65794-96-9 |
| 2-Nitroaniline | 88-74-4 |
| 3-Nitroaniline | 99-09-2 |
| 4-Nitroaniline | 100-01-6 |
| Nitrobenzene | 98-95-3 |
| 2-Nitrophenol | 88-75-5 |
| 4-Nitrophenol | 100-02-7 |
| N-Nitrosodimethylamine | 62-75-9 |
| N-Nitroso-di-n-propylamine | 621-64-7 |
| N-Nitrosodiphenylamine | 86-30-6 |
| 2,2-Oxybis (1-Chloropropane) | 108-60-1 |
| Pentachlorobenzene | 608-93-5 |
| Pentachloronitrobenzene | 82-68-8 |
| Pentachlorophenol | 87-86-5 |
| Phenol | 180-95-2 |
| Pyridine | 110-86-1 |
| 1,2,4-Trichlorobenzene | 120-82-1 |
| 2,4,5-Trichlorophenol | 95-95-4 |
| 2,4,6-Trichlorophenol | 88-06-2 |
| 1,2,4,5-Tetrachlorobenzene | 95-94-3 |

a. CAS = Chemical Abstract Service.

Table 5-5. Target polynuclear aromatic hydrocarbon analytes.

| PAH | CAS[a] Number |
|---|---|
| Acenaphthene | 83-32-9 |
| Acenaphthylene | 208-96-8 |
| Anthracene | 120-12-7 |
| Benzo(a)anthracene | 56-55-3 |
| Benzo(a)pyrene | 50-32-8 |
| Benzo(b)fluoranthene | 205-99-2 |
| Benzo(e)pyrene | 192-97-8 |
| Benzo(ghi)perylene | 191-24-2 |
| Benzo(k)fluoranthene | 207-08-9 |
| Chrysene | 218-01-9 |
| Dibenz(a,h)anthracene | 53-70-3 |
| Fluoranthene | 206-44-0 |
| Fluorene | 86-73-7 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 |
| 2-Methylnaphthlene | 91-57-6 |
| Naphthalene | 91-20-3 |
| Perylene | 198-55-0 |
| Phenanthrene | 85-01-8 |
| Pyrene | 129-00-0 |

a. CAS = Chemical Abstract Service.

***5.1.3.2 Volatile Organics.*** A sampling method for volatile organic compounds (VOCs) sampling train (SW-846 Method 0031) will be used during the test to determine the VOC emissions. Four sets of sorbent tubes will be collected during each sampling run, but only three will be analyzed. The fourth tube set will be an archive set in case of breakage during shipment or laboratory handling. Appendix A describes the sampling method for VOCs. The sampling method for VOCs tubes will be analyzed using SW-846 Method 8260.

These analyses will be used to assess the emissions of VOCs for use in the risk assessment. The target VOCs are listed in Table 5-6. Analyses will be performed using SW-846 Method 8260. In addition to the target analyte list, analyses will include identifying nontarget analyte peaks, TICs, based on the nearest internal standard and analytical instrument library search. Analysis methods are described in Appendix B.

Table 5-6. Target volatile organic analytes.

| Volatile | CAS[a] Number |
|---|---|
| Acetone | 67-64-1 |
| Acetonitrile | 75-05-8 |
| Acrylonitrile | 107-13-1 |
| Benzene | 71-43-2 |
| Bromobenzene | 108-86-1 |

Table 5-6. (continued).

| Volatile | CAS[a] Number |
|---|---|
| Bromochloromethane | 74-97-5 |
| Bromodichloromethane | 75-27-4 |
| Bromoform | 75-25-2 |
| Bromomethane | 74-83-9 |
| 2-Butanone (MEK) | 78-93-3 |
| n-Butylbenzene | 104-51-8 |
| sec-Butylbenzene | 135-98-8 |
| tert-Butylbenzene | 98-06-6 |
| Carbon disulfide | 75-15-0 |
| Carbon tetrachloride | 56-23-3 |
| Chlorobenzene | 108-90-7 |
| Chlorodibromomethane | 96-12-8 |
| Chloroethane | 75-00-3 |
| Chloroform | 67-66-3 |
| Chloromethane | 74-87-3 |
| 2-Chlorotoluene | 95-49-8 |
| 4-Chlorotoluene | 106-43-4 |
| 1,2-Dibromo-3-chloropropane | 96-12-8 |
| 1,2-Dibromoethane | 106-93-4 |
| Dibromomethane | 74-95-3 |
| 1,2-Dichlorobenzene | 95-50-1 |
| 1,3-Dichlorobenzene | 541-73-1 |
| 1,4-Dichlorobenzene | 106-46-7 |
| Dichlorodifluoromethane | 75-71-8 |
| 1,1-Dichloroethane | 75-34-3 |
| 1,2-Dichloroethane | 107-06-2 |
| 1,1-Dichloroethene | 75-35-4 |
| cis-1,2-Dichloroethene | 156-59-2 |
| trans-1,2-Dichloroethene | 156-60-5 |
| 1,2-Dichloropropane | 78-87-5 |
| 1,3-Dichloropropane | 142-28-9 |
| 2,2-Dichloropropane | 594-20-7 |
| 1,1-Dichloropropene | 563-58-6 |
| cis-1,3-Dichloropropene | 10061-01-5 |
| trans-1,3-Dichloropropene | 10061-02-6 |

Table 5-6. (continued).

| Volatile | CAS[a] Number |
|---|---|
| Ethylbenzene | 100-41-4 |
| Hexachlorobutadiene | 87-68-3 |
| 2-Hexanone | 591-78-6 |
| Iodomethane | 74-88-4 |
| Isopropylbenzene | 98-82-8 |
| p-Isopropyltoluene | 99-87-6 |
| Methylene chloride | 75-09-2 |
| 4-Methyl-2-pentanone (MIBK) | 108-10-1 |
| n-Propylbenzene | 103-65-1 |
| Styrene | 100-42-5 |
| 1,1,1,2-Tetrachloroethane | 630-20-6 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 |
| Tetrachloroethene | 127-18-4 |
| Toluene | 108-88-3 |
| 1,2,3-Trichlorobenzene | 87-61-6 |
| 1,2,4-Trichlorobenzene | 120-82-1 |
| Trichloroethane, 1,1,1- | 71-55-6 |
| Trichloroethane,1,1,2- | 79-00-5 |
| Trichloroethene | 79-01-6 |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | 76-13-1 |
| Trichlorofluoromethane | 75-69-4 |
| 1,2,3-Trichloropropane | 96-18-4 |
| 1,2,4-Trimethylbenzene | 95-63-6 |
| 1,3,5-Trimethylbenzene | 108-67-8 |
| Vinyl acetate | 108-05-4 |
| Vinyl chloride | 75-01-4 |
| m & p-Xylene | 179601-23-1 |
| o-Xylene | 95-47-6 |
| Xylenes (total) | 1330-20-7 |

a. CAS = Chemical Abstract Service.

***5.1.3.3 Dioxins and Furans.*** Emissions samples will be collected for PCDD/PCDF using an EPA Method 0023A sampling train. The sampling procedure is described in Appendix A. PCDD/PCDF is analyzed by high-resolution gas chromatography/mass spectrometry according to SW-846 Method 8290, described in Appendix B. PCDD/PCDF target analytes are shown in Table 5-7.

Table 5-7. Target polychlorinated dibenzo-p-dioxins and polychlorinated dibenzo furan analytes.

| Dioxin/Furan Compound | CAS[a] Number |
|---|---|
| 2,3,7,8-TCDD | 1746-01-6 |
| Total TCDD | 41903-57-5 |
| 2,3,7,8-TCDF | 51207-31-9 |
| Total TCDF | 30402-14-3 |
| 1,2,3,7,8-PeCDD | 40321-76-4 |
| Total PeCDD | 36088-22-9 |
| 1,2,3,7,8-PeCDF | 57117-41-6 |
| 2,3,4,7,8-PeCDF | 57117-31-4 |
| Total PeCDF | 30402-15-4 |
| 1,2,3,6,7,8-HxCDD | 57653-85-7 |
| 1,2,3,4,7,8-HxCDD | 39227-28-6 |
| 1,2,3,7,8,9-HxCDD | 19408-74-3 |
| Total HxCDD | 34465-46-8 |
| 1,2,3,6,7,8-HxCDF | 57117-44-9 |
| 1,2,3,4,7,8-HxCDF | 70648-26-9 |
| 1,2,3,7,8,9-HxCDF | 72918-21-9 |
| 2,3,4,6,7,8-HxCDF | 60851-34-5 |
| Total HxCDF | 55684-94-1 |
| 1,2,3,4,6,7,8-HpCDD | 35822-39-4 |
| Total HpCDD | 37871-00-4 |
| 1,2,3,4,6,7,8-HpCDF | 67562-39-4 |
| 1,2,3,4,7,8,9-HpCDF | 55673-89-7 |
| Total HpCDF | 38998-75-3 |
| Total OCDD | 3268-87-9 |
| Total OCDF | 39001-02-0 |

a. CAS = Chemical Abstract Service.

***5.1.3.4 Particulate, Hydrogen Chloride, and Chlorine.*** Emissions samples for particulate matter, hydrogen chloride, and chlorine will be collected according to EPA Method 26A. The sampling procedure is described in Appendix 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 hydrogen chloride and chlorine.

***5.1.3.5 Multiple Metals.*** Multiple metals emissions samples will be collected using an EPA Method 29 sampling train according to the procedure in Appendix A. Metals will be analyzed by inductively coupled plasma spectroscopy using SW-846 Method 6010. Mercury will be analyzed using cold vapor atomic absorption spectroscopy using SW-846 Method 7470. Target metal analytes are shown in Table 5-8.

Table 5-8. Target metal analytes.

| Metal | CAS[a] Number |
|---|---|
| Antimony | 7440-36-0 |
| Arsenic | 7440-38-2 |
| Barium | 7440-39-3 |
| Beryllium | 7440-41-7 |
| Cadmium | 7440-43-9 |
| Chromium | 7440-47-3 |
| Cobalt | 7440-48-4 |
| Lead | 7439-92-1 |
| Manganese | 7439-96-5 |
| Mercury | 7439-97-6 |
| Nickel | 7440-02-0 |
| Selenium | 7782-49-2 |
| Silver | 7440-22-4 |
| Thallium | 7440-28-0 |
| Zinc | 7440-66-6 |

a. CAS = Chemical Abstract Service.

***5.1.3.6 Sampling Point Access.*** The physical layout of the IWTU Facility dictates the use of a horizontal duct for process off gas sampling. Ports for isokinetic sampling trains (M0010, M26A, M0023A, and M29) are oriented with one port in the horizontal position and one port in the vertical position (see Appendix E). The sampling trains are designed to be oriented in a horizontal or near horizontal position in order to avoid spilling liquids contained in the impingers. To collect samples from the vertically oriented ports in the horizontal duct, the probe on each isokinetic sampling train will be attached to the remainder of the sampling train using a flexible, heated Teflon tube. This will allow the impingers to be oriented horizontally, while the probe is inserted vertically downward into the port. The Teflon tube will be treated as part of the probe and will be included in the probe recovery procedure of the sampling method.

## 5.2 Analytical Procedures

Appendix B describes the analytical methods to be used during the SPT.

## 5.3 Monitoring Procedures

During the SPT, the process off-gas carbon monoxide (CO) and oxygen ($O_2$) will be continuously monitored by installed CEMS. A temporary CEMS also will be used to monitor the process off-gases for total hydrocarbon using a flame ionization detector, for nitrogen oxides (NOx) using a chemiluminescence detector, and for CO and $O_2$.

The process off-gas CO and $O_2$ monitors will be operated during the test according to the protocols of Performance Specification 4B of 40 CFR 60, Appendix B. The process off-gas total hydrocarbon monitor will be operated during the test according to the protocols of Performance Specification 8A of

40 CFR 60, Appendix B and EPA Method 25A. The process off-gas NOx monitor will be operated during the test according to the protocols of Performance Specification 2 of 40 CFR 60, Appendix B and EPA Method 7E. The process off-gas monitors will be checked and calibrated daily during the SPT in accordance with the manufacturer's recommendations. Results of the monitoring will be included in the SPT report.

During the SPT, CO and $O_2$ monitor tests will include relative accuracy and EPA Method 10 (40 CFR Part 60, Appendix A) tests as required by 40 CFR Part 63, Subpart EEE, and the Appendix to Subpart EEE. Performance Specification 4B of Appendix B to 40 CFR Part 60 will be used. Initial setup, calibration, and calibration drift for the CO and $O_2$ monitors will be conducted prior to the SPT, and the acceptability of the monitors will be determined during the SPT. Results of the CO and $O_2$ monitor performance will be included in the SPT final report.

Appendix A describes the monitoring procedures to be used for the SPT.

## 5.4 Nitrogen Oxide Performance Test

During the SPT, a nitrogen oxide performance test will be conducted to determine emissions from the IWTU using 40 CFR Part 61, Appendix A, Test Method 7E. The test will be performed as required by the IWTU Permit to Construct at the worst-case normal production rate. The exact date of the test has not been determined, but will occur within 60 days of achieving maximum production rate, but no later than 180 days after initial startup. A notice of intent to test will be submitted to DEQ at 15 days prior to the scheduled test date. A test report will be submitted to DEQ within 30 days of the completion of the test.

## 5.5 Visible Emissions Observation

A visible emissions observation will be conducted and recorded during the nitrogen oxide performance test, as required by the Permit to Construct.

## 5.6 Interlock Testing

Following the completion of the final SPT condition, the IWTU will undergo a test of the AWFCO interlock system and the mercury adsorber bypass system. During this test, the CRR oxygen concentration will be set below the low-low limit by manually setting the input in the Distributed Control System. This action will trigger the associated alarm, the waste feed cutoff interlock, and the mercury adsorber bypass. This test will be documented in the SPT report.

## 5.7 Quality Assurance and Quality Control Procedures

Appendix C contains a QAPjP for the SPT.

## 5.8 Supplemental Sampling Information

Appendix F contains additional information related to the sampling that will be conducted during the SPT. This information includes:

- A description of the special procedures to be used during the sampling associated with the vertical sampling ports;
- A detailed description, of the total hydrocarbons and nitrogen oxides temporary Continuous Emission Monitor System (CEMS);

- Descriptions of the sample recovery procedures including the location of sample recovery and the procedures used to prevent cross contamination of samples;
- Copies of the datasheets to be used during the SPT; and
- Training requirements for the sampling crew and observers (training will be completed before the SPT begins, any observers who cannot attend the training will received minimum access training for IWTU and will require a full-time escort).

# 6. TEST SCHEDULE

The SPT will occur during facility startup and commissioning. Prior to the SPT, all components and systems will be tested to verify all equipment was installed correctly and is functioning properly. As part of the commissioning and testing, instrumentation will be calibrated. After all systems are tested individually, an integrated test will be conducted. The details of this testing are provided in PLN-3350, "IWTU Startup Plan."

## 6.1 Shakedown Period

The system may be operated for up to 720 hours on hazardous waste feed, and one 720-hour extension may be requested. The purpose of this shakedown period is to prepare the IWTU system for the SPT. In addition, monitors used for regulatory compliance purposes will be tested for accuracy during this phase of the commissioning. DEQ will be notified in advanced of this testing so that DEQ can observe the tests, if desired. This 720 hour time period begins when hazardous feed is introduced to the IWTU system.

Feed rate and operating restrictions during the shakedown period are shown in Table 7-1.

## 6.2 Pretest Activities

All process equipment used for compliance monitoring is shown in Table 3-1 of this SPT plan. Each of these instruments will be calibrated prior to the SPT. The oxygen and carbon monoxide CEMS will undergo calibration and relative accuracy testing prior to the SPT. Section 5.3 of this SPT plan provides specific information regarding the conduct of calibrations and relative accuracy testing for the $O_2$ and CO CEMS. Section 8.2 of the QAPjP (Appendix C to this SPT plan) discusses calibration of process instruments and calibration and relative accuracy testing of the $O_2$ and CO CEMS prior to the SPT.

With regard to other aspects of preparing for the SPT, the IWTU will undergo cold commissioning prior to the SPT, which will ensure operational readiness, including the ability to operate at SPT conditions. The planned commissioning activities are described in PLN-2020, "IWTU Commissioning, Transition and Readiness Plan," which was previously provided to DEQ. During the commissioning process CH2M-WG Idaho, LLC management and DOE management will do assessments to verify the facility is ready to conduct the SPT. These assessments will verify that the Safety Analysis is implemented, the HWMA/RCRA permit is implemented, and operational procedures are in place.

## 6.3 Planned Test Date

The SPT is expected to occur in May 2012.

## 6.4 Duration of Each Test

The SPT will consist of four replicate sampling runs at each of two test conditions. The IWTU will be operated on mixed waste, and process parameters will be established at the target values (as allowed by process conditions) for sufficient time prior to testing in order to establish steady process operations, as described in Section 4.5.1. Once this has been achieved, testing activities will begin. Each test run is anticipated to have a duration of 4 to 6 hours, including port changes, plus approximately 2 hours for sample recovery. Given the time required for sampling activities, it is planned that one run will be conducted per day. Assuming no interruptions to the testing activities, the SPT is expected to require approximately 32 to 48 hours of actual test time. An overall schedule of test days is shown in Table 6-1.

Table 6-1. Overall System Performance Test schedule.

| Day | Activity |
|---|---|
| Day 1 | Performance test contractor set-up. Pretest meeting. |
| Day 2 | Test Condition 1, Sampling Run 1. |
| Day 3 | Test Condition 1, Sampling Run 2. |
| Day 4 | Test Condition 1, Sampling Run 3. |
| Day 5 | Test Condition 1, Sampling Run 4. |
| Day 6 | Test Condition 2, Sampling Run 1. Packaging and shipping of samples from Test Condition 1. |
| Day 7 | Test Condition 2, Sampling Run 2. |
| Day 8 | Test Condition 2, Sampling Run 3. |
| Day 9 | Test Condition 2, Sampling Run 4. |
| Day 10 | Packaging and shipping of samples from Test Condition 2. |
| Day 11 | De-mobilization, contingency. |

## 6.5 Detailed Schedule of Planned Test Activities

A planned schedule for a typical test run is presented in Table 6-2. A pre-test briefing will be conducted prior to each test run for the specifics of the next sampling run, lessons learned and validation of calculations from the previous run, and other items as necessary.

Table 6-2. Anticipated run schedule.[a]

| Hour | Activity |
|---|---|
| 00:00 | System is operating on mixed waste at the target operating conditions. Testing crew begins setting up for testing. |
| 02:00 | Testing crew has all sampling trains in place and ready to begin testing. |
| 03:00 | Preliminary velocity traverses have been conducted and nozzle sizes selected.<br>Testing crew has completed test preparations and leak checks.<br>Ready to start first traverse with Method 0023A and Method 0010 sampling trains.<br>Ready to start first traverse with Method 26A and Method 29 sampling trains.<br>Method 0031 sampling apparatus setup and ready to begin sampling. |

Table 6-2. (continued).

| Hour | Activity |
|---|---|
| 03:00 | Start first sampling traverse of Method 0023A, Method 0010, Method 26A, and Method 29 sampling trains.<br>Start first Method 0031 tube set. |
| 03:40 | Complete first Method 0031 tube set. |
| 03:50 | Start second Method 0031 tube set. |
| 04:00 | Complete first traverse of Method 26A and Method 29 sampling trains and start second traverse of Method 26A and Method 29 sampling trains. |
| 04:00 to 04:30 | Conduct post-traverse leak checks of Method 26A and Method 29 sampling trains.<br>Switch sampling port locations of Method 26A and Method 29 sampling trains.<br>Conduct post-port change leak checks of Method 26A and Method 29 sampling trains. |
| 04:30 | Start second traverse of Method 26A and Method 29 sampling trains. |
| 04:30 | Complete second Method 0031 tube set. |
| 04:30 | Complete first traverse of Method 0023A and Method 0010 sampling trains. |
| 04:30 to 05:00 | Conduct post-traverse leak checks of Method 0023A and Method 0010 sampling trains.<br>Switch sampling port locations of Method 0023A and Method 0010 sampling trains.<br>Conduct post-port change leak checks of Method 0023A and Method 0010 sampling trains. |
| 04:40 | Start third Method 0031 tube set. |
| 05:00 | Complete first sampling traverse for Method 0023A and Method 0010 sampling trains and start second sampling traverse for Method 0023A and Method 0010 sampling trains.<br>Complete second traverse for Method 26A and Method 29 sampling trains. |
| 05:20 | Complete third Method 0031 tube set. |
| 05:30 | Start fourth Method 0031 tube set. |
| 05:30 | Complete second traverse of Method 26A and Method 29 sampling trains. |
| 05:30 to 05:40 | Conduct post-traverse leak check of Method 26A and Method 29 sampling trains.<br>Prepare trains for removal from duct. |
| 06:10 | Complete fourth Method 0031 tube set. |
| 06:30 | Complete second traverse of Method 0023A and Method 0010 sampling trains. |
| 06:30 to 06:50 | Conduct post-traverse leak checks of Method 0023A and Method 0010 sampling trains.<br>Prepare trains for removal from duct. |
| 06:50 to 07:00 | Remove all isokinetic sampling trains from duct.<br>Remove Method 0031 sorbent tubes from duct. |
| 7:00 to 9:30 | Recover samples, sample cleanup, sample recovery, and/or prepare for next run. |

a. Schedule repeated each sampling run. Time for setup and preliminary velocity traverses may be shortened following the first run. Sample recovery estimated to require 2 to 3 hours. Bed transfers will be required during a run and will be noted in the test log.

# 7. OPERATING LIMIT OBJECTIVES

Based on the data collected from all test runs of the SPT the operating limits for the IWTU will be proposed consistent with the provisions of HWMA/RCRA Subpart X. Parameters will be established from the operating conditions demonstrated during the test, operating requirements, manufacturer's recommendations, or good operating practice. If the required performance objectives of the test are achieved, it will be proposed that the IWTU be allowed to operate under the conditions described in this section.

Table 7-1 summarizes the anticipated operating limits. These operating limits also are proposed for the SPT period and for the shakedown period as described in Section 6 of this SPT plan.

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 discuss each operating parameter.

Table 7-1. Proposed IWTU operating parameter limits.

| Control Parameters | Limit | Comments |
|---|---|---|
| **GROUP 1 PARAMETERS**[a] | | |
| Maximum total waste feed rate (gal/min) | 3.5 | Hourly rolling average AWFCO |
| Minimum DMR off-gas temperature (°C) | 605 | Hourly rolling average AWFCO |
| Minimum DMR off-gas hydrogen concentration (vol%, dry) | 1.0 | 10-minute rolling average AWFCO |
| Minimum CRR off-gas temperature (°C) | 875 | Hourly rolling average AWFCO |
| Minimum CRR off-gas oxygen concentration (vol%, wet) | 2 | 10-minute rolling average AWFCO |
| Maximum off-gas cooler outlet temperature (°C) | 220 | Hourly rolling average AWFCO |
| Maximum DMR superficial gas velocity (surrogate for residence time) (ft/sec) | 1.3 | Hourly rolling average AWFCO |
| **GROUP 2 PARAMETERS**[b] | | |
| Maximum off-gas exhaust carbon monoxide (ppmv, dry) | 100 | Hourly rolling average AWFCO |
| Maximum DMR pressure (psig) | 12 | Instantaneous AWFCO |
| Maximum CRR pressure (in. w.c.) | 0 | Instantaneous AWFCO |
| **GROUP 3 PARAMETERS**[c] | | |
| Maximum DMR off-gas temperature ($^{o}$C) | 700 | Hourly rolling average AWFCO |
| Maximum DMR off-gas hydrogen concentration (vol%, dry) | 15 | 10-minute rolling average AWFCO |
| Minimum DMR superficial gas velocity (ft/sec) | 0.3 | Hourly rolling average AWFCO |
| Maximum CRR off-gas temperature ($^{o}$C) | 1,150 | Hourly rolling average AWFCO |
| Minimum off-gas cooler outlet temperature (°C) | 120 | Hourly rolling average AWFCO |
| Minimum HEPA filter inlet temperature (°C) | 120 | Hourly rolling average AWFCO |
| Maximum HEPA filter pressure differential (in. w.c.) | 5 | Hourly rolling average |
| Maximum mercury adsorber inlet temperature (°C) | 180 | Hourly rolling average AWFCO |

Table 7-1. (continued).

| Control Parameters | Limit | Comments |
|---|---|---|
| Minimum mercury adsorber inlet temperature (°C) | 120 | Hourly rolling average AWFCO |
| Maximum mercury adsorber bed age | NA | Determined by periodic breakthrough testing |
| Mercury adsorber activated carbon specifications | NA | MERSORB® HT-3 or equal |

a. Group 1 parameters are continuously monitored and recorded, and are interlocked with the AWFCO system. The values for the Group 1 parameters will be based on the SPT operating conditions

b. Group 2 parameters are continuously monitored and recorded, and are interlocked with the AWFCO system. The values for the Group 2 parameters are based on regulatory targets and SPT operating conditions.

c. Group 3 parameters are limits that are anticipated to be included in the permit and will be continuously monitored and recorded, and will be tied to the AWFCO interlock system, as indicated in the table. The values for the Group 3 parameters are based on manufacturer's specifications and/or operational and safety considerations rather than SPT operating conditions.

Table 7-2. Automatic waste feed cutoff setpoints.

| Control Parameters | Limit | Comments |
|---|---|---|
| **GROUP 1 PARAMETERS** | | |
| Maximum total waste feed rate (gal/min) | 3.8 | Hourly rolling average AWFCO |
| Minimum DMR off-gas temperature (°C) | 580 | Hourly rolling average AWFCO |
| Minimum DMR off-gas hydrogen concentration (vol%, dry) | 1.0 | 10-minute rolling average AWFCO |
| Minimum CRR off-gas temperature (°C) | 850 | Hourly rolling average AWFCO |
| Minimum CRR off-gas oxygen concentration (vol%, wet) | 1 | 10-minute rolling average AWFCO |
| Maximum off-gas cooler outlet temperature (°C) | <250 | Hourly rolling average AWFCO |
| Maximum DMR superficial gas velocity (surrogate for residence time) (ft/sec) | 1.5 | Hourly rolling average AWFCO |
| **GROUP 2 PARAMETERS** | | |
| Maximum off-gas exhaust carbon monoxide (ppmv, dry)[a] | 100 | Hourly rolling average AWFCO |
| Maximum DMR pressure (psig) | 12 | Instantaneous AWFCO |
| Maximum CRR pressure (in. w. c.) | 0 | Instantaneous AWFCO |
| **GROUP 3 PARAMETERS** | | |
| Maximum DMR off-gas temperature ($^{o}$C) | 700 | Hourly rolling average AWFCO |
| Maximum DMR off-gas hydrogen concentration (vol%, dry) | 15 | 10-minute rolling average AWFCO |
| Minimum DMR superficial gas velocity (ft/sec) | 0.3 | Hourly rolling average AWFCO |
| Maximum CRR off-gas temperature ($^{o}$C) | 1,150 | Hourly rolling average AWFCO |
| Minimum off-gas cooler outlet temperature (°C) | 120 | Hourly rolling average AWFCO |
| Minimum HEPA filter inlet temperature (°C) | 120 | Hourly rolling average AWFCO |
| Maximum mercury adsorber inlet temperature (°C) | 180 | Hourly rolling average AWFCO |
| Minimum mercury adsorber inlet temperature (°C) | 120 | Hourly rolling average AWFCO |

a. Corrected to 7% oxygen, dry. AWFCO interlock will not be active during the daily CEMS calibration period.

## 7.1 Development of Permit Limits

Permit limits for each control parameter will be established to comply with the requirements of the applicable environmental regulations. The following sections describe how each control parameter limit will be established.

### 7.1.1 Parameters Demonstrated During the Test

Group 1 parameter limits are based on the results of the SPT. The following paragraphs describe the limits proposed as Group 1 parameters for the IWTU.

***7.1.1.1 Maximum Total Waste Feed Rate (Group 1).*** The SPT will be conducted in order to demonstrate the maximum total waste feed rate to the IWTU. The maximum allowable total waste feed rate will be established from the overall average of the total waste feed rate demonstrated during each test run of either Condition 1 or Condition 2, depending on the suitability of the test results. The waste feed rate limit will be complied with on an hourly rolling average basis, and will be tied to the AWFCO system.

***7.1.1.2 Minimum Off-Gas Temperature at DMR and CRR (Group 1).*** The SPT will be conducted at the minimum DMR and CRR off-gas temperature, with maximized process gas flow (minimum residence time) because these conditions are least favorable for organic treatment. Organic emissions also are being measured for risk evaluation purposes because it is expected to represent bounding conditions for organic emissions. DMR and CRR gas temperatures will be monitored continuously. Based on a successful SPT, the minimum temperature limit at the DMR and CRR exit will be based on the average of the temperature values demonstrated in each run of the SPT. The limit will be based on an hourly rolling average and will be tied to the AWFCO system.

***7.1.1.3 Minimum DMR Off-Gas Hydrogen Concentration and Minimum CRR Off-Gas Oxygen Concentration (Group 1).*** The SPT will be conducted to demonstrate the minimum DMR off-gas hydrogen concentration and the minimum CRR off-gas oxygen concentration. Based on successful performance demonstration, the minimum DMR off-gas hydrogen concentration and the minimum CRR off-gas oxygen concentration limits will be based on the average values demonstrated during each run of the SPT. Compliance with these limits will be on a 10-minute rolling average basis and will be tied to the AWFCO system.

***7.1.1.4 Maximum Off-Gas Cooler Temperature (Group 1).*** The SPT will be conducted to demonstrate the maximum OGC temperature. Based on successful demonstration of PCDD/PCDF emissions control during the SPT, the maximum OGC temperature limit will be based on the average temperature values demonstrated during each run of the SPT. The limit will be established as an hourly rolling average and will be tied to the AWFCO system.

***7.1.1.5 Maximum DMR Superficial Gas Velocity (Group 1).*** The DMR superficial gas velocity will be maximized as an indicator of minimum gas residence time. The maximum DMR superficial gas velocity will be established from the mean of the maximum hourly rolling average velocities demonstrated during each run of the SPT. The limit will be based on an hourly rolling average and will be tied to the AWFCO system.

### 7.1.2 Parameters Established by Other Means

Group 2 and Group 3 parameters are determined from information other than the test results. These parameters and how the limits are to be established are described in the following sections.

***7.1.2.1 Maximum Process Off-gas Carbon Monoxide Concentration (Group 2).*** The maximum hourly rolling average process off-gas carbon monoxide concentration will be maintained at or below 100 ppmv (dry basis, corrected to 7% oxygen) during the SPT. A permit limit is expected specifying a maximum allowable process off-gas carbon monoxide concentration of 100 ppmv hourly rolling average, dry basis, based on the regulatory target.

***7.1.2.2 Maximum DMR and CRR Pressure (Group 2).*** A maximum DMR pressure of 12 psig, and a maximum CRR pressure of 0.0 in. w.c. will be established to prevent fugitive emissions. DMR and CRR pressures will be monitored continuously, and the waste feeds will be instantaneously cut off if either the DMR or CRR pressure exceeds the limit.

***7.1.2.3 Maximum DMR and CRR Off-Gas Temperature (Group 3).*** The temperature of the gases exiting the DMR and the CRR will be maintained below a predetermined limit to ensure performance. These limits are based on good operating practice. During the SPT, the DMR and CRR temperature will be operated at minimum temperature conditions, representing worst-case conditions for organic treatment. Maximum DMR and CRR off-gas temperature limits will be established as an hourly rolling average based on good operating practice.

***7.1.2.4 Maximum DMR Off-Gas Hydrogen Concentration (Group 3).*** The DMR off-gas hydrogen concentration is maintained below a preset maximum to ensure proper operation of the IWTU. The permit is expected to contain limits on the maximum DMR off-gas hydrogen concentration at this preset value. Compliance will be on a 10-minute rolling average basis.

***7.1.2.5 Minimum DMR Superficial Gas Velocity (Group 3).*** A minimum DMR superficial gas velocity will be established based on manufacturer's recommendations for proper operation. This limit will be complied with on an hourly rolling average basis.

***7.1.2.6 Minimum Off-Gas Cooler Outlet Temperature (Group 3).*** A minimum OGC temperature will be established based on manufacturer's recommendations for proper operation. This limit will be complied with on an hourly rolling average basis.

***7.1.2.7 Minimum HEPA Filter Inlet Temperature (Group 3).*** A minimum HEPA filter inlet temperature will be established based on manufacturer's recommendations for proper operation. This limit will be complied with on an hourly rolling average basis.

***7.1.2.8 Maximum HEPA Filter Differential Pressure (Group 3).*** The HEPA filter system is a passive unit without specific operational controls. The HEPA filter system will be monitored for pressure differential in accordance with manufacturer's recommendations. A maximum pressure differential limit of 5 in. w.c. has been established based on manufacturer's recommendations and will be complied with on a 60-second rolling average basis.

***7.1.2.9 Minimum and Maximum Mercury Adsorber Inlet Temperature (Group 3).*** A minimum and maximum temperature limit in the mercury adsorber carbon inlet will be established as an hourly rolling average, based on the equipment manufacturer's specifications and good operating practice.

***7.1.2.10 Maximum Mercury Adsorber Bed Age (Group 3).*** Mercury adsorber bed age is discussed in the Hazardous Waste Combustor Maximum Achievable Control Technology regulations as an operating parameter limit; however, establishing a specific maximum age is not practical in this unit. Periodic sampling of the carbon beds (as described in the Waste Analysis Plan [Attachment 2, Section C-2 of the HWMA/RCRA Permit]) will be conducted to determine breakthrough and changeout of the carbon beds.

***7.1.2.11 Mercury Adsorber Bed Specifications (Group 3).*** In order to maintain the effectiveness of the mercury adsorber carbon bed, it will be monitored in accordance with the manufacturer's recommendations, and will be changed out as required. The replacement carbon will be of the same type and manufacturer as used during the SPT, unless it can be shown that the replacement carbon meets or exceeds critical specifications of the carbon used during the SPT.

## 7.2 Post-SPT Operations

The interim period between completion of the SPT and receipt of final approval from DEQ for full operation authority could be several months. During this time, the IWTU intends to continue to operate full-time per the requirements of 40 CFR 264, "Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities," and 270, "EPA Administered Permit Programs: The Hazardous Waste Permit Program." During this period, the IWTU expects to operate within the operating envelope defined and demonstrated by the SPT. The proposed operating parameters are as follows:

- Feed rate: 3 gallons per minute
- DMR bed temperature: 640°C (±20°C)
- CRR bed temperature: 950°C (±50°C)
- CRR off-gas oxygen: 2% (±0.5%) wet basis
- OGC temperature: 180°C
- DMR superficial gas velocity: 1.0 ft/sec
- Process off-gas carbon monoxide: <100 ppmv, dry basis, corrected to 7% oxygen
- DMR off-gas hydrogen: 2% (±0.5%) dry basis.

# 8. TEST REPORT

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

1.0 EXECUTIVE SUMMARY
2.0 SYSTEM PERFORMANCE TEST SUMMARY
- 2.1 Engineering Description
  - 2.1.1 General Description
  - 2.1.2 Waste Feed Systems
  - 2.1.3 Solid Product Handling System
  - 2.1.4 Off-Gas Treatment System
  - 2.1.5 Process Continuous Monitoring System
  - 2.1.6 Continuous Emissions Monitoring
  - 2.1.7 Automatic Waste Feed Cutoff System
- 2.2 Summary of Test Plan and Objectives
- 2.3 Test Implementation Summary
  - 2.3.1 Deviations from the Test Plan

3.0 PROCESS OPERATIONS
- 3.1 Process Operating Conditions
- 3.2 Feed Material Characteristics
- 3.3 Effluent Characteristics

4.0 COMPLIANCE RESULTS
- 4.1 Organic Emissions
- 4.2 Particulate Emissions
- 4.3 Hydrogen Chloride and Chlorine Emissions
- 4.4 Metals Emissions
- 4.5 Process Off-gas Carbon Monoxide and Total Hydrocarbons
- 4.6 Process Off-gas Nitrogen Oxides
- 4.7 Dioxin and Furan Emissions
- 4.8 CEMS Performance Evaluation Results

5.0 QUALITY ASSURANCE/QUALITY CONTROL RESULTS
- 5.1 QA/QC Activities and Implementation
  - 5.1.1 QA Surveillance
  - 5.1.2 Sample Collection
  - 5.1.3 Sample Analysis
  - 5.1.4 Process Instrumentation
  - 5.1.5 Emissions Sampling Equipment
  - 5.1.6 Laboratory Analytical Instrumentation
- 5.2 Audits and Data Validation
- 5.3 Calculations
- 5.4 Conclusions

6.0 RISK EVALUATION
7.0 PROPOSED PERMIT OPERATION CONDITIONS
- 7.1 Development of Operation Limits
- 7.2 Specific Control Parameters

APPENDICES
- A. Process Operating Data
- B. Test Manager's Log
- C. Process Instrument Calibration Data
- D. Continuous Emissions Monitoring Data
- E. Sampling Report
- F. List of Samples
- G. Analytical Report
- H. Calculations
- I. Documentation to Support Metals Extrapolation
- J. Data Validation Report
- K. Corrective Action Requests
- L. CEMS Performance Evaluation Report

Figure 8-1. Example System Performance Test report outline.

## 8.1 Emissions Determinations

### 8.1.1 Particulate Matter Emission Concentration

The particulate matter emission concentration will be determined in terms of grains of filterable particulate matter per dscf of gas, corrected to 7% oxygen.

### 8.1.2 Metal Emission Concentration

The mass emission rate and the emission concentration of each target metal will be determined. In addition, the 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 μg of metal per dscm of gas, corrected to 7% oxygen.

### 8.1.3 System Removal Efficiency for Specific Metal Groups

In addition to determining the metal emission concentrations, the IWTU operator may wish to utilize the measured SRE for each metal volatility group in the risk assessment or for other purposes. The SRE of mercury, semivolatile metals (lead and cadmium combined), and low volatility metals (arsenic, beryllium, and chromium combined) can be determined using the following formula:

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

Where

| | | |
|---|---|---|
| SRE | = | system removal efficiency (%) |
| $M_{in}$ | = | mass feed rate of metal (or metal group) |
| $M_{out}$ | = | mass emission rate of metal (or metal group). |

### 8.1.4 Hydrogen Chloride and Chlorine Emission Concentration

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

### 8.1.5 Polychlorinated Dibenzo-p-Dioxin and Polychlorinated Dibenzofuran Emission Concentration

The emission concentration of PCDD/PCDF will be determined in terms of ηg of 2,3,7,8-TCDD toxicity equivalents per dscm of gas, corrected to 7% oxygen.

### 8.1.6 Carbon Monoxide Emission Concentration

The concentration of carbon monoxide will be continuously monitored and reported in terms of ppmv, dry basis, corrected to 7% oxygen.

### 8.1.7 Total Hydrocarbon Emission Concentration

During each test run, the total hydrocarbon emission concentration will be determined in terms of ppmv, dry basis, as propane, corrected to 7% oxygen.

### 8.1.8 Nitrogen Oxide Emission Concentration

During each test run, the nitrogen oxide emission concentration will be determined in terms of ppmv, dry basis, corrected to 7% oxygen, and in terms of mass per unit time.

## 8.2 Other Emission Determinations

Table 8-1 shows the emission determinations to be made in addition to the regulatory compliance emission determinations.

Table 8-1. Additional emission determinations.

| Parameter | Units |
|---|---|
| Off-gas flow rate | dscf/min, dscm/min |
| Off-gas velocity | ft/s, m/s |
| Off-gas temperature | °F, °C |
| Off-gas moisture content | vol% |
| Off-gas oxygen concentration | vol%, dry |
| Off-gas carbon dioxide concentration | vol%, dry |
| Off-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 | lb/h, g/s |
| PCDD/PCDF emission rate (each 2,3,7,8-substituted congener, and total toxicity equivalent) | lb/h, g/s |
| Speciated VOC emission rate (each target analyte plus each TIC) | lb/h, g/s |
| Speciated SVOC emission rate (each target analyte plus each TIC) | lb/h, g/s |
| PAH emission rate (each target analyte) | lb/h, g/s |

## 8.3 Process Data

Table 8-2 shows the process data points that 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 in the body of the report.

Table 8-2. Process data points to be monitored.

| Process Parameter | Units |
|---|---|
| Total waste feed rate | gal/min |
| DMR pressure | psig |
| CRR pressure | in. w.c. |
| DMR off-gas temperature | $^{o}C$ |
| DMR superficial gas velocity | ft/sec |
| CRR off-gas temperature | $^{o}C$ |
| DMR off-gas hydrogen concentration | vol%, dry |
| CRR oxygen concentration | vol%, wet |
| Off-gas cooler outlet temperature | $^{o}C$ |
| HEPA filter inlet temperature | $^{o}C$ |
| HEPA filter pressure differential | in. w.c. |
| Mercury adsorber inlet temperature | $^{o}C$ |

## 8.4 Risk Evaluation

The compounds listed in EDF-9483, "IWTU System Performance Test: Risk Evaluation for Organic Compounds Emissions," Tables 4 and 5 will be part of the sampling and analysis completed during the SPT. The results of the emissions analysis will be compared to the calculated numbers in Table 5 of the EDF. If the concentrations are at or below the release rates from Tables 4 and 5, then the SPT has demonstrated that operating the IWTU within the bounding conditions defined by the SPT does not pose a risk to workers or the public through its emissions.

# 9. PREFACE TO APPENDIXES

US EPA Sampling Methods and SW-846 Analytical Test Methods are sampling and analytical methods that have been evaluated and approved for use in complying with the RCRA regulations. These methods, although not required, provide guidance to samplers, analysts, data users, and other interested parties regarding methods that may be employed for the evaluation of solid waste and other testing specified in regulations issued under RCRA. The methods contain guidance on how to perform sampling and analytical procedures or techniques that samplers or laboratories can use as basic starting points for generating their own detailed Standard Operating Procedures (SOPs), either for their own general use or for a specific project application.

The sampling and analytical methods listed and described in Appendix A and Appendix B are general guidance from the methods themselves and are not intended to be prescriptive. While representative of the sampling and analytical methods used by the sampling contractor and analytical laboratory, they are not intended to be consistent in specific details.

The qualified stack emission testing contractor, working with a qualified emission testing laboratory, supplied supplemental sampling information with proposed sampling details (Appendix F), along with justification for solvent and rinse substitutions, described below. The contracted qualified stack emission testing contractor will have a Qualified Source Testing Individual (QSTI certified), experienced emissions expert, and test leader on-site.

The emission testing laboratory supplied supplemental sampling information and justification for solvent and rinse substitutions. This laboratory is one of only two full-service laboratories in the country with more than 200 individual stationary sources tested in the last 10 years. The laboratory has developed a number of industry-leading analytical approaches to support the testing requirements associated with the examination of stationary source emissions and indoor air quality. The laboratory has successfully developed a number of custom analytical applications, working with regulatory bodies to address unique testing applications that are not well suited for conventional testing applications. The laboratory services are designed to fulfill the requirements of major federal and state environmental programs, including trial burns and comprehensive performance tests. The laboratory holds numerous certifications/accreditations, including NELAP laboratory, certifications in 29 states, DoD ELAP, DOE (assessments for specialty services), and GSA contract laboratory. Applicable specialty program offerings include particulate, metals, fixed gases, and stationary source testing (volatile organic sampling trains [VOSTs], TCO/gravimetric, semivolatiles, PAHs, dioxins/furans, metals and chlorine).

The laboratory provided justification for solvent and rinse substitutions for Methods 0010 and 0023A as follows:

> For Method 0010, Methanol is substituted with Acetone in the similarly described solvent system from Method 0023A. The analytical laboratory recommends the same solvent recovery system for PAHs, SVOCs, and dioxins/furans. The only difference being that toluene is not needed for the PAHs or the SVOCs. Method 0010 solvent and glassware rinses using acetone followed by methylene chloride are the preferred approach. The acetone dries the probe and nozzle by removing all moisture droplets and the methylene chloride removes the remaining condensed semi-volatile compounds. The disadvantage of the 50/50 mixture of methanol in the laboratory is a procedural requirement to place this sample into water to remove the methanol before proceeding with the

continuous liquid-liquid extraction or Soxhlet extraction. This step is eliminated by the switch to acetone. Note that Method 0023A calls out this change explicitly as the solvent system, and allows semi-volatiles to be combined with dioxins and furans (See Sections 1.1, 5.6, and 7.2.2 of Method 0023A).

For Method 0023A the requirement to separate the toluene from the other two (2) solvents comes from the incompatibility of toluene in the SVOC analysis. Toluene has a high enough boiling point that it is not removed by the SVOC blowdown process. Also, note that if we are not combining any trains with the Method 0023A train, the need to separate the toluene is not required.

# Appendix A

# Sampling Methods

(see Section 6 and Table 6-1 for list of sampling methods references)

Table A-1. Method 26A Stack Gas Particulate Matter, Hydrogen Chloride, and Chlorine Sampling Procedure (Isokinetic).

Sample name: Stack Gas Method 26A (PM, Cl, $Cl_2$)

Sampler: Stack sampling team

Locations: Exhaust stack

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

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

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

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

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

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

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

Table A-1. (continued).

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

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

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

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

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

Table A-1. (continued).

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

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

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

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

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

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

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

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

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

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

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

Table A-1. (continued).

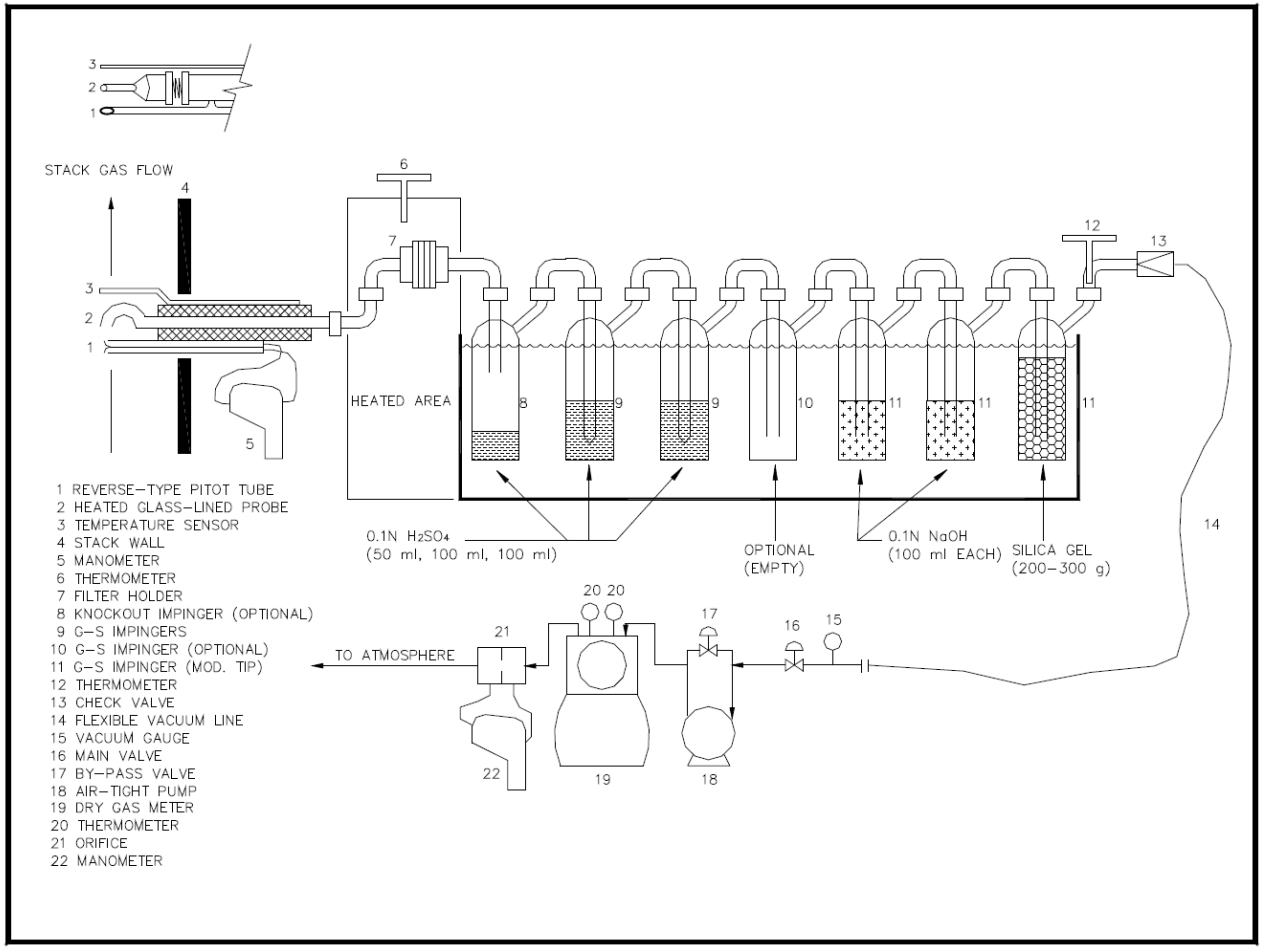


**Figure 1. Method 26A Sample Train for Particulate Matter, Hydrogen Chloride, and Chlorine**

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Table A-1. (continued).

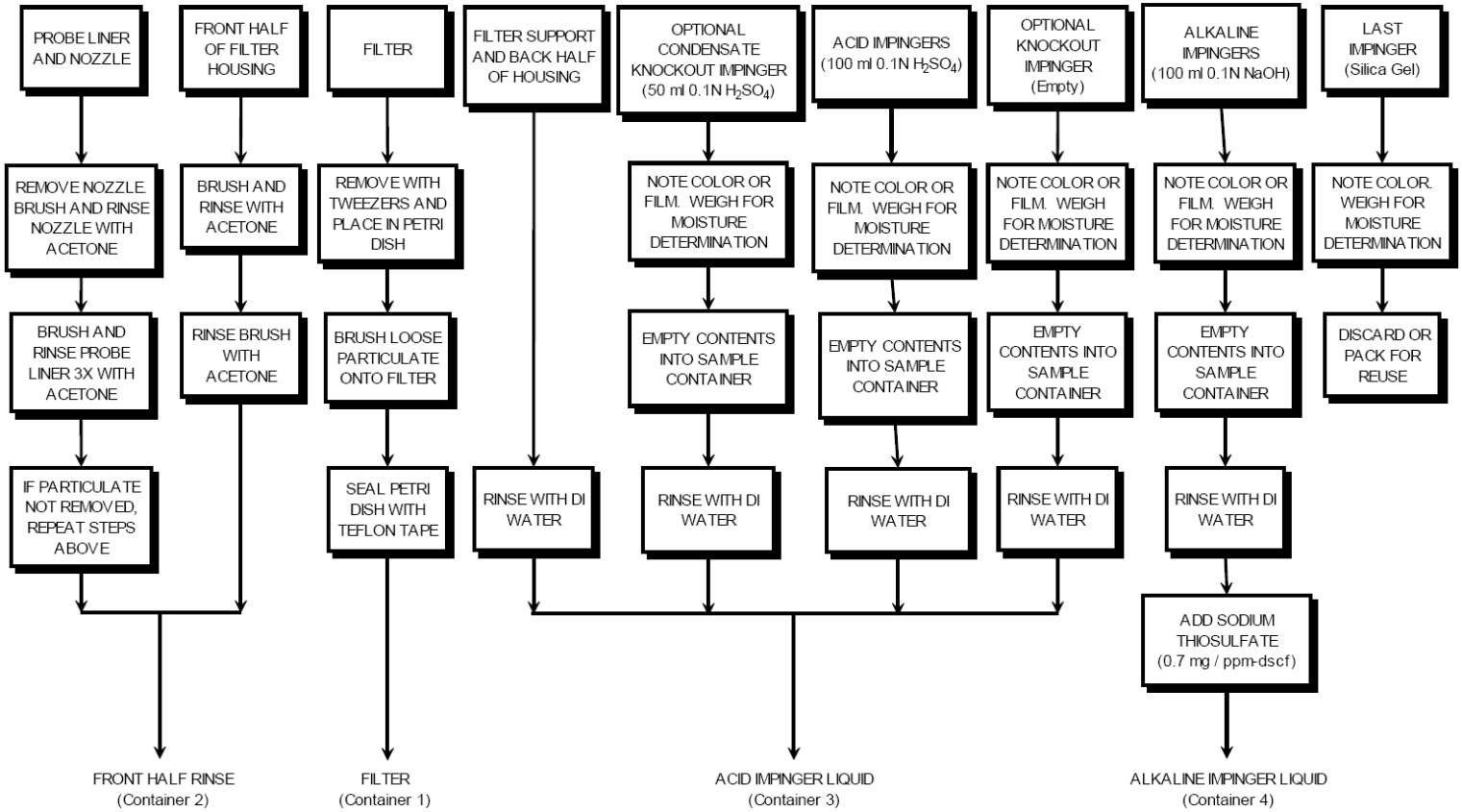


**Figure 2. Method 26A Particulate Matter, Hydrogen Chloride, and Chlorine Sample Recovery**

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Table A-2. Method 29 Stack Gas Multiple Metals Sampling Procedure (Includes Mercury, No Particulate).

Sample name: Stack Gas Method 29

Sampler: Stack sampling team

Locations: Exhaust stack

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

Frequency: Continuous for 120 minutes during each of three test runs.

Procedure Summary: Gases are sampled isokinetically to collect the metals Ag, As, Ba, Be, Cd, Cr, Co, Ni, Mn, Pb, Sb, Se, Tl, and Zn on a particulate filter and in absorbing solutions. The filter and absorbing solutions are recovered for subsequent metals analyses by inductively coupled plasma emission spectroscopy (ICP) and/or or graphite furnace AAS (GFAAS), and cold vapor AAS (CVASS). [Note: This procedure does not include collection of particulate matter (PM) samples for determination of PM concentration.]

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

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

The sampling train is assembled in a clean area at the test site and the train components are handled in a manner that minimizes contamination from air or by direct contact. A clean and inspected filter is placed in the filter holder. The first impinger is initially empty and serves as a condensate knockout impinger. (This impinger may be eliminated if the moisture to be collected is less than 100 ml.) The next two impingers each contain 100 ml of 5% $HNO_3$/10% $H_2O_2$ solution. The carryover impinger is empty and the next two impingers each contain 100 ml of 4% $KmnO_4$/10% $H_2SO_4$ solution. The last impinger contains 200 to 300 g of indicating silica gel.

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

Table A-2. (continued).

The sample train is operated according to Method 5 procedures for pretest and post-test leak-checks, isokinetic sampling rate, and data recording. The train leak rate must be the less than 0.02 cfm, or less than 4% of the average sampling rate. The sampling rate is adjusted during the test to maintain isokinetic conditions at the probe nozzle within ± 10% of 100%.

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

- Particulate Filter – A non-metallic tool is used to remove the particulate filter from its holder and place it in the original petri dish (Container 1). A nylon bristle brush is used to remove any PM or filter fibers from the filter gasket or the holder onto the filter, and the petri dish is sealed with tape and placed in a plastic bag.
- Front Half Rinse -- The internal surfaces of the nozzle, probe, and front half of the filter holder are cleaned by rinsing, brushing, and final rinsing with exactly 100 ml of 0.1N nitric acid. All rinses are placed into an sample bottle (Container 3). [Note: There is no Container 2 (Acetone Rinse) because the collection of PM samples is not included in this procedure.]
- Acidified Peroxide Impingers -- The liquid contents of the condensate knockout impinger (if used) and the two $HNO_3/H_2O_2$ impingers are measured to the nearest milliliter or weighed to the nearest 0.5 g and placed into a sample bottle (Container 4). The back half of the particulate filter holder is inspected for condensate, which if observed, is measured and/or weighed as described above and transferred to Container 4. The back half of the filter holder, the filter support, the optional condensate knockout impinger (if used), and the two $HNO_3/H_2O_2$ impingers, and all connecting glassware are rinsed with exactly 100 ml of 0.1N nitric acid, and the rinses are added to Container 4.
- Carryover impinger – The liquid contents of the initially empty impinger is measured to the nearest 0.5 ml and placed into a separate sample bottle (Container 5A). The impinger is then rinsed with exactly 100 ml of 0.10N nitric acid solution and the rinse is added to Container 5A.
- Acidified Permanganate Impingers -- The liquid contents of the acidified permanganate impingers are measured to the nearest 0.5 ml and placed into a separate sample bottle (Container 5B). These impingers and any connecting glassware are then rinsed a minimum of three times using a total of exactly 100 ml of fresh acidified potassium permanganate solution, and the rinses are added to Container 5B, being careful to also transfer any loose precipitated materials into the container. Triple rinsing of the acidified permanganate impingers is then repeated using a total of exactly 100 ml of water. The water rinses are also placed into Container 5B. If visible deposits remain in the impingers following the water rinses, they are rinsed with 25 ml of 8N hydrochloric acid, and the rinse is placed into a separate container (Container 5C) which contains 200 ml of water.

Table A-2. (continued).

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

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

The following reagent blank samples are collected once during the test program: one unused particulate filter; 300 ml of the 0.1N nitric acid solution; 100 ml of the water used in sample recovery; 200 ml of the acidified hydrogen peroxide solution, and 100 ml of the acidified potassium permanganate solution. If the acidified potassium permanganate impingers from any run are rinsed with HCl, then a 25 ml blank sample of the 8N HCl solution is also collected and added to 200 ml of water in a separate sample bottle.

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

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

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

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

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

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

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

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

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

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Table A-2. (continued).

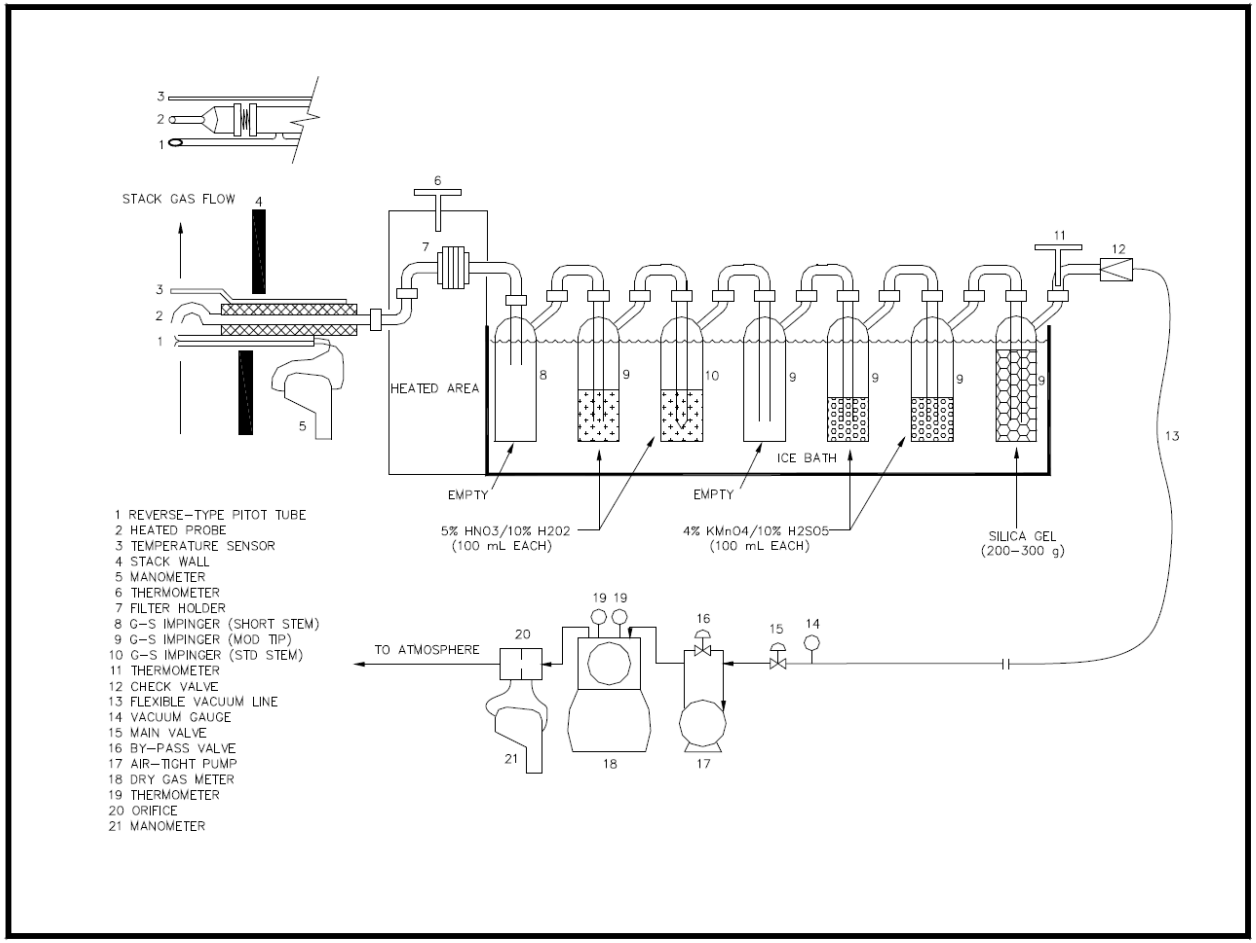


**Figure 1. Method 29 Sample Train for Multiple Metals (Including Hg) (No Particulate)**

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Table A-2. (continued).

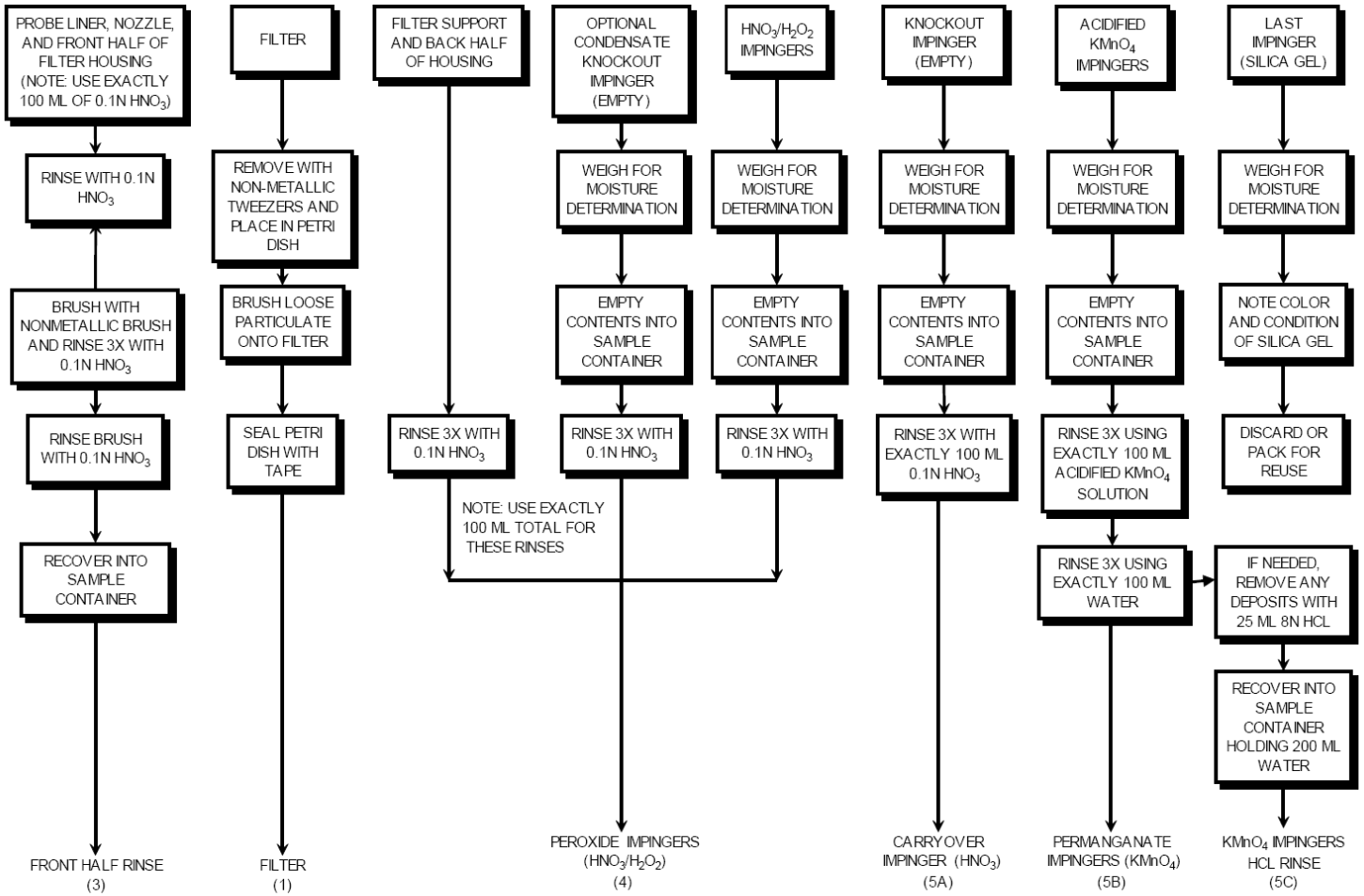


**Figure 2. Method 29 Multiple Metals (Including Hg) (No Particulate) Sample Recovery**

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Table A-3. Method 0031 Stack Gas Volatile organic Sampling Procedure.

Sample name: Stack Gas Method 0031 (SMVOC)

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 0031 volatile organic sampling train (SMVOC) as shown in Figure 1 of this table; Tenax and Anasorb sorbent cartridges sealed in glass culture tubes with Teflon lined screw caps; amber glass VOA vials (40 ml) with plastic screw caps and Teflon septa; screw capped glass container.

Frequency: Continuous with replacement of sorbent cartridges sets every 20 to 40 minutes for a total sampling time of 120 minutes. At least three sets of sorbent cartridges are collected during each test run.

Procedure Summary: Stack gases are sampled at a controlled rate of 0.5 to 1.0 L/min to collect volatile organic compounds (VOCs) on sorbent resin and in stack gas condensate. The mass of each target VOC collected is quantitatively determined by GC/MS analysis. (Note: This procedure is used for the collection of specific VOCs identified in the method that have boiling points between 24°C and 121°C.)

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

The sampling train components and reagents are prepared according to the procedures specified in Method 0031. As described in the method, the train glassware is cleaned with nonionic detergent in an ultrasonic bath, rinsed with hot tap water and organic-free water, and dried in an oven. New Tenax resin is soxhlet-extracted with methanol, vacuum dried, loaded into clean glass tubes, and thermally conditioned with organic-free nitrogen. New Anasorb resin, as received from the manufacturer, is loaded into clean glass tubes and thermally conditioned with organic-free nitrogen as specified in the method. All tubes are sealed with clean metal caps. Each cartridge (glass tube, sorbent, glass wool, and end caps) is permanently marked with an identification number and the direction of gas flow, and sealed in a glass culture tube that contains clean charcoal. Ten percent of the cartridges are subjected to GC/MS quality control measurements. Corrective action is taken if the target analytes are detected above their method detection limits. The dry gas meter is calibrated within 30 days before conducting the test program.

In the laboratory, the prepared cartridges are stored in a cooler at 4°C in an area free from volatile organic material. The cartridges are held for no more than 14 days before sampling. During pretest preparation, one set of cartridges is selected from the cartridge set and identified as a laboratory blank, which is retained at the laboratory for analysis with the field samples. For transfer to the test site, the cartridges are packed separate from other test supplies and placed on cold packs in insulated containers to keep their temperature less than 10°C for the duration of the trip. The laboratory blank cartridges are stored in the same manner as the field cartridges. At the test site, the insulated containers are stored in an area as free as possible from volatile organic sources and the cartridges are kept cold in the insulated containers until needed for a test run.

Table A-3. (continued).

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

A leak-check of the train assembly is performed by closing the isolation valve at the inlet to the first condenser, establishing ice bath water flow through the condensers, and using the pump to create a vacuum that is 25 cm (10 in.) Hg above normal operating pressure (total of approximately 38 cm Hg). The train leak rate must be less than 0.01 L/min before starting the sampling run. The train is returned to atmospheric pressure by admitting ambient air though charcoal at the train inlet.

The VOST is operated according to the procedures provided in Method 0031. The train controls are set to maintain a temperature 130°C ± 5°C at the sample probe outlet and 20°C at the inlet to the first cartridge. For each cartridge set, the stack gas is sampled for 20 to 40 minutes at 0.5 to 1.0 L/min to collect a nominal sample volume of 20 L. The sampling train operating conditions are recorded as required on field data sheets.

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

At the end of each test run, any water collected in the condensate trap is transferred into a VOA vial(s), the impinger is rinsed with minimum amounts of organic-free (HPLC) water, and the rinse is added to the vial. If there is not enough condensate and rinse to fill a VOA vial, HPLC water is added to eliminate the headspace. The vials are labeled with sample name, date, and test run number.

Field blank samples are collected once during each test run. One set of cartridges is attached to the sampling train while it is leak checked. The cartridges are removed from the train and stored with the test samples.

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

Samples are stored at less than 10°C in shipping packages, which are stored in an area away from other samples that may have high concentrations of VOCs. If shipped by truck, the samples are stored away from other chemicals or from where automotive exhaust fumes could become concentrated.

Reference: "Method 0031 – Sampling Method for Volatile Organic Compounds (SMVOC)", Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November 1986, and Updates.

Table A-3. (continued).

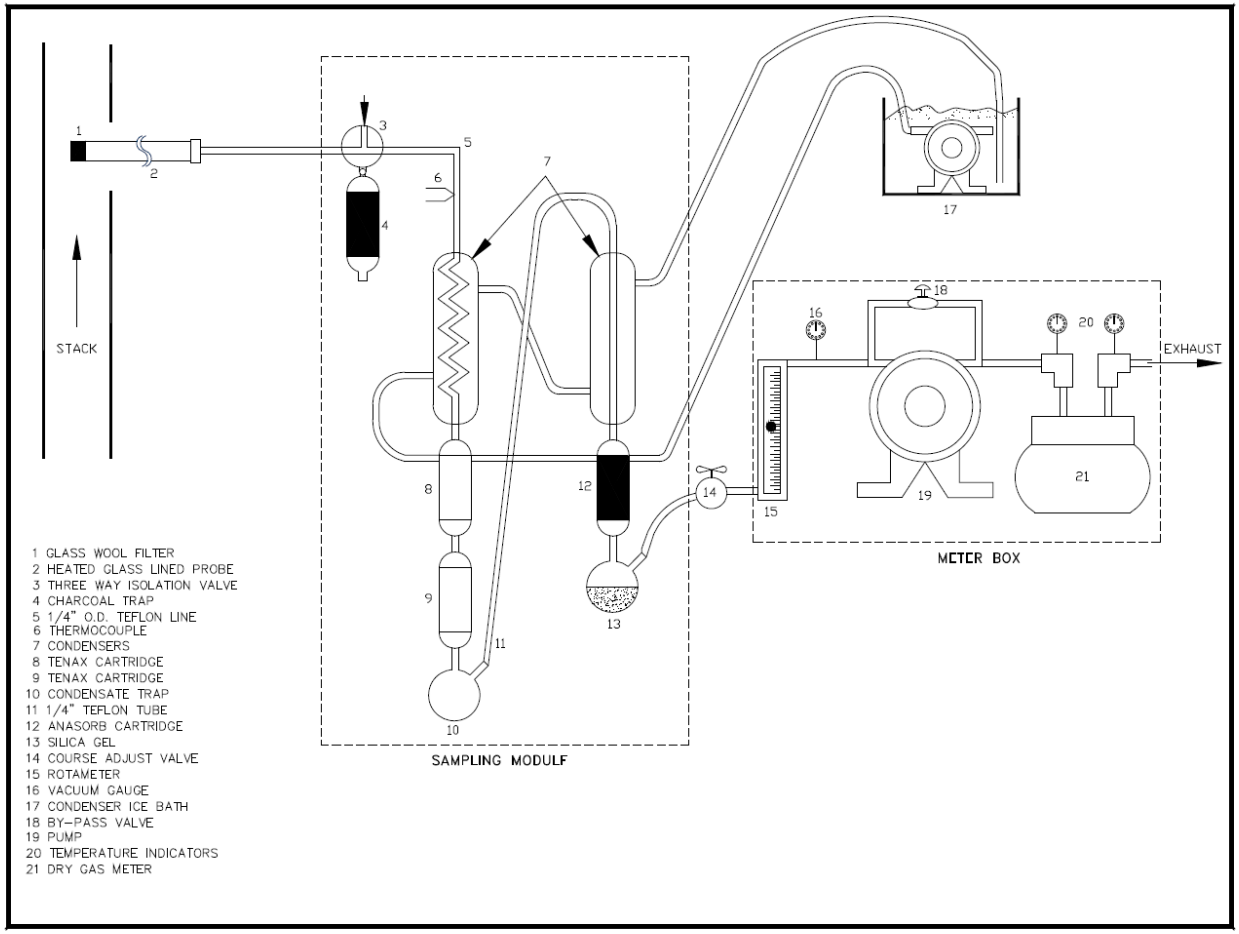


**Figure 1. Method 0031 Sample Train for Volatile Organics**

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Table A-3. (continued).

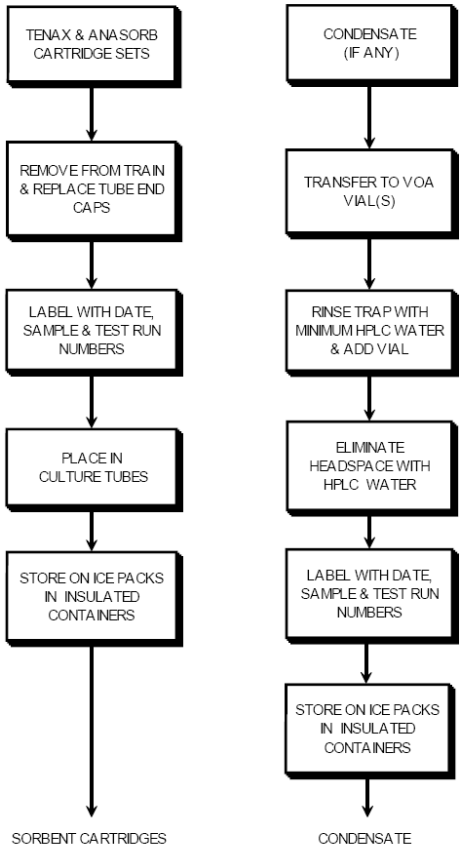


**Figure 2. Method 0031 Volatile Organic Sample Recovery**

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## A-4. Method 0023A Stack Gas PCDD/PCDF Sampling Procedure.

Sample name: Stack Gas Method 0023A (DF)

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 0023A sampling train as shown in Figure 1 of this table; tare weighed glass fiber particulate filters (without organic binders) sealed in glass petri dishes; prepared XAD-2 adsorbent resin traps; aluminum foil; amber glass bottles with Teflon-lined lids; scale balance; glass graduated cylinder.

Frequency: Continuous during each test run. Minimum sample time for each run is 3 hours, and the minimum sample volume for each run is 2.5 dry standard cubic meters. Sampling for these minimum times and volumes allows analytical non-detects to be counted as zero.

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

The Method 0023A train includes a glass probe nozzle, a heated sample probe with a borosilicate or quartz glass probe liner, heated particulate filter holder with Teflon frit filter support, a water cooled condenser, a water cooled adsorbent resin trap, a condensate knockout impinger, two water impingers, an empty impinger (optional), and a silica gel impinger. A water pump is provided to circulate ice bath water through the condenser and around the adsorbent trap. All of the impingers are placed in an ice bath. An air tight gas pump, dry gas flow meter, and flow rate meter complete the train.

All train components, reagents, and cleaning solutions are prepared according to the procedures specified in the methods referenced below. The pretest preparations include soaking the train glassware in hot (≥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 clean aluminum foil or clean glass plugs until sample train assembly.

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

- The filters are cleaned by soxhlet extraction using toluene. Following extraction, each filter is dried in a stream of clean dry inert gas and stored in a glass petri dish sealed with Teflon tape.
- XAD-2 resin is cleaned by rinsing twice with organic-free (HPLC) water followed by soxhlet extractions with HPLC water, methanol, methylene chloride, and toluene, and then dried using a flow of clean dry inert gas. An extract from a portion of the prepared XAD-2 resin is analyzed to confirm that it is free of significant background contamination. As an alternate to in-house preparation, resin that is certified clean by laboratory analysis may be purchased.
- The adsorbent trap is loaded with approximately 40 g of the XAD-2 resin and the open end of the trap is packed with clean glass wool. The adsorbent traps are spiked with isotope labeled PCDD/PCDF surrogate

Table A-4. (continued).

standards. The ends of the adsorbent trap are capped and the traps are wrapped in aluminum foil, sealed in a plastic bag, and stored in an insulated cold chest.

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

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

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

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

The stack gas sampling is conducted following the procedures given in Method 0023A. The sample probe exit and filter assembly temperatures are maintained at 248°F ± 25°F, and the gas sample entering the adsorbent module is maintained less than 68 °F. The sampling rate is adjusted during the test to maintain isokinetic conditions at the probe nozzle within ± 10% of 100%. A leak check is conducted during the test run whenever the sampling probe is removed from a sample port, such as during the stack traverse port change.

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

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

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

Table A-4. (continued).

- Front Half Rinse -- The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware are brushed and rinsed three times with acetone and rinsed three times with methylene chloride and two times with toluene. All rinses are placed into a glass sample bottle (Container No. 2). The final liquid level is marked on the sample container.
- XAD-2 Adsorbent Resin Trap -- The XAD-2 adsorbent resin trap is removed from the train, and both ends are capped. The trap is then labeled, covered with aluminum foil, sealed in a plastic bag and stored on ice in an insulated cold chest (Container No. 3).
- Back Half Rinse -- The back half of the filter holder, the filter support, transfer line (if used), and condenser (if separate from trap) are rinsed three times with acetone followed by two rinses with methylene chloride and two rinses with toluene. The back half rinses are placed into a glass sample bottle (Container No. 4). The final liquid level is marked on the sample container.
- Impinger water -- The liquid contents of the impingers are measured to the nearest milliliter or weighed to the nearest 0.5 g and are then discarded. Any color or film observed is noted on the sample recovery sheet.
- Silica Gel -- The silica gel contents of the last impinger are weighed to the nearest 0.5 g. The color and condition of the silica gel is noted on the sample recovery sheet.

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

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

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

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

Table A-4. (continued).

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

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

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

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

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

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

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

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

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

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

Table A-4. (continued).

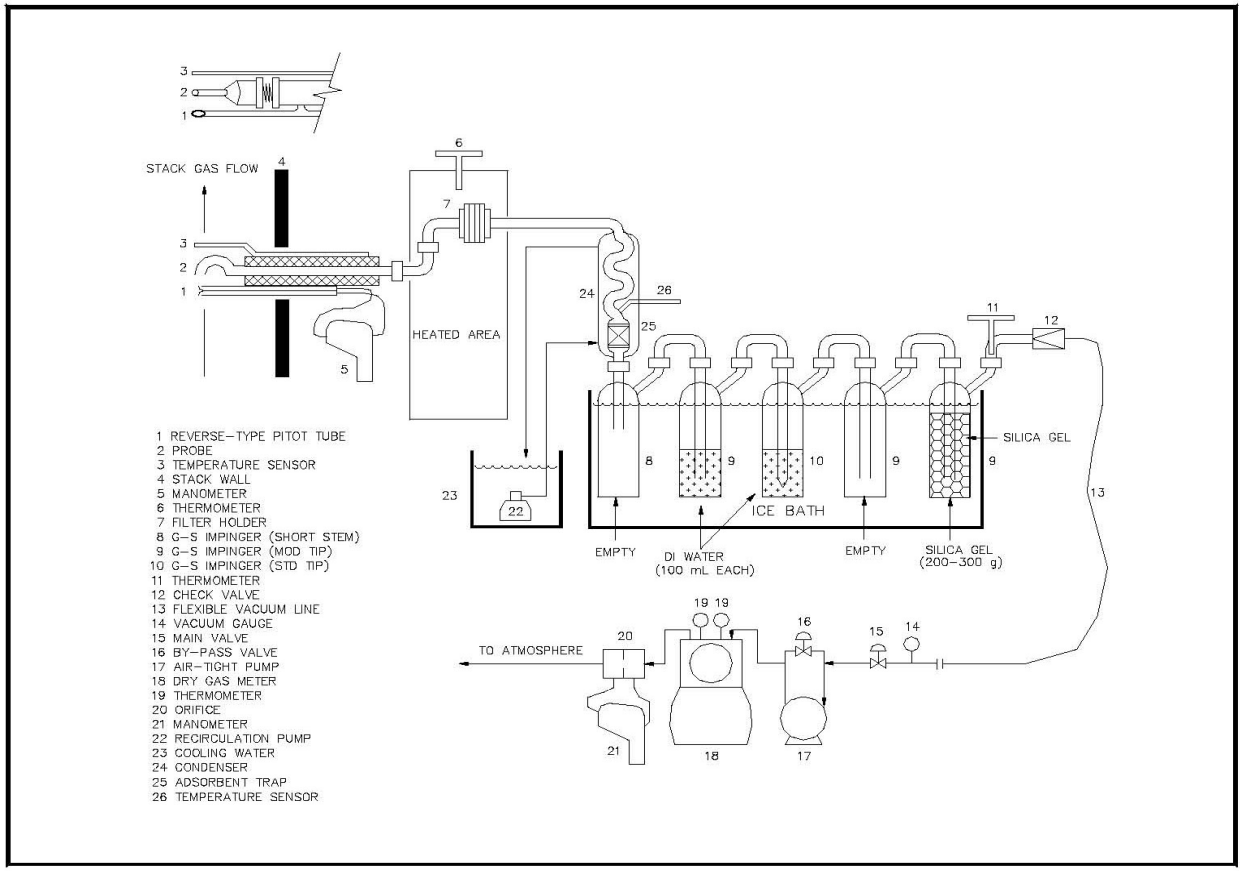


**Figure 1. Method 0023A Sample Train for PCDD/PCDF**

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Table A-4. (continued).

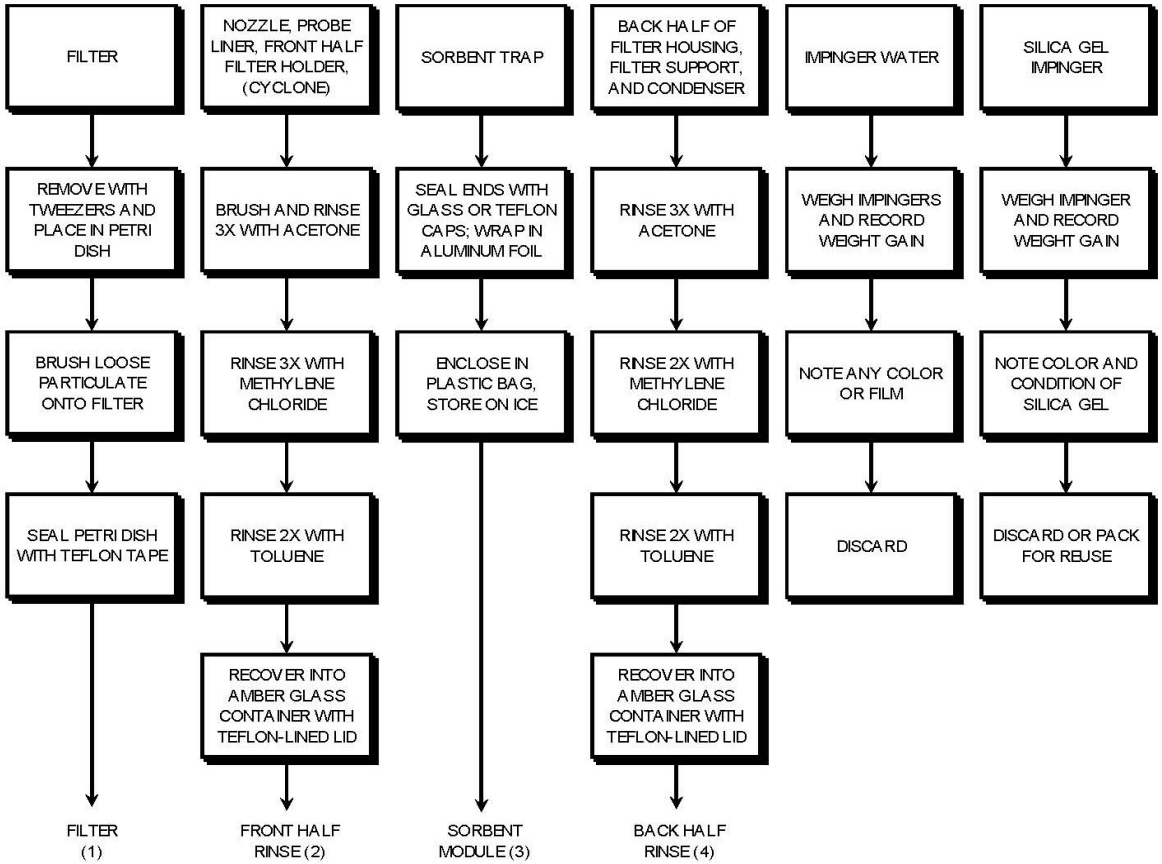


**Figure 2. Method 0023A PCDD/PCDF Sample Recovery**

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Table A-5. Method 0010 Stack Gas Semivolatile Organic Sampling Procedure.

Sample name: Stack Gas Method 0010 (SVOC)

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, methanol, and methylene chloride, then dried using a flow of clean inert gas. An extract is prepared from a portion of the XAD-2 resin and analyzed to confirm that it is free of significant background contamination. As an alternate to in-house preparation, resin that is certified clean by laboratory analysis may be purchased. The resin is stored at less than 120°F at all times.
- The adsorbent trap is loaded with approximately 40 g of the XAD-2 resin. The resin is spiked with an isotope labeled surrogate for the semivolatile organic 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.

Table A-5. (continued).

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 methanol/methylene chloride (1:1 v/v). All rinses are placed into a glass sample bottle (Container 2).
- XAD-2 Adsorbent Resin Trap -- The XAD-2 adsorbent resin trap is removed from the train, and both ends are capped. The XAD-2 adsorbent resin trap is then weighed to the nearest 0.5 gram and the weight recorded. The trap is then labeled, covered with aluminum foil, sealed in a plastic bag (Container 3) and stored in an insulated cold chest.
- Condensate Knockout Impinger -- The condensate collected in Impinger 1 is measured to the nearest milliliter or weighed to the nearest 0.5 g. and transferred a glass sample bottle (Container 4). The back half of the particulate filter holder is also inspected for condensate and recovered, if observed, by measuring or weighing and transferring to Container No. 4 as described above.
- Back Half Rinse -- The back half of the filter holder, the condenser, and the connecting line between the holder and the condenser are rinsed three times with methanol/methylene chloride (1:1 v/v). The condensate knockout impinger (Impinger 1) is rinsed with methanol/methylene

Table A-5. (continued).

chloride (1:1 v/v). The rinses are then placed into a glass sample bottle with a Teflon lined lid (Container 5).

- Impinger water -- The contents of impingers 2, 3, and 4 are measured to the nearest milliliter or weighed to the nearest 0.5 g, and observed for the presence or absence of film or color, which is recorded on the sample recovery sheet. If no color or film is present, the impinger water may be discarded. If color or film is observed, the impinger water is recovered and added to the condensate sample (Container 4).
- Silica Gel -- The silica gel contents of impinger 5 are weighed to the nearest 0.5 g.

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

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

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

"Method 0010 – Appendix A – Preparation of XAD-2 Adsorbent Resin", Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates

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

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

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

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

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

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

Table A-5. (continued).

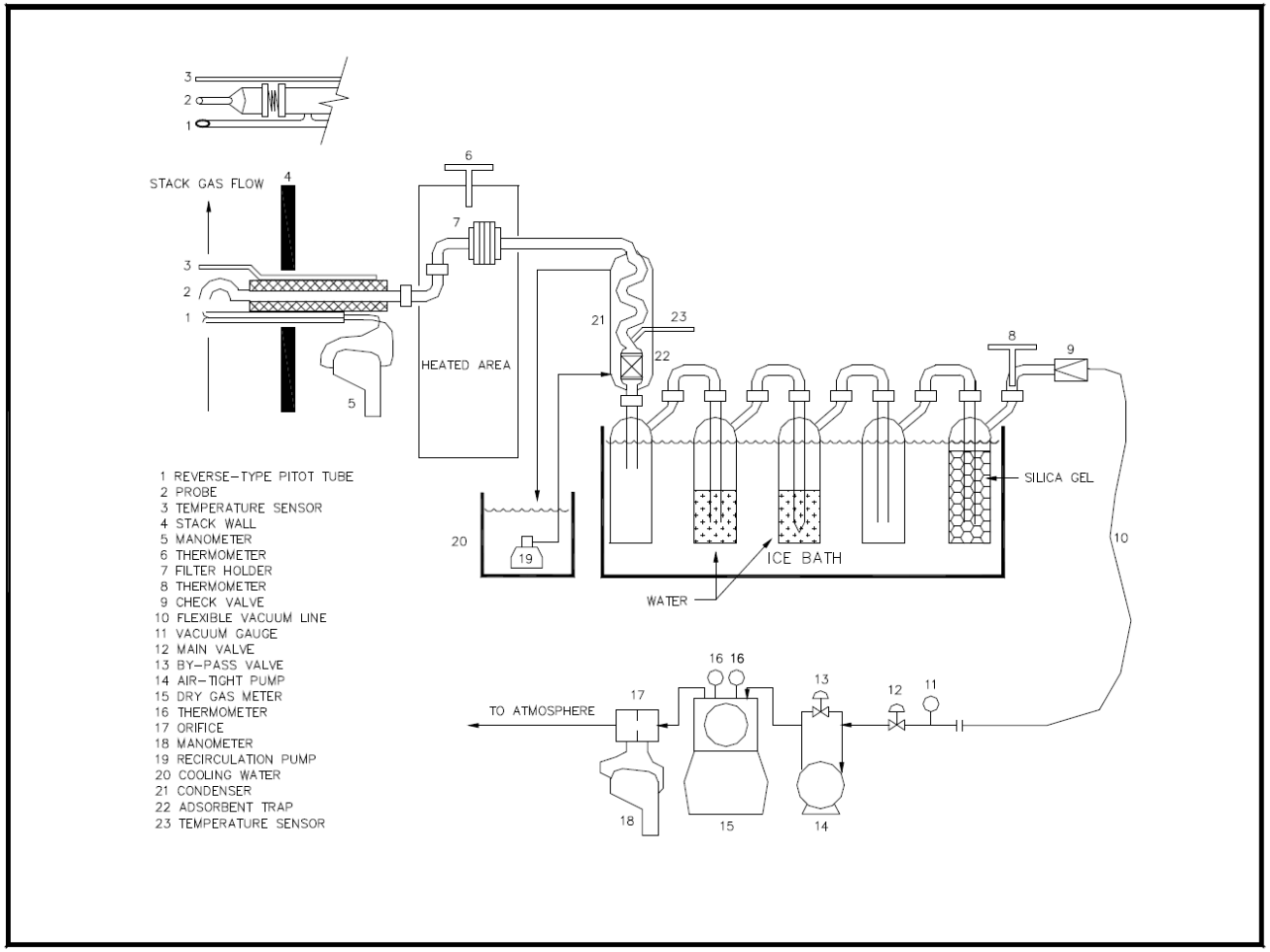


**Figure 1. Method 0010 Sample Train for Semivolatile Organics**

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Table A-5. (continued).

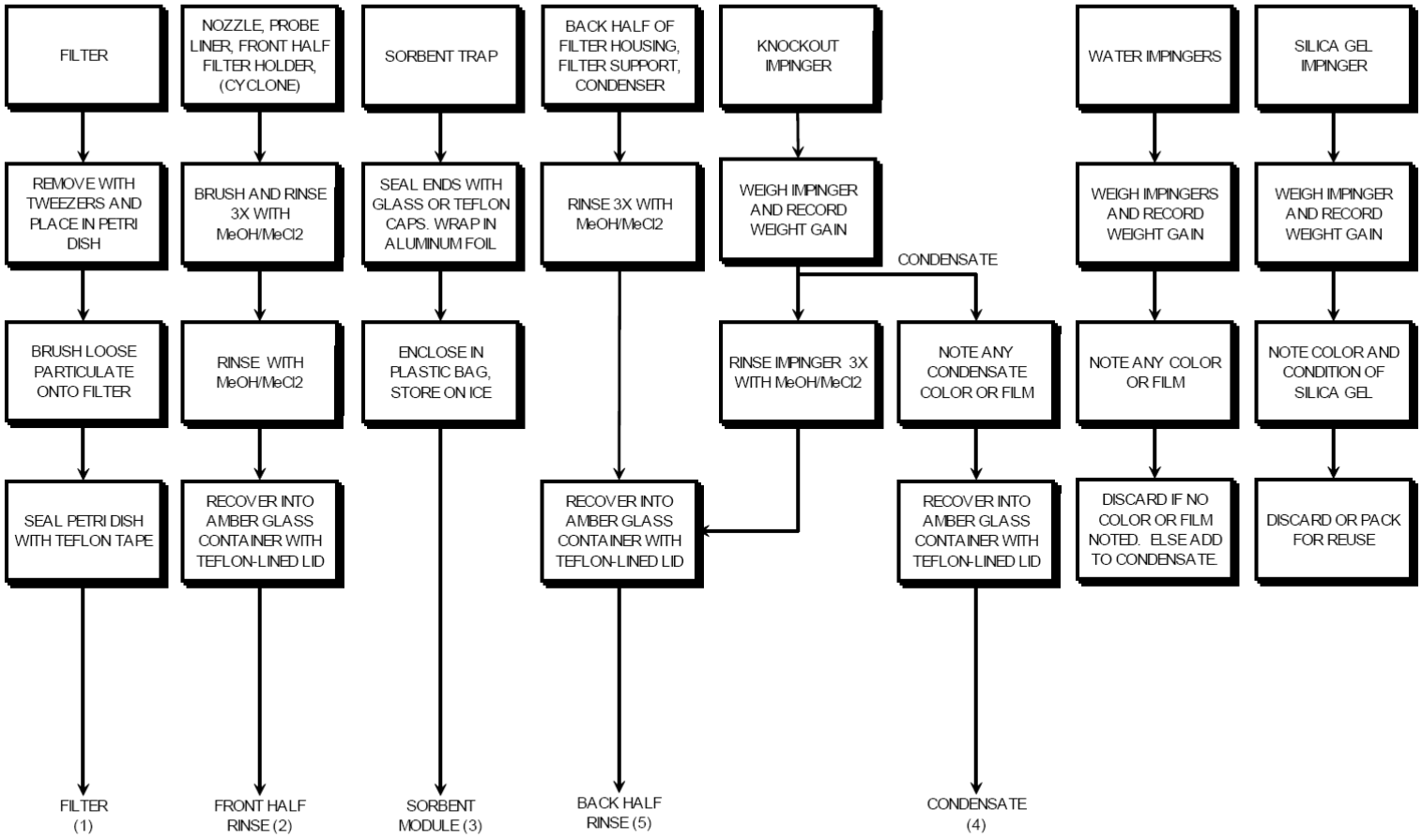


**Figure 2. Method 0010 Semivolatile Organics Sample Recovery**

Table A-6. Stack Gas Continuous Emissions Monitoring Procedures for Carbon Monoxide and Total Hydrocarbons.

| | |
|---|---|
| Sample name: | Stack gas CEMS (CO and THC) |
| Sampler: | Monitoring system operator |
| Equipment: | Extractive type sampling system, carbon monoxide (CO) and total hydrocarbon analyzers as shown in Figure 1 of this table.<br><br>Stack gas extraction probe, filter, condenser, liquid trap, air-to-air heat exchanger, gas analyzers<br><br>Hydrocarbon System: Stack gas extraction probe, heated sample line, filter, gas analyzers |
| Location: | Exhaust stack |
| Frequency: | Continuous during each sampling run. |
| Procedure Summary: | For carbon monoxide and oxygen monitoring, gas samples drawn into the probe are filtered to remove particulate matter, cooled to condense liquids which are captured in a liquid trap, and reheated to slightly above the sample dew point temperature.<br><br>The conditioned gas is continuously monitored by analyzers for carbon monoxide.<br><br>For hydrocarbon monitoring, gas samples are drawn into the probe, transported through a heated sample line, filtered to remove particulate matter, and analyzed directly. |
| References: | "Performance Specification 4B - "Specifications and Test Procedures for Carbon Monoxide and Oxygen Continuous Monitoring Systems in Stationary Sources", Appendix B – Test Methods, New Source Performance Standards, 40 CFR 60.<br><br>"Performance Specification 8A - "Specifications and Test Procedures for Total Hydrocarbon Continuous Monitoring Systems in Stationary Sources", Appendix B – Test Methods, New Source Performance Standards, 40 CFR 60.<br><br>"Method 25A - Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer", Appendix A – Test Methods, New Source Performance Standards, 40 CFR 60. |

# Appendix B

# Analytical Methods

(see Section 9 for list of analytical methods references)

Table B-1. Analysis of Total Metals using Microwave Assisted Acid Digestion of Aqueous Samples & Extracts (Method 3015)

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Procedure Summary:

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

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

The absorbed power is determined by the following relationship:

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

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

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

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

References:

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

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

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

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Table B-2. Analysis of Total Metals using Acid Digestion of Sediments, Sludges, & Soils (Method 3050)

| | |
|---|---|
| Procedure Summary: | Mix sample thoroughly and sieve. Weigh to the nearest 0.01 g and transfer a 1–2 g sample (wet weight) or 1 g sample (dry weight) to a digestion vessel.<br><br>For the digestion of samples for GFAA or ICP-MS, add 10 ml of $HNO_3$, mix the slurry, and cover with a watch glass of vapor recovery device. Heat the sample to 95°C ± 5°C and reflux for 10 to 15 minutes. Allow sample to cool, add 5 ml $HNO_3$, replace cover and reflux an additional 30 minutes. Using a ribbed watch glass or vapor recovery system, allow the solution to evaporate to approximately 5 ml or heat at 95°C ± 5°C for two hours. (Note: Volumes and digestion times will vary if using a direct energy coupling device, such as a microwave.) Cool the sample and add 2 ml of water and 3 ml of $H_2O_2$. Cover the vessel with a watch glass or vapor recovery device and return to heat to begin the peroxide reaction. Heat until effervescence subsides and cool the vessel. Continue to add $H_2O_2$ with warming until effervescence is minimal or general sample appearance is unchanged. Reduce volume to approximately 5 ml of heat at 95°C ± 5°C for two hours. Cool and dilute to 100 ml with water. Filter, centrifuge, or allow to settle as necessary.<br><br>For the digestion of samples for FLAA or ICP-AES, add 2 ml of water and 3 ml of $H_2O_2$. Cover the vessel with a watch glass or vapor recovery device and return to heat to begin the peroxide reaction. Heat until effervescence subsides and cool the vessel. Continue to add $H_2O_2$ with warming until effervescence is minimal or general sample appearance is unchanged. Reduce volume to approximately 5 ml of heat at 95°C ± 5°C for two hours. Add 10 ml of HCl to the peroxide digestate, cover with a watch glass or vapor recovery device and reflux at 95°C ± 5°C for 15 minutes. (Note: Volumes and digestion times will vary if using a direct energy coupling device, such as a microwave.) Filter digestate through Whatman No. 41 filter paper (or equivalent), collect filtrate in a 100 ml volumetric flask, and make to volume.<br><br>Note: Additional steps are provided in this method to improve solubilities and recoveries of antimony, barium, lead, and silver when necessary. |
| References: | Metals Preparation Methods 3050 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.<br><br>Methods 6010, 6020, and 7000 series, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.<br><br>EPA Method 29 Code of Federal Regulations, Title 40, Part 60, Appendix A |

Table B-3. Preparation of Method 29 Stack Gas Samples for Multiple Metals Analyses (Including Mercury)

Matrices:

Particulate filter (Container 1)
Front-half acetone rinse (Container 2)
Front-half nitric acid rinse (Container 3)
Acidified peroxide impinger solutions plus nitric acid rinses (Container 4)
Knockout impinger contents plus nitric acid rinse (Container 5A)
Acidified permanganate impinger solutions plus water rinse (Container 5B)
Acidified permanganate impinger HCl rinse plus water (Container 5C)
Acetone reagent blank (Container 7)
0.1N nitric acid reagent blank (Container 8A)
Deionized water blank (Container 8B)
Acidified $H_2O_2$ impinger solution blank (Container 9)
Acidified $KMnO_4$ impinger solution blank (Container 10)
8N HCl reagent blank (Container 11)
Particulate filter blanks (Container 12)

Procedure Summary:

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

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

Front-half acetone rinse (Container 2) - The acetone rinse is measured to the nearest 1 mL (or to the nearest 0.5 g.) and evaporated to dryness in a 250 mL beaker. When the determination of particulate matter (PM) is required by the test plan, the residue is dried to constant weight and the results are recorded to the nearest 0.1 mg. The front-half acetone rinse residue is then dissolved with 10 mL $HNO_3$ and combined with the front-half nitric acid rinse in Container 3 before it is prepared for digesting.

Front-half nitric acid rinse (Container 3) – The rinse sample pH is checked and adjusted to <2 with $HNO_3$ if necessary. The sample is transferred with water rinse into a beaker with a ribbed watch glass. The sample volume is reduced to approximately 20 mL by heating on a hot plate at a temperature just below boiling. The sample is then digested following the procedures in Method 3010A, Method 3015, or the Parr Bomb operating manual, as appropriate. This digestate is added to the particulate filter digestate and mixed thoroughly. The combined digestate is filtered through a Whatman 541 filter paper and diluted with water to 300 mL (or other appropriate volume depending on the expected concentration). This diluted sample is labeled Analytical Fraction 1A for multiple metals analysis. A 50 mL aliquot of Fraction 1A is transferred to a new sample bottle, which is labeled as Analytical Fraction 1B for mercury analysis.

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

- Sample Fraction 2A - The sample pH is measured and adjusted, if needed, to 2 or less. The sample is transferred with water into a beaker with a ribbed watch glass.

Table B-3. Preparation of Method 29 Stack Gas Samples for Multiple Metals Analyses (Including Mercury)

- The sample volume is reduced to approximately 20 mL by heating on a hot plate at a temperature just below boiling. The sample is then acid digested following procedures in Methods 3010 or 3015, Parr Bomb operating manual, or Method 29.

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

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

Quality Control:

Acetone reagent blank (Container 7) - A 100 mL portion is processed in the same manner as the Container 2 sample. (The acetone sample residue is combined with the nitric acid reagent blank.)

Nitric acid reagent blank (Container 8A) – A 100 mL portion is processed in the same manner as the Container 3 sample. (The acetone residue is added to the nitric acid blank and the mixture is digested.) A separate 100 mL portion of the nitric acid blank is processed in the same manner as Container 5A to produce Analytical Fraction Blank 3A, which is analyzed for mercury. This analytical result is combined with the analytical results from Analytical Blank Fractions 2B, 3B, and 3C as described below for Containers 9, 10, and 11.

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

As an option, two additional particulate filter blanks may be processed in the same manner as provided for in Section 8.1 of Method 0060. The average of the three particulate weights is then used as the front-half blank correction value.

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

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

Table B-3. Preparation of Method 29 Stack Gas Samples for Multiple Metals Analyses (Including Mercury)

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

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

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

References:

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

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

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

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

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

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

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

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

"Method 7470A – Mercury in Liquid Wastes (Manual Cold-Vapor Technique)", Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

Table B-3. Preparation of Method 29 Stack Gas Samples for Multiple Metals Analyses (including Mercury)

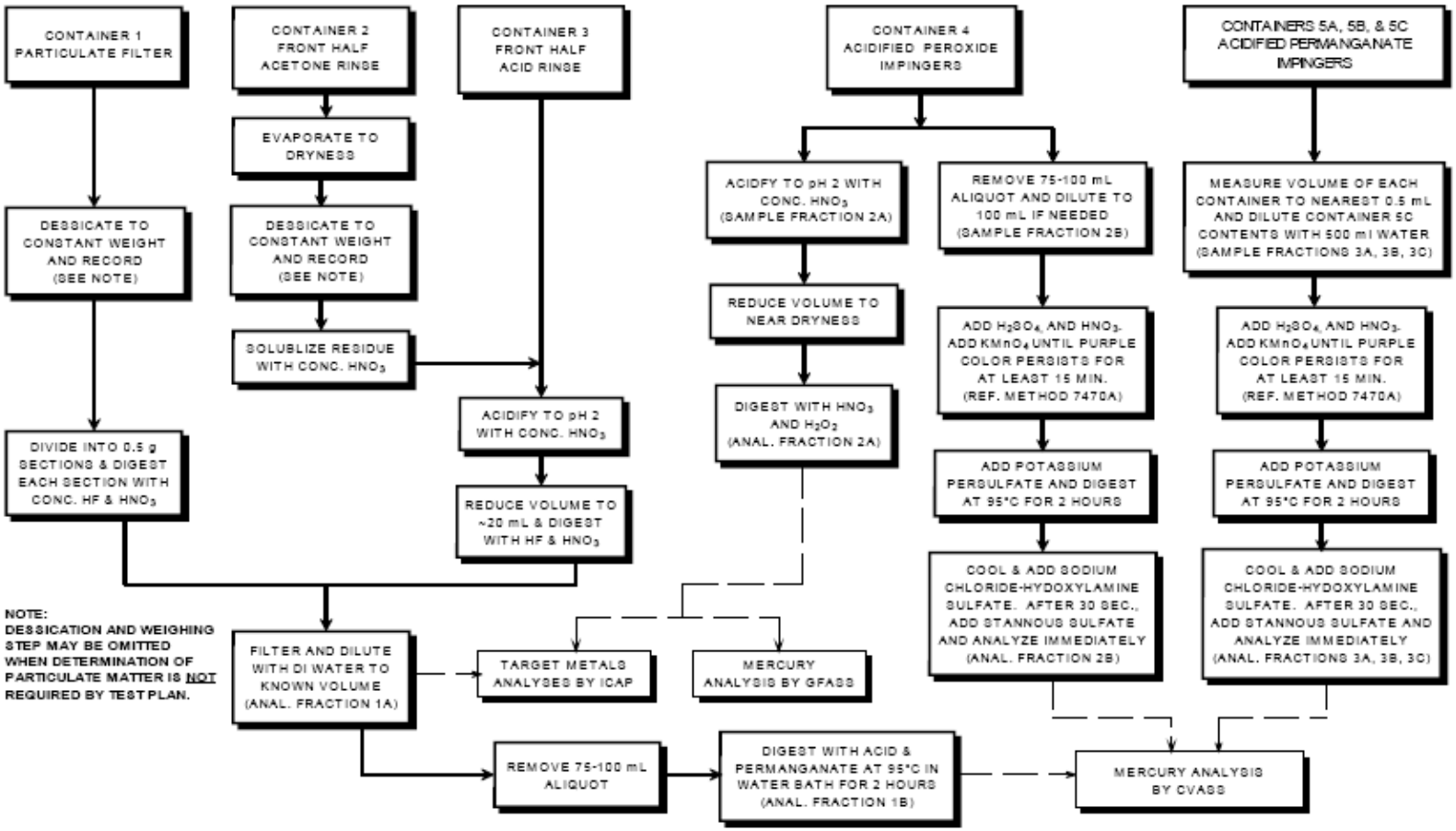


**Figure 1. Preparation of Methods 29 and 0060 Stack Gas Samples for Multiple Metals Analyses (Including Hg)**

Table B-3. Preparation of Method 29 Stack Gas Samples for Multiple Metals Analyses (including Mercury)

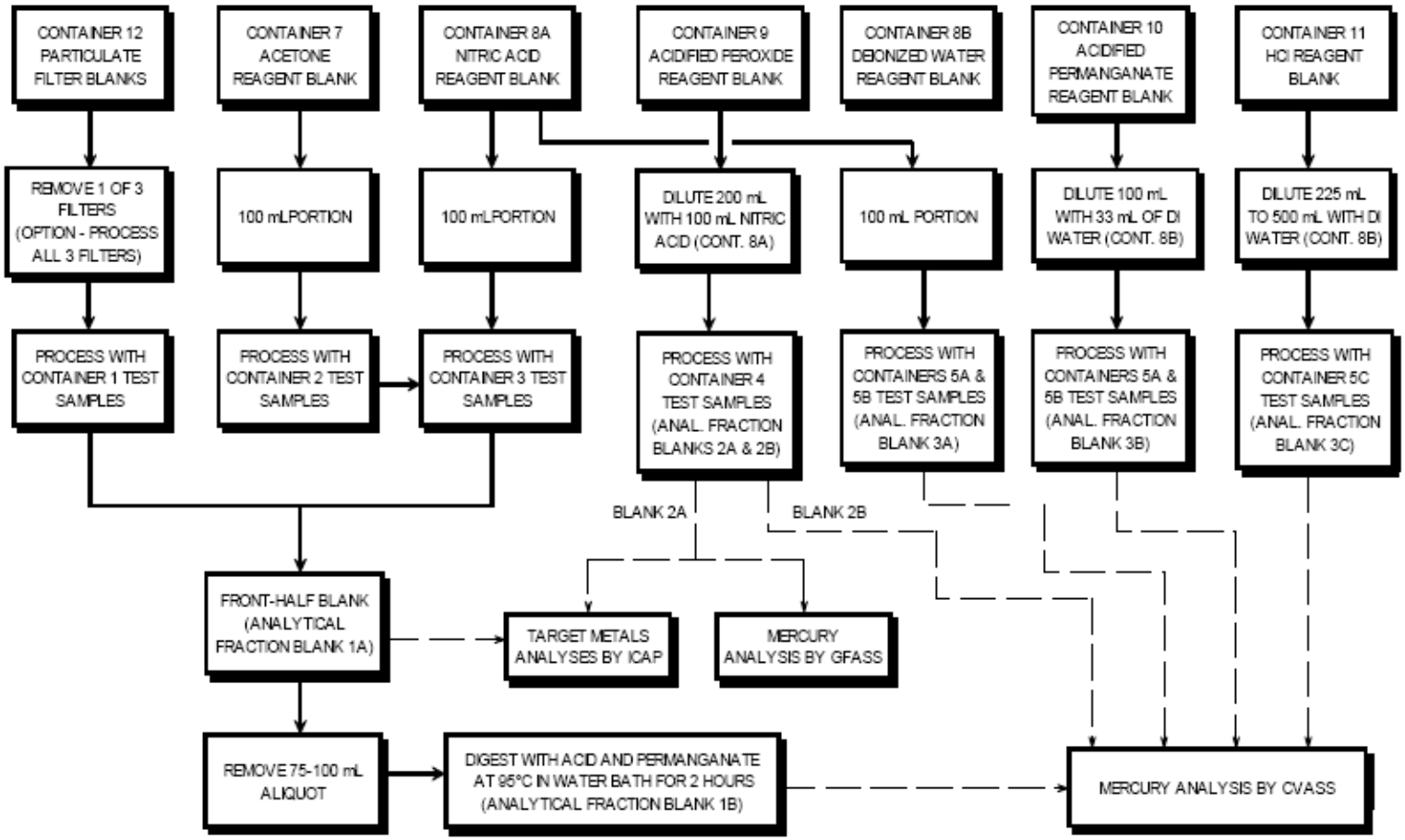


**Figure 2. Preparation of Quality Control Samples for Multiple Metals Analyses (Including Hg)**

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Table B-4. Analysis of Multiple Metals Samples using SW-846 Method 6010 (ICP)

Matrices: SW-846 Method 3015 or 3051 extracats

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

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

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

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

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

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

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

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

Table B-5. Analysis of Mercury Samples using SW-846 Method 7470 (CVAA)

Matrices: Aqueous Samples

Procedure Summary: **SW-846 Methods 7470**

For sample preparation, transfer an aliquot of sample, to a BOD bottle or equivalent. Add $H_2SO_4$ and $HNO_3$. Add $KmnO_4$ and then $K_2(SO_4)_2$ and heat for 2 hours in a water bath maintained at 95°C. Cool and add sodium chloride-hydroxylamine sulfate and then $SnSo_4$ and aerate and then allow to stand quietly without manual agitation.

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

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

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

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

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

References: Mercury in Liquid Waste Method 7470 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates.

Table B-5. Analysis of Mercury Samples using SW-846 Method 7470 (CVAA)

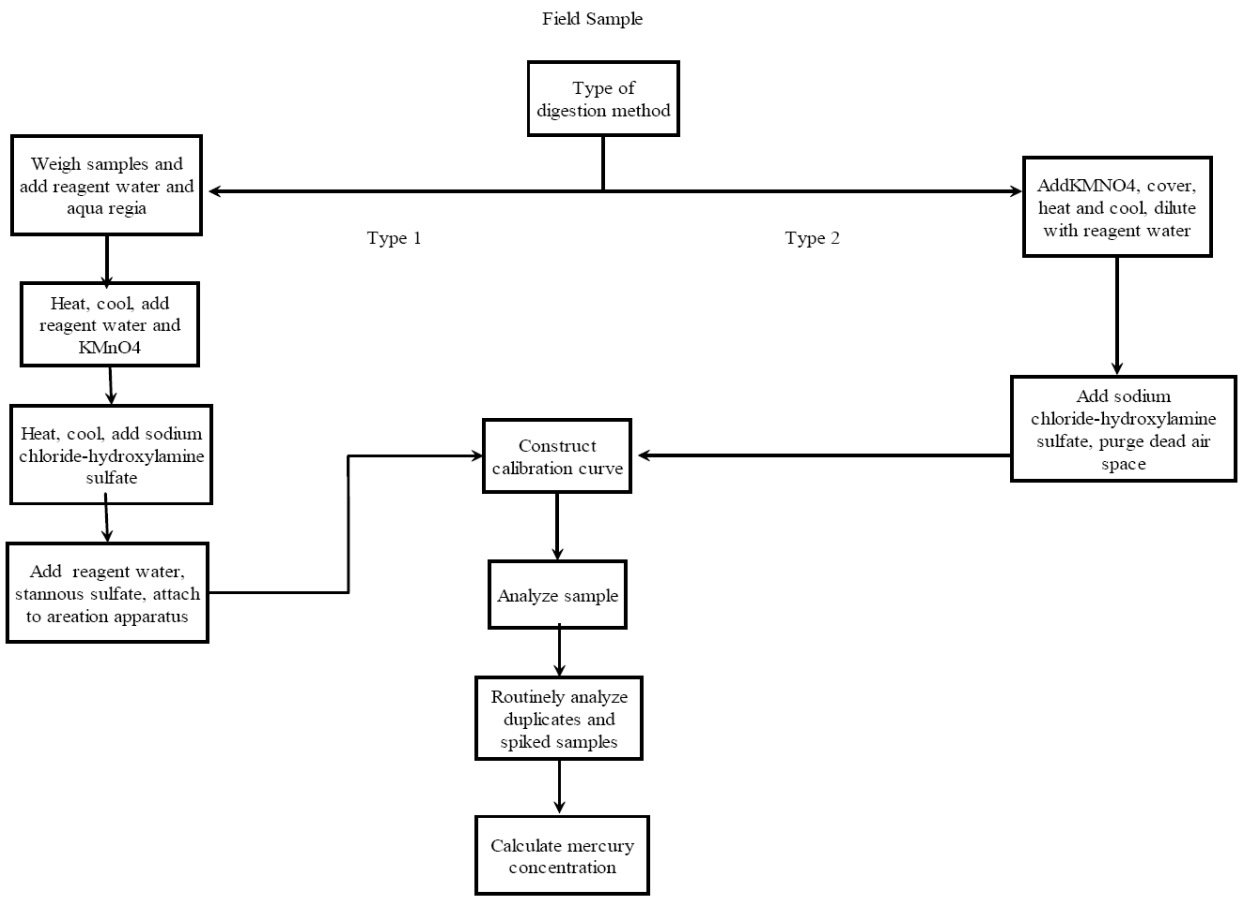


Figure 1. Preparation and Analysis of Methods 7470 Mercury Samples)

Table B-6. Total Chlorine/Chloride Analysis

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

Table B-7. Analysis of Particulate Matter in M5 Samples (Front Half Rinse Only)

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

Table B-8. Analysis of Hydrogen Chloride and Chlorine in M26A Samples

| | |
|---|---|
| Matrices: | Acid Impinger Liquid - sulfuric acid solution for HCl analysis (Container 3)<br>Alkaline Impinger Liquid - sodium hydroxide solution for $Cl_2$ analysis (Container 4) |
| Procedure Summary: | The M26A Impinger samples will be analyzed for hydrogen chloride and chlorine using an ion chromatograph. All chloride ions found in the acid impinger solution are assumed to occur due to the absorption of HCl. All chloride ions found in the alkaline impinger solution are assumed to result from the absorption of $Cl_2$. |
| References: | Method 9057, Protocol for the Analysis of Samples from HCl/$Cl_2$ Emission Sampling Trains, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986, and Updates. |

Table B-9. Analysis of Volatile Organics in SMVOC Samples (Method 0031)

| | |
|---|---|
| Matrices: | SMVOC sorbent resins (Tenax and Anasorb)<br>SMVOC condensate (water) |
| Procedure Summary: | SMVOC tubes will be analyzed separately.<br><br>SMVOC tube contents will be spiked with surrogate and internal standards, thermally desorbed, bubbled through organic-free water, and trapped on an analytical adsorbent trap.<br><br>Target analytes are identified and quantified according to the procedures in SW-846 Method 5041A. The analysis will be extended to identify and quantify as many non-target analytes as possible. These tentatively identified compounds (TICs) will include all non-target peaks that are at least 10% of the nearest internal standard, and the identification and quantitation will be made using EPA procedures for TICs. This includes forward and reverse library searches using a response factor of 1 for the nearest internal standard, unless a specific response factor can be determined and used for quantitation. Analysis of the tubes will be performed using GC/MS following SW-846 Method 5041A for volatile organics on SMVOC.<br><br>Condensate samples will be spiked with surrogate and internal standards and analyzed by GC/MS directly using a purge-and-trap device following SW-846 method 8260B. The TIC analysis will also be performed on the condensate sample as described above for the SMVOC tubes. |
| References: | Method 5041A, Protocol for Analysis of Sorbent Cartridges from Volatile Organic Sampling Train: Wide-bore Capillary Column Technique, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1986, and Updates.<br><br>Method 8260B, Volatile Organics by GC/MS, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, September 1986, and Updates. |

Table B-10. Analysis of M0023 Samples for PCDD/PCDF
(Separate Front Half and Back Half Samples)

Matrices:

Particulate Filter (Container 1)
Front half rinse - acetone, methylene chloride, and toluene (Container 2)
Adsorbent module (Container 3)
Back half rinse -acetone, methylene chloride, and toluene (Container 4)
Reagent blanks -acetone, methylene chloride, and toluene reagent blanks (archive)
Adsorbent resin module trip blank (archive)
Particulate filter trip blank (archive)

Procedure Summary:

Exactly 1.0 mL of the surrogate spiking solution is added uniformly onto the surface of the particulate filter (Container 1). The filter is then transferred to the extraction thimble of a Soxhlet extraction system.

The front half rinse (Container 2) is concentrated to 1-2 mL using a Kuderna-Danish (K-D) concentrator apparatus followed by nitrogen blow down at a temperature of less than 37°C. The front half rinse sample container is rinsed three times with small portions of methylene chloride and these rinses are added to the K-D concentrate and the mixture is further concentrated to near dryness. This concentrate is added to the particulate filter in the extraction thimble. Internal standards are added to the thimble and the thimble contents are extracted for 16 hours. The Soxhlet extract is concentrated to about 10 mL in a rotary evaporator and to 10 µL in a nitrogen evaporative concentrator. The concentrated extract is dissolved in 5 mL of hexane to form the front half sample (Sample 1).

The XAD resin is transferred from the adsorbent module (Container 3) to a separate extraction thimble using toluene to rinse the glass module and the rinse catch beaker. The glass wool plug is added to the thimble.

The back half rinse (Container 4) is concentrated to 1-2 mL using a K-D apparatus followed by nitrogen blow down at a temperature of less than 37°C. The back half rinse sample container is rinsed three times with small portions of methylene chloride and the rinses are added to the K-D concentrate and the solution is further concentrated to near dryness. The K-D concentrate is added to the thimble (which contains the resin, container rinses, and glass wool plug). Internal standards are added to the thimble and the thimble contents are extracted with toluene for 16 hours. The Soxhlet extract is then concentrated to about 10 mL in a rotary evaporator and to 100 µL in a nitrogen evaporative concentrator. The concentrated extract is then dissolved in 5 ml of hexane to form the back half sample (Sample 2).

Sample 1 (front half) and Sample 2 (back half) are each spiked with 40 µL of alternate standards and divided into two equal portions. One portion of each sample is archived for possible repeat analysis if needed. The other portions are subjected to the chromatographic cleanup steps of Method 8290.

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

Quality Control:

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

Table B-10. Analysis of M0023 Samples for PCDD/PCDF
(Separate Front Half and Back Half Samples)

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

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

Table B-10. Analysis of M0023 Samples for PCDD/PCDF
(Separate Front Half and Back Half Samples)

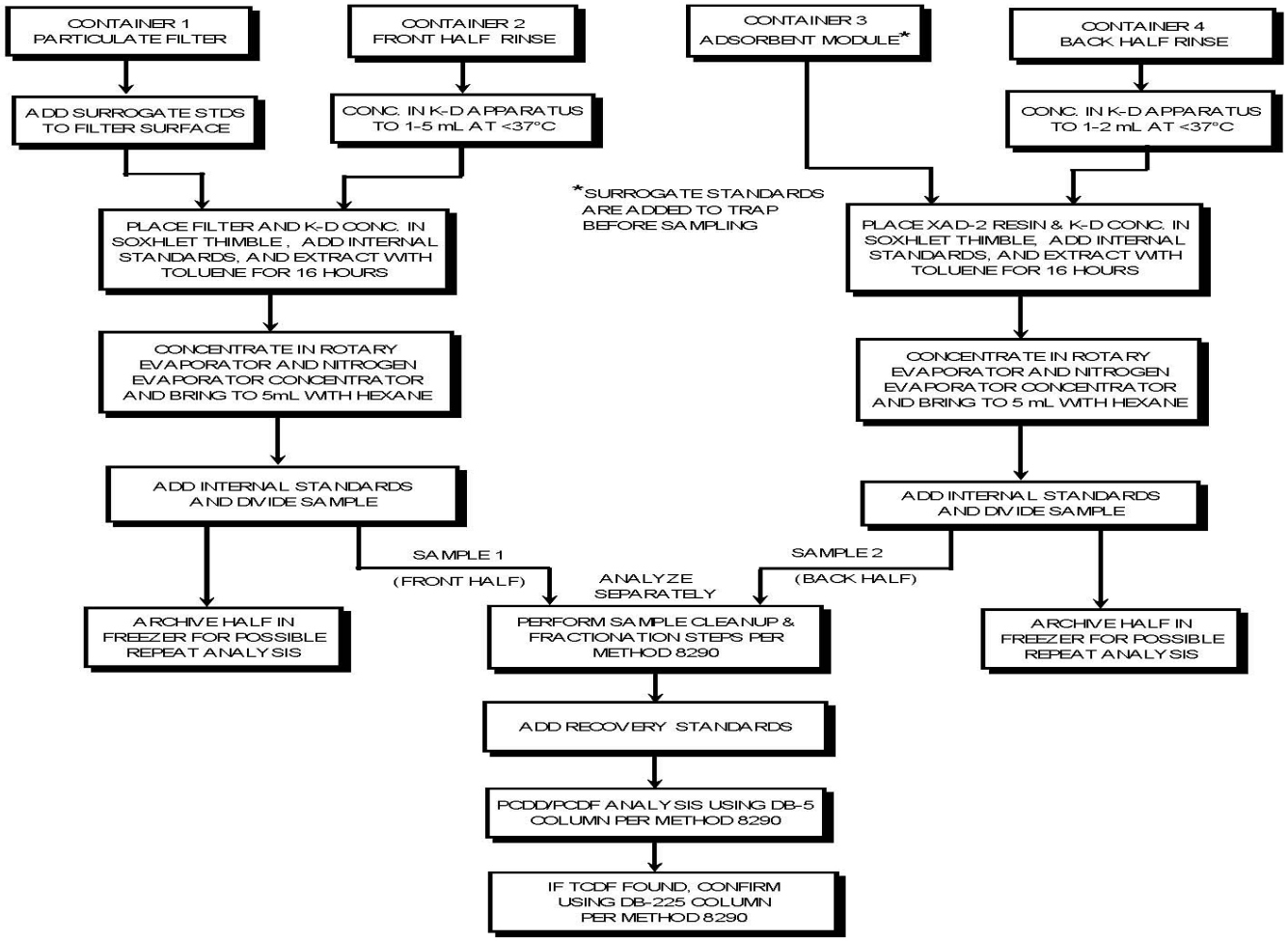


**Figure 1. Recovery, Preparation, and Analysis of M0023A Samples for PCDD/PCDF**

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Table B-11. Analysis of Method 0010 Samples for SVOCs using SW-846 Methods 3542/8270C

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:

**SAMPLE PREPARATION**

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 (Container No. 2) 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 methanol and methylene chloride adding the rinses to the same amber glass jar. Store this portion at 4°C until the front half rinse extraction is completed.

Transfer the liquid contents of the filtered front half rinse (Container No. 2) to a separatory funnel for extraction (Method 3510 with exceptions as noted). Rinse the sample container three times with methylene chloride transferring the rinse to the funnel. Add sufficient organic free water to the funnel to cause the organic and aqueous/methanol phases to separate into two distinct layers. The methylene chloride layer will be at the bottom. Continue to add water until the methylene chloride layer does not increase in volume. Add additional methylene chloride, if necessary, so that the ratio of water/methanol to methylene chloride is approximately 3:1. Add sodium hydroxide until the pH of the water layer is > 11 but <14. Shake vigorously for 2 minutes with periodic venting to release excess pressure. Allow the organic layer to separate for at least 10 minutes. Collect the methylene chloride in an amber glass bottle. Add a volume of methylene chloride (approximately equal to the volume of the first extraction) to the separatory funnel and repeat the extraction collecting the methylene chloride in the same amber glass bottle. Repeat the extraction a third time collecting the methylene chloride. Acidify the water remaining after the third extraction with sulfuric acid to a pH of <2 but > 0. Repeat the methylene chloride extraction on the acidified sample three times collecting the methylene chloride in an amber glass bottle.

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

Table B-11. Analysis Preparation of Method 0010 SVOC Samples using SW-846 Method 3542 (Continued)

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

To perform the final concentration, remove the Snyder column and evaporative flask. Add new boiling chips to the concentrator tube. Attach a two-ball micro Snyder column to the concentrator tube. Attach the solvent vapor recovery glassware to the Snyder column. Prewet the Snyder column with about 0.5 ml of methylene chloride. Place the K-D apparatus in a hot water bath. When the apparent volume reaches 4 –5 ml remove the K_D apparatus and allow to cool and drain for at least 10 minutes. If the volume is 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$^{o}$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

Table B-11. Analysis Preparation of Method 0010 SVOC Samples using SW-846 Method 3542 (Continued)

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 equal portions. One portion will be sent to the freezer for archive and the other portions will be used for individual analyses, as prescribed in the test plan.

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.

Procedure Summary:

**SEMIVOLATILE ORGANIC ANALYSIS- METHOD 8270C**

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

Quality Control:

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

Table B-11. Analysis Preparation of Method 0010 SVOC Samples using SW-846 Method 3542 (Continued)

processed in a manner identical to the field sample. The XAD-2 resin used for the method blank will be obtained from the same batch used for preparation of the field traps.

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

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

References: 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

Method 8270C, Semivolatile Organics by Gas Chromatography, Mass Spectrometry (GC/MS), Test Methods for Evaluating Solid Waste, SW-846, Third Edition, November1986, and Updates.

# Appendix C

# Quality Assurance Project Plan

# QUALITY ASSURANCE PROJECT PLAN FOR THE SYSTEM PERFORMANCE TEST OF THE IDAHO NUCLEAR TECHNOLOGY AND ENGINEERING CENTER INTEGRATED WASTE TREATMENT UNIT

## 1.0 QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM AND DISTRIBUTION LIST

**Project Title:** Idaho Nuclear Technology and Engineering Center, (INTEC),
System Performance Test (SPT) for the Integrated Waste Treatment Unit (IWTU)
Idaho Falls, Idaho

**Expected SPT Date:** January 2012

**Project Approvals:**

| Name/Function/Organization | Signature | Date |
|---|---|---|
| SPT Team Lead | | |
| Test Manager | | |
| Sampling Contractor Lead | | |
| Technical Advisor | | |
| Laboratory Analysis Coordinator | | |
| Quality Assurance Officer | | |

Quality Assurance Project Plan (QAPjP) Approval Statement: The individuals listed above:

1) Have received, read, and agreed to the appropriate information pertaining to assigned project responsibilities listed and provided in this QAPjP, and

2) Agree that no testing methods will be modified. If any sampling and analytical methods are modified as specified in the associated SPT plan, they are to be identified and explained in the sampling and analytical narratives, and discussed in the body of the test report. Approval for modifications to the SPT-specified sampling and analytical methods will be requested as appropriate from the Idaho Department of Environmental Quality (DEQ).

**QAPjP Distribution List**

| Project Organization Title | Organization/Name | No. of Copies |
|---|---|---|
| SPT Team Lead | INTEC | 1 |
| Test Manager | Test Management Contractor | 1 |
| Sampling Contractor Lead | Sampling Contractor | 1 |
| Laboratory Analysis Coordinator | Contract Analytical Laboratory | 1 |
| Quality Assurance Officer | Sampling Contractor | 1 |
| Idaho Department of Environmental Quality | DEQ | 3 |
| U.S. Environmental Protection Agency (EPA) | EPA Region 10 | 2 |

The QAPjP was distributed to these individuals, was read by the individuals, and they agree to the appropriate information pertaining to their project responsibilities as listed and provided in this QAPjP.

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**ACRONYMS AND ABBREVIATIONS**

| | |
|---|---|
| ASTM | American Society for Testing Materials |
| CEM | continuous emissions monitor |
| CEMS | continuous emissions monitoring system |
| CFR | Code of Federal Regulations |
| $Cl^-$ | chloride ion |
| $Cl_2$ | molecular chlorine |
| CMS | continuous monitoring system |
| COC | chain of custody |
| CRR | Carbon Reduction Reformer |
| DEQ | Idaho Department of Environmental Quality |
| DMR | Denitration Mineralization Reformer |
| DOE | Department of Energy |
| DQO | data quality objective |
| dscf | dry standard cubic foot |
| dscm | dry standard cubic meter |
| EDL | estimated detection limit |
| EPA | U.S. Environmental Protection Agency |
| GC/MS | gas chromatography/mass spectrometry |
| g | gram |
| gr | grain |
| HC | hydrocarbon |
| HCl | hydrogen chloride (gas) or hydrochloric acid (aqueous) |
| Hg | mercury |
| HEPA | high-efficiency particulate air |
| HWC-MACT | Hazardous Waste Combustor-Maximum Achievable Control Technology |
| HWMA | Hazardous Waste Management Act |
| ICP | inductively coupled argon plasma spectroscopy |
| INTEC | Idaho Nuclear Technology and Engineering Center |
| IWTU | Integrated Waste Treatment Unit |
| L | liter |
| L/min | liters per minute |
| lb | pound |
| $m^3$/min | cubic meters per minute |
| MDL | method detection limit |
| mg | milligram |
| μg | micrograms |
| μg/$m^3$ | micrograms/cubic meter |
| min | minute |
| mL | milliliter |
| mm | millimeter |
| MS | matrix spike |
| MSD | matrix spike duplicate |
| NA | not applicable |

| | |
|---|---|
| NFM | Nuclear Facility Manager |
| ηg | nanogram |
| PAH | polyaromatic hydrocarbon |
| PCDD | polychlorinated dibenzo-p-dioxin |
| PCDF | polychlorinated dibenzofuran |
| PDS | post-digestion spike |
| PE | performance evaluation |
| PM | particulate matter |
| ppmv | parts per million dry volume |
| QA | quality assurance |
| QAO | quality assurance officer |
| QAPjP | Quality Assurance Project Plan |
| QC | quality control |
| RCRA | Resource Conservation and Recovery Act |
| RDL | reliable detection limit |
| RF | response factor |
| RFA | request for analysis |
| RL | reporting limit |
| RPD | relative percent difference |
| RSD | relative standard deviation |
| SMVOC | sampling method for volatile organic compounds |
| SOP | standard operating procedure |
| SPT | System Performance Test |
| SQL | sample quantitation limit |
| SVOC | semivolatile organic compound |
| SW846 | *Test Methods for Evaluating Solid Wastes Physical/Chemical Methods (SW-846)*, Third Edition, 1986 and updates (December 1997) |
| 2,3,7,8-TCDD | 2,3,7,8-tetrachlorodibenzo-p-dioxin |
| THC | total hydrocarbons |
| THOR® | Thermal Organic Reduction |
| TIC | tentatively identified compound |
| VOC | volatile organic compound |

# 3.0 PROJECT DESCRIPTION

## 3.1 GENERAL

This Quality Assurance Project Plan (QAPjP) is an integral part of the System Performance Test (SPT) plan for the Idaho Nuclear Technology and Engineering Center (INTEC) Integrated Waste Treatment Unit (IWTU) located at the Idaho National Laboratory Site, and must be used with this plan.

## 3.2 QUALITY ASSURANCE PROJECT PLAN SCOPE

This QAPjP presents the organization, objectives, functional activities, and specific quality assurance (QA) and quality control (QC) activities for the IWTU SPT. This QAPjP also describes the specific QA/QC protocols that will be followed for sampling, sample handling and storage, chain of custody, and laboratory analysis during the SPT.

All QA/QC procedures will be in accordance with applicable professional technical standards, government regulations and guidelines, and specific project goals and requirements. The SPT plan and this associated QAPjP have been prepared in accordance with U.S. Environmental Protection Agency (EPA) hazardous waste thermal treatment system testing and QAPjP guidance documents, in particular the following:

- *Requirements for Quality Assurance Project Plans*, EPA QA/R-5 EPA/240/B-01/003, EPA, March 2001
- *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*, QAMS-005/80, EPA, 1983
- *Guidance for Quality Assurance Project Plans*, EPA QA/G-5, EPA/240/R-02/009, December 2002
- "National Emission Standards for Hazardous Air Pollutants from Hazardous Waste Combustors," 40 Code of Federal Regulations (CFR) 63, Subpart EEE, February 2010
- *Annual Book of ASTM Standards*, American Society for Testing and Materials, latest annual edition
- "New Source Performance Standards, Test Methods and Procedures," 40 CFR 60, Appendix A, EPA, December 2009
- *Test Methods for Evaluating Solid Wastes Physical/Chemical Methods*, SW-846, Third Edition, EPA, 1986 and updates
- *Human Health Risk Assessment Protocol for Hazardous Waste Combustion Facilities*, EPA 530D-98-001C, EPA Region 6, July 1998.

## 3.3 FACILITY BACKGROUND INFORMATION

INTEC, located at the Idaho National Laboratory Site, is a Department of Energy (DOE) facility that was previously used to reprocess nuclear fuel. INTEC currently has approximately 840,700 gallons of liquid radioactive waste in storage. This waste is comprised of acid decontamination solutions that contain high levels of sodium. This waste also contains toxic metals and trace concentrations of organics that are regulated under the Hazardous Waste Management Act (HWMA)/Resource Conservation and Recovery Act (RCRA).

Based upon an agreement with the Idaho Department of Environmental Quality (DEQ), DOE is preparing to treat this waste. CH2M-WG Idaho, LLC has been contracted by DOE to build and operate the IWTU to

process this waste. The IWTU has been constructed at INTEC and will use a patented steam reforming technology. This technology was selected because it will immobilize the radionuclides, sodium, potassium, sulfate, chlorides, fluorides, and nonvolatile heavy metals into a stable granular solid product, while destroying nitric acid, nitrates, and organic compounds present in the waste.

The process converts nitrates and nitrites directly to nitrogen gas. Organic material in the waste is converted to carbon dioxide and water vapor. The off-gas (mainly carbon dioxide, water, nitrogen, and oxygen) from the process is filtered through sintered metal filters, high-efficiency particulate air (HEPA) filters, and sulfur-impregnated granulated activated carbon. The filtered off-gas will then be vented to the atmosphere through a stack. The facility is defined as a miscellaneous treatment unit and has received a HWMA/RCRA Part B Permit.

### 3.4 WASTE FEED AND TREATMENT PROCESS OVERVIEW

Radioactive liquid waste has been generated over the last five decades at INTEC, formerly called the Idaho Chemical Processing Plant, as a result of nuclear fuel reprocessing activities. From December 1963 until June 2000, the Waste Calcining Facility and the New Waste Calcining Facility processed the liquid waste into a granular, solid form. As of April 1, 2011, approximately 840,700 gallons of waste remained in the Tank Farm Facility at INTEC. Waste in the Tank Farm Facility is referred to as sodium-bearing waste.

The IWTU is designed to treat liquid sodium-bearing waste to produce a solid treatment product for ultimate disposal. The IWTU will utilize steam reforming technology, which includes a dual fluidized-bed process that uses superheated steam, carbon, and other additives to convert the sodium-bearing waste into a solid, granular treatment product that is packaged into canisters suitable for ultimate disposal. The process is named the IWTU because two fluidized-bed steam reformers, the Denitration Mineralization Reformer (DMR) and the Carbon Reduction Reformer (CRR), are integrated into a single treatment process with a common air pollution control system. The DMR, CRR, and material transfer and loadout systems utilize filters that are integral to the processing system used to capture and package the solid treatment product; these filters are not part of the air pollution control system. The IWTU air pollution control system includes the process HEPA filter system (which is located downstream from the DMR and CRR) and the building ventilation HEPA filter system.

More engineering details can be found in Section 3.0 of the SPT plan.

### 3.5 TESTING REQUIREMENTS

The objectives of the SPT are to gather emissions data, while operating at "bounding" conditions processing mixed waste feed materials to establish operating conditions that will be included in the HWMA/RCRA permit. More specifically, the objectives of the SPT are as follows:

1. Evaluate emissions performance against the following targets:

   - Particulate emission concentration less than or equal to 0.0016 grains per dry standard cubic foot (gr/dscf), corrected to 7% oxygen

   - Emission concentration of dioxins and furans not exceeding 0.11 ηg/dscm, expressed as toxic equivalents of 2,3,7,8-tetrachlorodibenzo-p-dioxin toxicity equivalents, corrected to 7% oxygen

   - Mercury emission concentration less than or equal to 8.1 micrograms/dry standard cubic meter (μg/dscm), corrected to 7% oxygen

   - Emission concentration of semivolatile metals (cadmium and lead, combined) less than or equal to 10 μg/dscm, corrected to 7% oxygen

- Emission concentration of low volatility metals (arsenic, beryllium, and chromium, combined) less than or equal to 23 μg/dscm, corrected to 7% oxygen
- Carbon monoxide emissions concentration less than or equal to 100 ppmv, dry basis, hourly rolling average, corrected to 7% oxygen
- Hydrocarbon (HC) emission concentration of less than or equal to 10 ppmv, as propane, dry basis, hourly rolling average, corrected to 7% oxygen.

2. Establish permit operating limits:
   - Demonstrate maximum waste feed rate
   - Demonstrate maximum process gas flow rate
   - Establish appropriate operating limits for the process and off-gas treatment system components.
3. Gather information for evaluating risk:
   - Measure emissions of specific volatile and semivolatile organic emission products, plus tentatively identified volatile and semivolatile organics (tentatively identified compounds [TICs])
   - Measure emissions of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDF)
   - Measure emissions of representative volatile, semivolatile, and nonvolatile metals.
4. Demonstrate nitrogen oxides emissions performance for air permit compliance.

### 3.6 TEST OPERATING AND SAMPLING PROTOCOLS

The proposed SPT is comprised of two test conditions, each with four replicate sampling runs. (Three sampling runs are required by regulation. The fourth run was added as a contingency.) During the SPT, the IWTU will be operated at maximum waste feed rate, maximum process gas flow rate, minimum DMR and CRR temperatures, and "bounding" conditions for the remainder of the process components. The two test conditions are identical, except the target DMR temperature. The first test condition will target a minimum DMR temperature that is expected to provide compliant performance with a very high degree of confidence. The second test condition will target a somewhat lower DMR temperature, which would provide more operating flexibility for the system.

The sampling and analyses during these test conditions are summarized in Table 3-1.

### 3.7 PLANNED TEST DATE

The SPT is expected to occur in January 2012.

**Table 3-1. Sampling and Analysis for the IWTU (Both Test Conditions)**

| Sample Name | Sampling Location/ Access | Sampling Equipment | Sampling Reference Method | Sample Size/Frequency | Analytical Parameters | Analytical Reference Method [a] |
|---|---|---|---|---|---|---|
| Process off-gas | Nonisokinetic port | SMVOC | SW846 Method 0031 (SMVOC) | 4 tube sets per run; 40 minutes per tube set. Up to 20 liters of gas per tube set | Target VOCs and TICs | SW846 Method 5041A, 8260B |
| Process off-gas | Isokinetic ports | Method 0023A Sampling Train | EPA Method 0023A | Minimum 3 dscm[b,c] (~4-hour run) | PCDD/PDCFs | SW846 Method 8290 |
| Process off-gas | Isokinetic ports | Method 0010 Sampling Train | SW846 Method 0010 | Minimum 3 dscm[b,c] (~4-hour run) | Target SVOCs and TICs and target PAHs | SW846 Method 3015 & 3542, 8270C |
| Process off-gas | Isokinetic ports | Method 29 Sampling Train | EPA Method 29 | 60 minutes[b,c] | Metals: Sb, As, Ba, Be, Cd, total Cr, Co, Pb, Mn. Ni, Se, Ag, & Tl<br>Hg | SW846 Method 3050, 6010<br>SW846 Method 7470 |
| Process off-gas | Isokinetic ports | Method 26A | EPA Method 26A | As recommended in method guidance[b,c] | Particulate HCl/$Cl_2$ | EPA Method 5<br>SW846 Method 9057 |
| Process off-gas | CEMS port | Installed CO and $O_2$ CEMS | 40 CFR 60, Appendix B, Specification 4B | Continuous | CO, $O_2$ | 40 CFR 60, Appendix B, Specification 4B |
| Process off-gas | Nonisokinetic port | Temporary THC CEMS | 40 CFR 60, Appendix B, Method 8A | Continuous | THC | 40 CFR 60, Appendix B, Method 8A |

Notes:
[a] Reference Method Sources:
"ASTM" refers to *American Society for Testing Materials, Annual Book of ASTM Standards*, Annual Series.
"SW846" refers to *Test Methods for Evaluating Solid Waste*, Third Edition, November 1986 and Updates.
"EPA Method" refers to *New Source Performance Standards*, Test Methods and Procedures, Appendix A, 40 CFR 60.
[b] The exact volume of gas sampled will depend on the isokinetic sampling rate.
[c] Sampling train operation includes measuring process off-gas: velocity, pressure, and temperature (EPA Method 2); moisture (EPA Method 4); and oxygen and carbon dioxide concentration (EPA Method 3).

## 4.0 ORGANIZATION OF PERSONNEL, RESPONSIBILITIES, AND QUALIFICATIONS

### 4.1 GENERAL

The project organization for this SPT is summarized in Section 1.7 of the SPT plan. During the test, the test manager will be responsible for ensuring that the processes run properly and that the unit achieves the desired test conditions on each test day. As such, the test manager, working through the nuclear facility manager NFM), will recommend targets for the unit operations. The test manager will be responsible for ensuring that all the applicable process data are collected during each test run.

The sampling contractor lead and quality assurance officer (QAO) from the sampling contractor are experienced in the technical coordination and QA/QC associated with testing thermal treatment systems. The sampling contractor is experienced in conducting the sampling called for in the SPT and will conduct the sampling for this SPT. The analytical laboratory is experienced in the analysis of emissions and process samples, and will provide analytical services for this SPT.

Sampling contractor and analytical laboratory information will be submitted to the DEQ as a separate submittal.

### 4.2 SPT TEAM LEAD

The SPT team lead is responsible for the overall coordination of the SPT. Specific team lead responsibilities include:

- Overall responsibility for the safe completion of the SPT
- Review of the final results and test reports and submittal to the DEQ.
- Preparation of the final HWMA/RCRA permit modification request and submittal to DEQ

### 4.3 TEST MANAGER

The test manager is responsible for the execution of the SPT, QAPjP, preparation of the final test report, and interpretation of the test results. During the SPT, the test manager is responsible for the overall implementation of the SPT. The test manager will serve as the focal point between the sampling contractors on testing-related matters. Specific test coordinator responsibilities include:

- Ensuring compliance with the SPT plan and the QAPjP by all project team members during the test
- Documenting testing activities in a field logbook
- Providing sample checklists, labels, and request for analysis (RFA) and chain of custody (COC) forms for use by the sampling contractor and the process sample coordinator
- Assisting the team lead in interfacing with the regulatory observers and oversight contractors
- Coordinating among the sampling teams during the SPT, especially regarding decisions to start, stop, hold, or repeat sampling runs
- Inspecting the process equipment, process controls, process operations, data acquisition and recording systems, and sampling activities for compliance with this QAPjP and the SPT plan

- Providing field review of process operating logs, completed sample collection sheets, sampling logs, COC forms, and RFA forms
- Interfacing with the laboratory analysis coordinator while samples are being analyzed
- Supervising production of the test reports
- Certifying the overall test results and the final test reports
- Preparing operating specifications for the system based on the results of the test.

### 4.4 SAMPLING CONTRACTOR LEAD

The sampling contractor lead's responsibilities include the following:

- Provide oversight of the sampling and direction to the sampling team to ensure safe completion of sampling activities and sample recovery
- Interface with the test manager to obtain direction for sampling activities.

### 4.5 QUALITY ASSURANCE OFFICER

The QAO's responsibilities include the following:

- Ensuring all individuals included in the QAPjP distribution list receive current copies of revisions, as applicable
- Reviewing the sampling and analytical reports for completeness and accuracy
- Conducting or coordinating any required audits of the data reduction or laboratory procedures to ensure compliance with the QAPjP
- Validating analytical data generated for completeness of the reports, including documentation of the required QA/QC analyses and corrective actions
- Preparing a report of the QA/QC activities that summarizes the findings, including a statement for inclusion in the SPT report executive summary regarding if any of the test data are invalid or unusable.

### 4.6 PROCESS SAMPLING COORDINATOR/SAMPLE CUSTODIAN

A process sampling coordinator/sample custodian will be appointed who will have overall responsibility for the collection and handling of all test samples. The process sampling coordinator has the following responsibilities:

- Coordinating the preparation and shipment of process sampling equipment to the test site
- Either or both directing or participating in process sampling activities
- Recording field test data required by the process sampling methods
- Reviewing and approving process sample collection sheets and field data sheets prepared by others

- Overseeing recovery of process samples and preservation of process samples in the field
- Documenting all required process samples are collected
- Performing all QA activities required by the process sampling methods
- Preparing a draft and final report of process sampling activities.

The sample custodian has the following responsibilities:

- Controlling issuance of sample containers at the test site
- Packaging the process and process off-gas samples, ensuring that the samples are preserved during shipment to the laboratory
- Preparing COC and RFA forms for all samples or reviewing the same test documentation prepared by other test team members
- Coordinating the shipment of all samples to the laboratory
- Monitoring the shipment of samples to the laboratory to ensure that all samples are received on schedule and with all preservation requirements being met. Any discrepancies should be immediately reported to the QAO, test coordinator, and INTEC test project manager.

The process sampling coordinator/sample custodian will be an employee of the sampling contractor or test management contractor.

### 4.7 SAMPLING TEAM LEADER

The sampling team leader will have overall responsibility for collecting and handling all process off-gas-related samples. The sampling team leader has the following oversight responsibilities:

- Preparing and shipping sampling equipment to the test site
- Preparing and calibrating sampling equipment
- Either or both directing or participating in sampling activities
- Recording field test data required by the sampling methods
- Reviewing and approving sample collection sheets and sampling field data sheets
- Overseeing recovery of sampling-related samples and preservation of those samples
- Notifying the sample custodian of all samples collected
- Reducing sampling data and performing all calculations and QA activities required by the sampling methods
- Ensuring the isokinetic calculations are independently verified daily and presenting these findings at the daily brief
- Preparing a draft and final report of sampling activities.

### 4.8 INTEC PROCESS OPERATIONS

The process operations will be responsible for the operation of the IWTU. Their duties will include:

- Maintaining the IWTU within specified target limits
- Maintaining logs of process data, as required
- Downloading and providing the IWTU one-minute operating data to the test coordinator in Microsoft Excel or ASCII format

### 4.9 LABORATORY ANALYSIS COORDINATOR

The laboratory analysis coordinator will have overall responsibility for the analysis of all process and process off-gas-related samples. The laboratory analysis coordinator has the following responsibilities:

- Receiving, verifying, and documenting that incoming field samples correspond to the sample COC information
- Notifying the sample custodian, QAO, test coordinator, and test manager of any discrepancies or problems in the COC and RFA information, preservation, or sample condition
- Maintaining records of incoming samples
- Tracking samples through processing, analysis, and disposal
- Designating QC samples for analysis during the project
- Verifying that laboratory personnel are trained and qualified in specified laboratory QC and analytical procedures
- Verifying that laboratory QC and analytical procedures are being followed as specified in this QAPjP, the laboratory-specific QA/QC plan, and the laboratory-specific analytical standard operating procedures (SOPs)
- Reviewing QC and sample data during analysis and determining if repeat analyses are needed
- Submitting certified QC and sample analysis results and data packages to the test coordinator
- Notifying the QAO and test coordinator of any QC excursions during the preparation and analysis of the field samples or associated QC samples
- Archiving analytical data
- Preparing a statement of the analysis activities for inclusion in the SPT report executive summary regarding if any of the test data are invalid or unusable.

## 5.0 QUALITY ASSURANCE OBJECTIVES AND QUALITY CONTROL OBJECTIVES

### 5.1 GENERAL

The project objectives are summarized in Section 3.0 of this QAPjP. The selected sampling contractor and the contract laboratory are well experienced in the types of sampling and analyses represented by this SPT. The analytical laboratory will provide method detection limits (MDLs) and reporting limits (RLs) for each analysis. The laboratory will provide the best and lowest detection limits for all testing parameters. Agency-issued audit samples, as may be provided, will be included in the analysis scope as a third party check of the laboratory accuracy.

The overall QA objective is to produce a complete data set that can be used to fully assess and validate the emissions from the system relative to the identified performance criteria. This includes a number of quality indicators necessary to assess the precision and accuracy of all the test data.

The field and laboratory data obtained during this SPT will be reviewed by the QAO. The data quality will be discussed with respect to meeting each of the data quality objectives (DQOs) and the overall project objective. Data that are outside of the target DQO limits will be evaluated relative to the impact(s) on the overall project objective of assessing the thermal treatment unit's performance. The data evaluation and validation will be included in the final test report.

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

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

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

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

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

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

**Table 5-1. Test Analytical Data Quality Objectives**

Page 1 of 2

| Parameter | QC Type | Precision | Accuracy |
|---|---|---|---|
| **Process Samples** | | | |
| Metals | Duplicates | ≤ 30% RPD | --- |
| | Matrix Spikes | < 30% RPD | ± 35% |
| **Method 0031 Sampling Train (SMVOC)** | | | |
| SMVOC Tubes | Spiked Resin Blanks | < 25% RPD | 75 - 125% |
| SMVOC Tubes | Surrogate Spikes | ≤ 35% RSD | 50 - 130% |
| SMVOC Condensate | Surrogate Spikes | ≤ 35% RSD | 50 - 130% |
| SMVOC Breakthrough Analysis | Breakthrough Analysis | --- | Note a |
| SMVOC Audit | Audit Sample | --- | 50 - 150% |
| **Method 0023A Sampling Train (PCDD/PCDFs)** | | | |
| Method 0023A PCDD/PCDFs | Spiked Resin Blanks | ≤ 25% RPD | 75 - 125% |
| | PCDD/PCDF $C^{13}$ Labeled Sampling Surrogate Spike | ≤ 35% RSD | Note b |
| | PCDD/PCDF $C^{13}$ Isotope Dilution Internal Standard Spikes | --- | Note c |
| | EPA Audit Sample | --- | 50-150% |
| **Method 0010 Sampling Train (SVOCs & PAHs)** | | | |
| Method 0010 Sampling Train (SVOCs) | Spiked Resin Blanks | ≤ 30% RPD | 70 - 130% |
| | C13 Labeled Sampling Surrogate Spike | ≤ 35% RSD | 50 - 150% |
| | Semivolatile Surrogate Spikes | ≤ 35% RSD | Note d |
| **Method 29 Sampling Train** | | | |
| Method 29 Metals | Duplicates | ≤ 35% RPD | --- |
| | Matrix Spike and Post-Digestion Spikes | ≤ 35% RPD | --- |
| | Reference Sample | --- | 70-130% |
| | EPA Audit Samples | --- | 70-130% |
| **Method 26A Sampling Train** | | | |
| Particulate Matter | Replicate Weighings | ± 0.5 mg | ± 0.5 mg |
| Hydrogen Chloride/Chlorine | Duplicates | ≤ 35% RPD | --- |
| | Matrix Spikes | ≤ 35% RPD | ± 30% |
| | Reference Sample | --- | ± 10% |
| | EPA Audit Samples | --- | 70-130% |
| **Continuous Emissions Monitors** | | | |
| Carbon Monoxide | Performance Specification 4B | ± 3% of Span | ± 5% of Span |
| Oxygen | Performance Specification 4B | ≤ 0.5% $O_2$ | ≤ 0.5% $O_2$ |
| Total Hydrocarbons | EPA Method 25A | ± 3% of Span | ± 5% of Certified Calibration Gas |
| Nitrogen Oxides | EPA Method 7E | ± 3% of Span | ± 5% of Certified Calibration Gas |
| **Orsat Analyses** | | | |
| Carbon Dioxide | Certified Calibration Gas | --- | ± 0.2% $CO_2$ |
| Oxygen | Ambient Air | --- | 20.9% ± 0.5% |

**Table 5-1. Test Analytical Data Quality Objectives**

Page 2 of 2

**Notes:**

**Note a: Breakthrough Analysis Criterion.**
The first Tenax® tube is analyzed separately from the combined second Tenax® and the Anasorb® tubes to determine the possible target VOC breakthrough to the second Tenax® tube. The analysis of the combined Tenax® and Anasorb® tubes should show less than 30 percent of each target VOC collected on the front Tenax® tube. Breakthrough of each target VOC to the combined Tenax® and Anasorb® tubes above this level may cause loss of desorption efficiency and would indicate a possible negative bias in the emissions calculations. This criterion does not apply when less than 75 ηg of target VOC is detected on the combined Tenax® and Anasorb® tubes.

**Note b: Method 23 PCDD/PCDF Sampling Surrogate Compound Recoveries**

| Compound | Target Recovery |
|---|---|
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 70 – 130% |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | 70 – 130% |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 70 – 130% |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 70 – 130% |
| $^{37}Cl_4$-2,3,7,8-TCDD | 70 – 130% |

**Note c: Method 23 PCDD/PCDF Isotope Dilution Internal Standard Compound Recoveries**

| Compound | Target Recovery |
|---|---|
| $^{13}C_{12}$-2,3,7,8-TCDD | 40 – 130% |
| $^{13}C_{12}$-2,3,7,8-TCDF | 40 – 130% |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 40 – 130% |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 40 – 130% |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 40 – 130% |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 40 – 130% |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 25 – 130% |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 25 – 130% |

**Note d: Semivolatile Surrogate Compound Spike Recoveries**

| Compound | Aqueous Target Recovery | Solid Target Recovery |
|---|---|---|
| Nitrobenzene-$d_5$ | 35 - 114% | 23 - 120% |
| 2-Fluorobiphenyl | 43 - 116% | 30 - 115% |
| Phenol-$d_5$ | 10 - 94% | 24 - 113% |
| 2-fluorophenol | 21 - 100% | 25 - 121% |
| 2,4,6-Tribromophenol | 10 - 123% | 19 - 122% |

PAH - Polynuclear Aromatic Hydrocarbons
PCDD/PCDF - polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans
QC - quality control
SMVOC - Sampling Method for Volatile Organic Compounds
SVOC - Semivolatile Organic Compounds
RPD - relative percent difference
RSD - relative standard difference

### 5.2 PRECISION AND ACCURACY

A number of procedures will be implemented to assess the precision and accuracy of the sampling and analytical data. All sampling and analytical activities will be conducted following referenced procedures. All reference materials used as calibration standards, surrogate compounds, or laboratory control samples will be of the highest purity commercially available. The calibration of instruments used during analysis will be verified each day that samples are analyzed as described in later sections of this QAPjP. Data precision and accuracy will be assessed by evaluating the results from multiple analyses of the same parameter, and analysis of standards, duplicates, and spiked samples. Field and laboratory contamination will be assessed by the analysis of reagent, instrument, method, and field and trip blanks.

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

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

Accuracy values in Table 5-1 include components of both random error and bias, expressed as a percentage of the "true" or "known" value (for reference materials) or percent analyte recovery (for spiked samples). The QA/QC program will focus upon controlling measurement error within the estimated limits of measurement uncertainty, as specified in Table 5-1. It should be noted that these limits are estimates that are, in most cases, described in the referenced analytical methods or in QA/QC guidance for hazardous waste thermal treatment. They represent the range of results that can be expected from these methods based on actual field sampling results and laboratory-based QA/QC studies. Therefore, it is reasonable to expect that the measurement errors associated with this SPT will be within the objectives shown in Table 5-1. QA/QC determinations that fall outside the target range will be flagged, and an assessment of the impact, if any, on the usefulness of the data on the overall results and conclusions of the SPT will be provided in the final test report.

The analytical laboratory's SOPs for each analysis will be followed. If during test sample analyses, an analytical result exceeds the calibrated range for a target analyte, or any other analytical anomalies are noted with any test samples, the test project coordinator and QAO are to be contacted immediately to discuss the results and issues and possible options before proceeding with the subject analyses. If ongoing QA/QC procedures reveal that a measurement's error has exceeded the estimated data quality limits, the source of the excessive error will be identified and corrective action will be taken, as described in Section 14.0. If data fall outside the acceptable range of precision and accuracy, even after corrective action has been taken, those data points will be flagged in the final report. The precision and accuracy for those measurements will be reported as determined using the actual data. Also, alternative procedures (either sampling or analytical) may be considered and recommended if possible.

#### 5.2.1 Method 0031 Sampling Method for Volatile Organic Compounds

The volatile organic sampling train (SW-846, Method 0031) will be used to sample process off-gases for the target volatile organic compounds (VOCs) and TICs.

#### 5.2.1.1 Spiked Resin Blanks

During the preparation of the sampling method for volatile organic compounds (SMVOC) tube sets for the SPT, two SMVOC tube sets will be spiked with the SW-846 Method 5041A/8260B-specified surrogate spike compounds. These samples will be retained by the laboratory and included with the field samples when they are returned from the field for analysis. The relative recoveries of the surrogate spikes from these samples provide an accuracy assessment of the SMVOC analytical methodology. The relative recoveries of the isotopically labeled surrogate and internal standard spikes applied to every SMVOC sample analysis provide an ongoing accuracy assessment of the SMVOC analytical methodology. The two pairs of the spiked SMVOC tubes provide a precision (reproducibility) assessment for SMVOC methodology. The consistency of the isotopically labeled surrogate spike recoveries applied to every SMVOC sample analysis provides an ongoing precision assessment of the SMVOC analytical methodology.

#### 5.2.1.2 Sampling Method for Volatile Organic Compounds Audit

The SMVOC audit includes SMVOC sampling of a compressed gas cylinder containing a mixture of volatile organic compounds in nitrogen. During the SMVOC audit, the same technician who performed or will perform the SMVOC sampling operates the SMVOC. The SMVOC audit kit includes the audit cylinder, an electrically heated manifold, and connecting glassware and adapters. Using the same SMVOC equipment and SMVOC tubes prepared for the SPT, the technician will set up the audit cylinder per the accompanying directions and sample its contents. A minimum of four SMVOC tube set pairs will be collected. The SMVOC audit samples are then submitted for analysis along with the other test SMVOC samples. The SMVOC audit tubes will be analyzed for the select target list of VOC analytes. The SMVOC audit sample analysis results are submitted for regulatory agency assessment.

An audit cylinder will be requested at least 60 days before of the actual test date. The cylinder will be sent directly to the facility to arrive by the requested test/audit date. The custody seal on the SMVOC audit cylinder must remain intact until the regulatory observers are present to observe its breaking during the onsite audit. The regulatory observers must continually observe the SMVOC audit procedure. The auditee may only sample the audit cylinder using the SMVOC; NO OTHER SAMPLING OF THE CYLINDER, SUCH AS BAG SAMPLES, IS ALLOWED. At the end of the audit, the audit kit must be secured for return by the auditee.

The SMVOC audit results will be provided to the audit cylinder coordinator for review. The SMVOC audit is a pass/fail audit. The successful SMVOC audit will determine the concentration of each target VOC within 50–150% of the known value. The audit cylinder coordinator will issue a pass/fail determination.

#### 5.2.1.3 Sampling Method for Volatile Organic Compounds Breakthrough Analysis

A breakthrough assessment for the SMVOC will be conducted for the test samples. The analysis of the combined second Tenax® tube and Anasorb® tube should indicate less than 30% of the target VOC concentration that is collected on the first Tenax® tube. Breakthrough of the target VOC to the second Tenax® tube/Anasorb® tube above this level may cause loss of the desorption efficiency and result in a low bias in the analytical result. This criterion does not apply when less than 75 ng of the target VOC is detected on the second Tenax® tube/Anasorb® tube.

### 5.2.2 Method 0023A PCDD/PCDF Sampling

EPA Method 0023A will be used to sample the process off-gas for emissions of PCDD/PCDFs. Prior to use in the field, all the XAD-2 resin traps for use in the Method 0023A sampling train are spiked with isotopically labeled PCDD/PCDF sampling surrogate compounds as noted in Table 5-1. Two of the XAD-2 resin traps prepared for this SPT also will have PCDD/PCDF MSs applied and are retained by the laboratory. These two XAD resin traps then are included with the field samples when they are returned to the laboratory for analysis. The recoveries of the PCDD/PCDF isotopically labeled sampling surrogates

and MS compounds from these two retained XAD resin trap samples provide an accuracy assessment of the Method 0023A analytical methodology. The recoveries of the isotopically labeled sampling surrogates spiked to every field-use XAD resin sample provide a field-through-analysis accuracy assessment of the Method 0023A methodology.

The recoveries of the internal standard, alternate standard, and recovery standard compounds applied during the preparation and analysis of the Method 0023A sampling train components provide accuracy and precision measurements for the sample preparation and analysis steps. Table 5-1 notes the RPD and RSD values for the Method 0023A PCDD/PCDF analyses.

#### 5.2.3 Method 0010 Semivolatile Organic Compound and Polyaromatic Hydrocarbon Sampling

SW-846 Method 0010 will be used to sample the process off-gas for emissions for the target semivolatile organic compounds (SVOC), including polyaromatic hydrocarbon (PAH) target compounds and TICs. Prior to use in the field, all the XAD-2 resin traps for use in the Method 0010 sampling train are spiked with an isotopically labeled SVOC sampling surrogate compound, as noted in Table 5-1. Two of the XAD-2 resin traps prepared for this SPT also will have SVOC MSs applied and are retained by the laboratory. These two XAD resin traps then are included with the field samples when they are returned to the laboratory for analysis. The recoveries of the SVOC isotopically labeled sampling surrogate and MS compounds from these two retained XAD resin trap samples provide an accuracy assessment of the Method 0010 analytical methodology. The recoveries of the isotopically labeled sampling surrogates spiked to every field-use XAD resin sample provide a field-through-analysis accuracy assessment of the Method 0010 methodology.

The recoveries of the internal standard, alternate standard, and recovery standard compounds applied during the preparation and analysis of the Method 0010 sampling train components provide accuracy and precision measurements for the sample preparation and analysis steps. Table 5-1 notes the RPD and RSD values for the Method 0010 SVOC analysis.

#### 5.2.4 Method 29 Metals Sampling

Non-detect results are possible for Method 29 process off-gas samples for metals due to the relatively low feed rates of metals and the corresponding low concentrations of metals expected in the process off-gas. The SPT includes collecting a blank Method 29 train to assess blank contamination levels. The analysis program includes post-digestion spikes of actual Method 29 samples. The recovery and reproducibility of these spikes and analyses will be used to verify that any non-detect values for Method 29 process off-gas samples are valid. The spiked samples will be spiked with metal analytes at 3 to 5 times the MDL to demonstrate the recovery and reproducibility of the preparation and analysis methods.

Metals audit samples provide an additional accuracy check of the Method 29 methodology. Metals audit samples are usually provided in the form of a spiked filter and an aqueous solution for nonmercury metals, and an aqueous solution for mercury. These samples serve as blind audit samples. Metals audit samples will be submitted for analysis along with the balance of the Method 29 test samples if provided by the regulatory agency.

#### 5.2.5 Method 26A Particulate Sampling

The gravimetric analysis of the Method 26A samples for particulate analyses includes replicate weighing to assess analysis precision. The analytical balance accuracy is verified using Class S weights. Method 26A particulate matter samples are evaporated, and dried and desiccated to constant weight.

#### 5.2.6 Method 26A Chloride Sampling

The analysis of the Method 26A samples for chloride includes a system matrix spike/matrix spike duplicate (MS/MSD) analyses to evaluate the precision and accuracy of the ion chromatography

analytical methodology. Chloride audit samples provide an additional accuracy check of the Method 26A methodology. Chloride audit samples in the form of spiked aqueous solutions are analyzed as blind audit samples. Chloride audit samples will be submitted for analysis along with the balance of the Method 26A test samples if provided by the regulatory agency.

Matrix spikes to Method 26A field samples and reagent blanks will be performed at the greater of 2 times the sample's apparent native concentration or 10 times the MDL used for the ion chromatography method to demonstrate the recovery and reproducibility of the method. The relative recoveries of the MSs provide a measure of the accuracy of the chloride ion analysis methodology. Additionally, precision is assessed by duplicate analyses of field, reagent blank, calibration standard, and QC samples.

#### 5.2.7 Installed Continuous Emissions Monitoring System

The precision of the installed carbon monoxide and oxygen continuous emissions monitoring system (CEMS) analyzers will be assessed using the recommended calibration gases in accordance with the appendix to 40 CFR 63, Subpart EEE. Precision will be assessed using the following equation:

$$\text{Precision}(\%drift) = \left(\frac{R_f - R_i}{Span}\right)x100$$

Where

$R_f$ = final monitor response at end of the test run

$R_i$ = initial monitor response at start of the test run

Span = maximum range of the analyzer.

The accuracy of all CEMS analyzers will be evaluated by measuring percent accuracy as defined by the following equation:

$$\text{Accuracy}(\%) = \left(\frac{R_a - R_c}{Span}\right)x100$$

Where

$R_a$ = analyzer-indicated concentration of the calibration gas

$R_c$ = certified concentration of the calibration gas

Span = maximum range of the analyzer.

Accuracy of carbon monoxide and oxygen CEMS analyzers will be evaluated by a relative accuracy test audit conducted prior to the SPT. The relative accuracy test audit will include calibration drift tests, response time tests, calibration error tests, and relative accuracy tests per the appendix to 40 CFR 63, Subpart EEE. During the SPT, the CEMS analyzers will be calibrated daily before the start of testing.

#### 5.2.8 Temporary Total Hydrocarbon and Nitrogen Oxide Continuous Emissions Monitoring Systems

A temporary HC analyzer will be used to measure total hydrocarbon (THC) or HC emissions. The temporary THC continuous emissions monitor (CEM) will be calibrated and operated in accordance with 40 CFR 60, Appendix A, Method 25A. The precision and accuracy of the temporary THC analyzer will be assessed

during the SPT using the recommended calibration gases in accordance with the procedures of Method 25A. During the SPT, the THC analyzers will be calibrated before, at the midpoint, and at the end of each test run.

A temporary nitrogen oxide analyzer will be used to measure nitrogen oxide emissions. The temporary nitrogen oxide CEM will be calibrated and operated in accordance with 40 CFR 60, Appendix A, Method 7E. The precision and accuracy of the temporary nitrogen oxide analyzer will be assessed during the SPT using the recommended calibration gases in accordance with the procedures of Method 7E. During the SPT, the nitrogen oxide analyzer will be calibrated before, at the midpoint, and at the end of each test run.

### 5.3 DETECTION LIMITS AND REPORTING

How the detection limits will be used in data reduction and reporting is described in Section 11.0.

For inorganic analyses and nonisotope dilution method organic analyses, the laboratory report will provide both the MDL, and either the reliable detection limit (RDL) or the laboratory-specified reporting limit (RL). The laboratory will maintain records documenting the MDL, and the RDL or RL determinations. Generally, all non-detects for target analytes will be reported and assessed at the laboratory-determined MDL. If an analyte is detectable at some value between the MDL and the RDL or RL, the detected value will be reported and flagged as estimated. If matrix interference(s) occurs or sample dilutions are necessary, a sample-specific MDL or sample quantitation limit (SQL) may be reported. If a sample-specific MDL or SQL is applicable, the documentation of the serial dilutions or other measures taken to arrive at the SQL will be documented in the analytical report.

For isotope dilution organic analysis methods, the non-detects for the isotope dilution methods will be determined using the SW-846 Method 8290 definition of an estimated detection limit (EDL) without the use of empirical factors or other mathematical manipulations specific to the laboratory.

The analytical report of any target analyte that is non-detect shall include the MDL, and the RDL or RL, or the EDL, as appropriate.

### 5.4 COMPLETENESS

Data completeness represents the valid data collected from the total number of valid tests conducted. Samples resulting from test runs that are judged invalid based on field performance indicators or aborted runs will not be submitted to the laboratory for analysis. Because a sample possibly may be lost or broken, the data from each individual analytical parameter may not be complete for all test runs. The impact of any occurrence of sample loss will be assessed with regard to the objective of obtaining valid runs and will be discussed in the final test report. The completeness objective of this SPT is to generate sufficient data for the regulatory agencies to judge the performance of the system.

### 5.5 REPRESENTATIVENESS AND COMPARABILITY

The sampling procedures chosen for the SPT are, wherever possible, approved EPA or American Society for Testing Materials (ASTM) sampling methods. These methods are typically employed on thermal treatment system tests. The use of standard sampling methods affirms sample consistency and representativeness.

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

# 6.0 SAMPLING AND MONITORING PROCEDURES

## 6.1 GENERAL

The objective of this SPT is to collect representative samples and produce data of sufficient quality to evaluate the performance of the IWTU against identified performance and risk criteria. To meet this objective requires minimizing the potential sources of sample contamination or bias imparted to the samples by the sampling equipment, ambient conditions, handling, and preservation. The SPT samples will be collected using the methods summarized in Table 6-1. The total numbers of field samples expected to be generated during the SPT also are summarized Table 6-1.

Guidelines followed to determine sampling equipment to be used, sampling points, and the frequency at which samples are to be taken are presented in the SPT plan, and are incorporated here by reference. The reference sources for the standard sampling methods include:

- 40 CFR 60, "Standards of Performance for New Stationary Sources," Appendix A, "Test Methods," EPA
- SW-846, *Test Methods for Evaluating Solid Waste*, Third Edition, 1986 and updates
- American Society for Testing and Materials, *Annual Book of ASTM Standards*.

The sampling and analytical procedures to be used during the SPT are located in Appendixes A and B of the SPT plan. Reference sampling and analytical methods are identified throughout the SPT plan and this QAPjP. The specifics of the sampling and analytical methodologies followed during the SPT by the selected sampling and analytical contractors include the referenced nationally recognized methods (ASTM, SW846, 40 CFR 60, Appendix A, etc.) and/or the contractor-specific SOPs.

During sampling and analysis, situations may still arise that require modifying the specific sampling or analytical procedures included or referenced in the SPT plan or this QAPjP. For the process off-gas samples, there are few alternatives to the analysis methods prescribed in SW-846 or 40 CFR 60. For the samples from the single sampling train (for example, Method 0010 for SVOCs and PAHs), the proposed procedures for the sample extractions and splitting of extracts for the respective analyses are noted in the analytical procedure in Appendix C of the SPT plan and are discussed in this QAPjP. Laboratory SOPs, which include a number of procedures for special circumstances, will be followed. If the laboratory finds it necessary to adjust the analytical methods, the changes will be documented following the corrective action procedures noted in Section 14.0 of this QAPjP. Any such changes must be approved by the test-designated QAO. Regulatory observer approval will be requested if significant deviations from planned sampling procedures are encountered during the SPT.

**Table 6-1. Sample Collection Locations, Equipment, and Methods**

| Sample Name | Sampling Reference Method | Sample Container | Analysis | General Procedure/Frequency | Test | Field QC | Total Field Samples |
|---|---|---|---|---|---|---|---|
| SM VOC Resin Tubes | SW846 Method 0030 | SMVOC Resin Tubes | Target VOCs & TICs | Collect four (4) tube sets per run. ~20 liters of gas sampled per tube set | 16 | -- | 16 |
| SM VOC Condensate | | 40 mL glass VOA | Target VOCs & TICs | Collect one (1) composite gas condensate for each test run | 8 | -- | 8 |
| SM VOC Field Blank Resin Tubes | | SMVOC Resin Tubes | Target VOCs & TICs | Collect one (1) tube set per run | -- | 8 | 8 |
| SM VOC Trip Blank Resin Tubes | | SMVOC Resin Tubes | Target VOCs & TICs | Collect one (1) tube set per shipment | -- | 1 | 1 |
| SM VOC Condensate Trip/Field Blank | | 40 mL glass VOA | Target VOCs & TICs | Collect one (1) glass VOA per test condition | -- | 2 | 2 |
| SM VOC Audit Resin Tubes (if provided) | | SMVOC Resin Tubes | Target VOCs & TICs | Collect four (4) tube sets at sampled volumes of audit gas sampled per tube set pair | -- | 4 | 4 |
| Particulate Filter | EPA Method 0023A | Petri Dish | PCDD/PCDFs | Collect integrated sample for PCDD/PCDFs during each test run. Sample for a minimum 180 minutes to sample a minimum of 3.0 dry standard cubic meters of gas. | 8 | 2-blank train | 10 |
| Acetone and Methylene Chloride Rinses | | 500 mL glass sample bottle | PCDD/PCDFs | | 8 | 2-blank train | 10 |
| Toluene Rinses | | 500 mL glass sample bottle | PCDD/PCDFs | | 8 | 2-blank train | 10 |
| XAD-2 Resin | | Resin Trap | PCDD/PCDFs | | 8 | 2-blank train | 10 |
| Acetone Reagent Blank | | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | 1 | 1 |
| Methylene Chloride Reagent Blank | | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | 1 | 1 |
| Toluene Reagent Blank | | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | 1 | 1 |
| PCDD/PCDF Audit Sample (if provided) | | Ampoule with spiked XAD-2 Resin | PCDD/PCDFs | As provided by Agency | -- | 1 | 1 |
| Particulate Filter | EPA Method 29 | Petri Dish | Metals and Mercury | Collect integrated sample for metals and mercury during each test run. Sample for a minimum 60 minutes to | 8 | 2-blank train | |
| Front Half Nitric | | 250 mL glass | Metals and Mercury | | 8 | 2-blank | |

| Sample Name | Sampling Reference Method | Sample Container | Analysis | General Procedure/Frequency | Test | Field QC | Total Field Samples |
|---|---|---|---|---|---|---|---|
| Acid Rinses | | sample bottle | | sample approximately 0.75-1.0 dry standard cubic meters of gas. | | train | |
| Acidified Peroxide Impingers and Rinses | | 1-liter glass sample bottle | Metals and Mercury | | 8 | 2-blank train | |
| Empty Impinger and Rinses | | 250 mL glass sample bottle | Mercury Only | | 8 | 2-blank train | |
| Acidified Permanganate Impingers and Rinses | | 500 mL glass sample bottle | Mercury Only | | 8 | 2-blank train | |
| Acidified Permanganate Impingers 8N HCl Rinses | | 250 mL glass sample bottle | Mercury Only | | 8 | 2-blank train | |
| Particulate Filter Reagent Blank | | Petri Dish | Metals and Mercury | Reagent Blank | -- | 1 | 1 |
| Nitric Acid Reagent Bland | | 250 mL glass sample bottle | Metals and Mercury | Reagent Blank | -- | 1 | 1 |
| Acidified Peroxide Reagent Blank | | 250 mL glass sample bottle | Metals and Mercury | Reagent Blank | -- | 1 | 1 |
| Acidified Permanganate Reagent Blank | | 250 mL glass sample bottle | Mercury Only | Reagent Blank | -- | 1 | 1 |
| 8N HCl Reagent Blank | | 250 mL glass sample bottle | Mercury Only | Reagent Blank | -- | 1 | 1 |
| Metals Audit Sample Filter (if provided) | SW846 Method 6010B or 6020A | Filter in Petri Dish | Metals Only | As provided by Agency | -- | 1 | 1 |
| Metals Audit Sample Liquid (if provided) | | Ampoule with spiked liquid | Metals Only | As provided by Agency | -- | 1 | 1 |
| Mercury Audit Sample Liquid | SW846 Method 7470A/7471A | Ampoule with spiked liquid | Mercury Only | As provided by Agency | -- | 1 | 1 |
| Method 0010 Particulate Filter | SW846 Method 0010 | Petri Dish | SVOCs & TICs and PAHs | Collect integrated sample for SVOCs and PAHs during each test run. Sample for a minimum 180 minutes to sample a minimum of 3.0 dry standard cubic meters of gas. | 8 | 2-blank train | 10 |
| Method 0010 Acetone and Methylene | | 500 mL glass sample bottle | SVOCs & TICs and PAHs | | 8 | 2-blank train | 10 |

| Sample Name | Sampling Reference Method | Sample Container | Analysis | General Procedure/Frequency | Test | Field QC | Total Field Samples |
|---|---|---|---|---|---|---|---|
| Chloride Rinses | | | | | | | |
| Method 0010 XAD-2 Resin | | Resin Trap | SVOCs & TICs and PAHs | | 8 | 2-blank train | 10 |
| Method 0010 Condensate | | 1 liter glass bottle | SVOCs & TICs and PAHs | | 8 | 2-blank train | 10 |
| Method 0010 Acetone Reagent Blank | | 250 mL glass sample bottle | SVOCs & TICs and PAHs | Reagent Blank | -- | 1 | 1 |
| Method 0010 Methylene Chloride Reagent Blank | | 250 mL glass sample bottle | SVOCs & TICs and PAHs | Reagent Blank | -- | 1 | 1 |
| Method 26A Particulate Filter | EPA Method 26A | Petri Dish | Filterable Particulate | Collect integrated sample for particulate and $HCl/Cl_2$ during each test run. Sample for a minimum 60 minutes to sample approximately 0.75-1.0 dry standard cubic meters of gas | 8 | -- | 8 |
| Method 26A Front Half Acetone Rinses | | 250 mL glass sample bottle | Filterable Particulate | | 8 | -- | 8 |
| Method 26A Sulfuric Acid Impingers and Rinses | | 1 liter glass bottle | Chloride Ion | | 8 | -- | 8 |
| Method 26A Sodium Hydroxide Impingers and Rinses | | 500 mL glass sample bottle | Chloride Ion | | 8 | -- | 8 |
| Method 26A Particulate Filter Reagent Blank | | Petri Dish | Filterable Particulate | Reagent Blank | -- | 1 | 1 |
| Method 26A Acetone Reagent Blank | | 250 mL glass sample bottle | Filterable Particulate | Reagent Blank | -- | 1 | 1 |
| Method 26A Deionized Water Reagent Blank | | 250 mL glass sample bottle | Chloride Ion | Reagent Blank | -- | 1 | 1 |
| Method 26A Sulfuric Acid Reagent Blank | | 250 mL glass sample bottle | Chloride Ion | Reagent Blank | -- | 1 | 1 |
| Method 26A Sodium | | 250 mL glass sample bottle | Chloride Ion | Reagent Blank | -- | 1 | 1 |

| Sample Name | Sampling Reference Method | Sample Container | Analysis | General Procedure/Frequency | Test | Field QC | Total Field Samples |
|---|---|---|---|---|---|---|---|
| Hydroxide Reagent Blank | | | | | | | |
| Chloride in Acid Media Audit Sample | | Ampoule with spiked liquid | Chloride Ion | As provided by Agency | -- | 1 | 1 |
| Chloride in Basic Media Audit Sample | | Ampoule with spiked liquid | Chloride Ion | As provided by Agency | -- | 1 | 1 |
| Carbon Dioxide and Oxygen by Orsat | EPA Methods 3 and 3A | 25-50 L Tedlar gas sample bag | Carbon Dioxide and Oxygen by Orsat | Collect two 1-hour integrated bag sample for on-site Orsat analysis. | 8 | -- | 8 |
| Carbon Monoxide | EPA Method 10; 40 CFR 266 Appendix IX | Installed Continuous Emissions Monitor | Carbon Monoxide | Continuous during each test run | -- | -- | -- |
| Total Hydrocarbons | EPA Method 25A | Temporary Continuous Emissions Monitor | Total Hydrocarbons as Propane Equivalents | Continuous for two 60-minute periods during each test run. | -- | -- | -- |

ASTM – American Society for Testing and Materials
EPA Method – New Source Performance Standards, Test Methods and Procedures, Appendix A, 40 CFR 60
mL – milliliter
PAH – Polyaromatic Hydrocarbon
PCDD/PCDF – Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzo furans
QA – quality control
SMVOC – Sampling Method for Volatile Organic Compounds
SVOC – Semivolatile Organic Analysis
SW846 – Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates
TIC – Tentatively Identified Compound

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

Process samples will be collected using dedicated sampling equipment (sample bottles, funnels, jars, etc.) at each sampling location, thus eliminating the potential for cross contamination from one sample matrix to another. New sampling containers are used for each test run. If the same equipment will be used for more than one run, the equipment will be decontaminated by additional solvent rinses. Any decontamination solution generated will be collected by the facility operators for proper disposal.

During the SPT, the IWTU will be operated and tested at the conditions specified in the SPT plan. The following samples will be collected during the SPT:

- Process off-gas samples

  SW-846 Method 0031 for VOCs and TICs

  SW-846 Method 0010 for SVOCs and TICS and PAHs

  SW-846 Method 0023A for PCDD/PCDFs

  EPA Method 26A for particulate and HCl and Cl2

  EPA Method 29 for metals

  Temporary CEMS for hydrocarbons (HC or THC) (EPA Method 25A) and for nitrogen oxides (EPA Method 7E)

  Installed CEMS for carbon monoxide.

Refer to Section 7.0 of this QAPjP. Sample tracking is documented using unique alphanumeric sample numbering applied to every sample, completed sample collection forms, completed RFA forms, completed COC forms, and sample collection checklists.

## 6.2 FIELD SAMPLING METHODS

### 6.2.1 Process Off-gas Samples

During the SPT, the sampling contractor lead and the sample custodian are responsible for monitoring the sampling team's adherence to the standard sampling procedures, especially sampling train preparation; leak checks and recoveries (including blank trains); and reagent, field, and trip blanks. The sampling team leader is responsible for operation and recovery of the sampling equipment and process off-gas samples. The sample custodian is responsible for preparing the process off-gas and process samples for shipment to the laboratory. Sampling train calibration procedures are discussed in Section 8.0.

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

During each test run, Tedlar® bag samples of process off-gas will be collected to determine the process off-gas carbon dioxide and oxygen concentrations using EPA Method 3 (Orsat Analysis). The process off-gas carbon dioxide and oxygen concentrations are used to determine the process off-gas molecular weight. The Tedlar® bag samples may be collected from the exhaust of one of the isokinetic sampling trains or using a separate impinger and vacuum pump setup. Certified calibration gases containing at least 5% volume carbon dioxide or at least 5% volume oxygen or both may be used as reference

standards for the Orsat apparatus. Ambient air may be used as a reference standard for the oxygen portion of the Orsat apparatus.

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

#### 6.2.1.1 Sampling Method for Volatile Organic Compounds

The SMVOC will be used to sample process off-gas for the target VOCs and TICs. The SMVOC sampling apparatus will be inspected and leak-checked prior to each test run. Four sets of SMVOC tubes will be used during each sampling run to sample a nominal 20 liters of process off-gas per tube set. Three of the four sets will be analyzed by Method 5041A/8260B. The fourth set will serve as an archive set in the event of sample breakage during shipping and handling. During the analysis, the first Tenax® tube will be analyzed separately from the second Tenax® tube and the Anasorb® tube to assess breakthrough. Field blank and trip blank tube sets may be analyzed as a single sample or separately because breakthrough criterion need not be applied to these samples.

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

- **Surrogate Spikes**—isotopically labeled compounds applied to the sample just prior to analysis. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.

- **Volatile Internal Standard Compounds**—these compounds are applied to the sample extract just prior to gas chromatography/matrix spectrometry (GC/MS) analysis. Internal standards area counts and retention times are used to determine if the sample response has been affected by method analytes, surrogates, or matrix interferences.

- **Matrix Spike Compounds**—these compounds are spiked onto the condensate portion of the Method 0031 samples and onto two sets of blank Tenax resins. The MS compounds are spiked onto an aliquot of the Method 0031 condensate and blank resin sample before GC/MS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.

#### 6.2.1.2 Method 0010 for Semivolatile Organic Compounds and Polyaromatic Hydrocarbons

The Method 0010 sampling train will be used during this SPT to sample process off-gas for target SVOCs, TICs, and PAHs. During each test run, the Method 0010 sampling train will be assembled and leak-checked. The sampling train will be operated for approximately 4 hours to collect a minimum of 3 dscm of process off-gas during each sampling run. The sampling time to achieve the target sample volume will depend upon the probe nozzle size and sampling rate. However, sampling rate will not exceed 1 dscf/min. At the end of each run, the sampling train will be disassembled and all train samples collected.

##### 6.2.1.2.1 Method 0010 Recovery

The Method 0010 sample recovery considerations are covered in the sampling procedure in Appendix B of the SPT plan. For the combined Method 0010 sampling train, the following general procedures will be used for sample recovery:

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

- **Front-half rinse**—The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware are brushed and rinsed three times with acetone/methylene chloride (1:1 v/v) followed by brushing and rinsing three times with methylene chloride. All rinses are placed into a glass sample bottle (Container 2). The final liquid level is marked on the sample container.

- **XAD-2 adsorbent resin trap**—The XAD-2 adsorbent resin trap is removed from the train and both ends are capped. The XAD-2 adsorbent resin trap is then weighed to the nearest 0.5 g 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 to a glass sample bottle (Container 4). The back half of the particulate filter holder also is inspected for condensate and recovered, if observed, by measuring or weighing and transferring to Container No. 4 as described previously.

- **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 followed by brushing and rinsing three times with methylene chloride./methylene chloride (1:1 v/v). The condensate knockout impinger (Impinger 1) is rinsed with acetone and /methylene chloride (1:1 v/v). The rinses are then placed into a glass sample bottle with a Teflon-lined lid (Container 5). The final liquid level is marked on the sample container.

- **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 the last impinger are weighed to the nearest 0.5 g. The color and condition of the silica gel are noted on the sample recovery sheet. The silica gel may be discarded or recovered for reuse.

The Method 0010 sample recovery procedure requires separate recovery of the front-half components (filter element and probe rinses) and back-half components (XAD-2 resin trap and condenser rinses). For the front-half and back-half solvent rinses, Method 0010 requires a series of two solvent rinses: acetone, followed by methylene chloride. Method 0010 also requires recovery of the condensate fraction for the analysis.

#### 6.2.1.2.2 Method 0010 Preparation for Analysis

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

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

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

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

Four types of spiking materials will be applied to the Method 0010 sampling train samples:

- **Sampling surrogate spikes**—Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD-2 resin tube preparation and prior to sampling. The recovery of these compounds provides a comprehensive accuracy indication (to final analysis) of the SVOCs found using the Method 0010 sampling method.

- **Surrogate spikes**—Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.

- **Semivolatile internal standard compounds**—These compounds are applied to the sample extract just prior to GC/MS analysis. Internal standards area counts and retention times are used to determine if the sample response has been affected by method analytes, surrogates, or matrix interferences.

- **Matrix spike compounds**—These compounds are spiked onto the condensate portion of the Method 0010 samples and onto a blank XAD resin. The MS compounds are spiked onto an aliquot of the Method 0010 condensate and blank resin sample before GC/MS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.

Commensurate with the procedures allowed under at 40 CFR 63.1208(b)(1)(iii) for the Method 0023A analysis for dioxin/furan congeners, any target SVOC or PAH compound that is not detected in the analysis fractions of the Method 0010 sampling train will be counted as zero in determining the emissions data for that compound.

#### 6.2.1.3 Method 0023A for PCDD/PCDFs

A Method 0023A sampling train will be used to sample process off-gas dioxin and furans (PCDD/PCDFs). The extracts from the Method 0023A sampling train will be analyzed for PCDD/PCDFs using Method 8290 High Resolution Gas Chromatography/High Resolution Mass Spectrometry.

The (40 CFR 63.1208(b)(1)(iii) requires that the Method 0023A sampling train be operated for a minimum of 180 minutes (3 hours) to sample a minimum of 2.5 dscm of process off-gas during each sampling run so that any PCDD/PCDF congener that is non-detect may be counted as zero in determining compliance.

Prior to each test run, the Method 0023A sampling train will be assembled and leak-checked. At the end of each run, the sampling train will be disassembled and all train samples collected.

##### 6.2.1.3.1 Method 0023A Recovery

The Method 0023A sample recovery considerations are covered in the sampling procedure in Appendix B of the SPT plan. For the combined Method 0023A sampling train, the following general procedures will be used for sample recovery:

- **Particulate filter**—The filter is removed from its holder and placed into its original petri dish (Container 1), which is sealed with Teflon tape and sealed in a plastic bag.

- **XAD-2 adsorbent resin trap**—The XAD-2 adsorbent resin trap is removed from the train, and both ends are capped. The XAD-2 adsorbent resin trap is then weighed to the nearest 0.5 g and the weight recorded. The trap is then labeled, covered with aluminum foil, sealed in a plastic bag, and stored in an insulated cold chest.

- **Front-half and back-half rinses**—The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware are brushed and rinsed three times each with acetone and methylene chloride and two times with toluene. The rinses are placed into a glass sample bottle (Container 2). The back half of the filter holder, the filter support, transfer line (if used), and condenser (if separate from trap) are rinsed three times with acetone and then soaked two times with three separate portions of methylene chloride for 5 minutes each. The rinses and soaks are added to the front-half rinses in Container 2placed into a glass . The final liquid level is marked on the sample bottle (cContainer 4).

- **Toluene rinse**—The front-half and back-half methylene chloride rinses and soak procedures described previously are repeated followed by two rinses using toluene as the solvent. The rinses and soak liquids are collected in a separate glass sample bottles (Containers 32 and 4). (Note: The knockout impinger, Impingers 2, 3, and 4, and connecting glassware are not included in this procedure.). The final liquid level is marked on the sample container.

- **Impinger water**—The contents of the knockout impinger and Impingers 2, 3, and 4 are measured to the nearest milliliter or weighed to the nearest 0.5 g and observed for the presence or absence of film or color, which is recorded on the sample recovery sheet. The impinger water is then discarded.

- **Silica gel**—The silica gel contents of the last impinger are weighed to the nearest 0.5 g. The color and condition of the silica gel are noted on the sample recovery sheet. The silica gel may be discarded or recovered for reuse.

The Method 0023A sample recovery procedure does not requires separate recovery of the acetone, methylene chloride/acetone rinses, and the toluene rinses. Method 0023A requires a series of three solvent rinses: acetone, followed by methylene chloride, and then toluene. Method 0023A does not require recovery of the condensate fraction for the PCDD/PCDF analysis.

#### 6.2.1.3.2 Method 0023A Preparation for Analysis

Sample preparation considerations are covered in the analysis procedure for the Method 0023A train in Appendix C of the SPT plan and are generally described in the following paragraphs.

Method 0023A requires the sampling train from the toluene fractions to be analyzed separately from the other fractions. However, based on the information that has been collected using this procedure since Method 0023A became final, it is now known that the toluene rinse will not significantly increase the amount of recovered dioxins/furans. Surrogate compounds discussed below are applied to the single fraction.

The fraction consists of the methylene chloride, acetone, and toluene solvent rinses. The particulate filter and XAD-2 resin are extracted together. The condensed solvent rinses are added to the filter and XAD-2 resin in the Soxhlet apparatus. Isotope dilution internal standard compounds for the PCDD/PCDFs are spiked directly onto the filter before the Soxhlet extraction. The sample is Soxhlet-extracted for 16 hours using toluene. The front-half extract is removed from the Soxhlet extractor and is then blown down to less than 5 mL.

For PCDD/PCDFs, a number of spiking materials are applied to the Method 0023A sampling train samples:

- **Sampling surrogate spikes**—Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD tube preparation prior to sampling use. The recovery of these compounds provides a comprehensive accuracy indication (to final analysis) of the PCDD/PCDFs found using the Method 0023A sampling method. Per Section 7.3.3 of Method 0023A, these same PCDD/PCDF surrogates are added to the Method 0023A filter after sampling just prior to Soxhlet extraction.

- **Isotope dilution internal standard spikes**—Isotopically labeled PCDD/PCDF compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.

- **Alternate standard spike**—For PCDD/PCDFs, an isotopically labeled compound may be applied to the sample after Soxhlet extraction and before extract cleanup. The recovery of this compound reflects the impacts on the sample of the extract cleanup step of sample preparation.

- **PCDD/PCDF recovery standards**—Isotopically labeled compounds applied to the Soxhlet extracts just before GC/MS analysis. These compounds provide the relative response factors, which are used to calculate analyte concentrations.

As noted previously, the Method 0023A sampling train will be operated a minimum of 180 minutes (3 hours) to sample a volume of 2.5 dscm of process off-gas during each sampling run. In accordance with 40 CFR 63.1208(b)(1)(iii), any PCDD/PCDF congener that is non-detect will be counted as zero in determining compliance.

#### 6.2.1.4 EPA Method 29 for Metals

An EPA Method 29 sampling train will be used to sample process off-gas for the project target metals. Samples are subjected to acid digestion using nitric and hydrofluoric acid per the methodology described in Method 29. Nonmercury metals will be analyzed by SW-846 Method 6010B/6020 ICP/MS. Accuracy and precision are measured using MS analyses. The five Method 29 sampling train fractions will undergo seven separate analyses as follows:

The nitric acid probe rinse and the particulate filter will be combined and digested in the laboratory as the front-half composite sample and analyzed for Hg and the non-Hg target metals

The condensate knockout impinger (Impinger 1) and the $HNO_3/H_2O_2$ impingers (Impingers 2-3) catches will be digested in the laboratory and analyzed for Hg and the non-Hg target metals

The empty impinger (Impinger 4) catch will be digested in the laboratory and analyzed for Hg

The $KMnO_4/H_2SO_4$ impinger (Impingers 5–6) catches will be digested in the laboratory and analyzed for Hg

The 8N HCl rinse of Impingers 5-6 will be digested in the laboratory and analyzed for Hg.

#### 6.2.1.5 EPA Method 26A for Particulate Matter, Hydrogen Chloride, and Molecular Chlorine

An EPA Method 26A sampling train will be used to sample the process off-gas for particulate matter (PM), HCl, and $Cl_2$. The process off-gas HCl/$Cl_2$ is sampled by bubbling the gas through impingers containing 0.1N $H_2SO_4$ (acid) and 0.1N NaOH (alkaline) solutions in series. In the acid impingers, HCl gas is captured. Any $Cl_2$ in the sampled process off-gas passes through to the acid impingers and is captured in the alkaline impingers. The chloride concentrations of the acid and alkaline impinger samples are analyzed separately for chloride ion, and are reported as HCl and $Cl_2$ catches, respectively. During the sampling train recovery, the pH of the NaOH impingers are checked separately and noted on the sampling train recovery sheet. Precision for these samples is determined through the use of duplicate analyses of the calibration standard, QC, reagent blank, and field samples. Accuracy is determined by MS analyses.

In the alkaline impinger, $Cl_2$ is captured in solution by its dissociation into chloride ion and hypochlorite ion. The analysis of the alkaline impinger includes the careful addition by the laboratory analyst of sodium thiosulfate ($Na_2S_2O_3$) to reduce the hypochlorite in the sample to chloride ion. The sample is then analyzed for chloride ion concentration. In the data reduction, the chloride analysis result for the alkaline impingers is reported as $Cl_2$. Adding thiosulfate is not necessary to preserve the $Cl_2$ captured by the alkaline impingers and should not be added in the field as part of the sample recovery. Randomly adding thiosulfate in the field could result in overdosing. Overdosing with thiosulfate can lead to formation of a thiosulfate peak in the ion chromatography output that will mask the chloride peak during the analysis, resulting in necessary sample dilutions and elevated chloride detection limits.

The analysis of Method 26A includes $H_2SO_4$, NaOH, and deionized water reagent blanks to assess blank contamination. Precision for the chloride analyses is determined through the use of duplicate analyses of all calibration standards, all QC samples, and all field samples. Accuracy is determined by MS analyses of field samples and reagent blanks.

The process off-gas particulate emissions are determined by separate analysis of the Method 26A sampling train tare-weighed filter and the front-half acetone probe rinses.

The tare-weighed particulate filter PM catch is determined by the differential weight of the particulate collected by the Method 26A sampling train. Filter samples are dried to a constant weight to the nearest 0.1 mg. Constant weight shall mean a difference between two consecutive weighings of no more than 0.5 mg.

The acetone probe rinse fraction is transferred to a tare-weighed flask, evaporated and dried to a constant weight to the nearest 0.1 mg. Constant weight shall mean a difference between two consecutive weighings of no more than 0.5 mg.

The corresponding test run filter and probe rinse residue differential weights are added together. The sum is reported as the test run filterable particulate catch.

The analysis of the Method 26A includes acetone reagent blank used to assess blank contamination. Precision for the PM analyses is determined by replicate weighings. Accuracy is determined by using Class-S weight to verify the scale accuracy.

### 6.3 FIELD QUALITY CONTROL SAMPLES

Field QC samples will be collected during the test to provide an indication of QA for the test samples. The field QC samples include:

- Tenax® and Anasorb® blanks for SMVOC
- XAD-2 resin blanks for Method 0023A and Method 0010

- Tenax® and Anasorb® field and trip blanks for SMVOC
- Reagent blanks for all sampling trains
- Blank trains for Method 0023A, Method 0010, and Method 29.

Table 6-1 includes the field QC samples that will be collected.

#### 6.3.1 Resin Blanks

During the preparation of the Tenax® and Anasorb® tubes sets for the SPT, two SMVOC Tenax® and Anasorb® resin tube sets will be spiked with standard surrogate spike compounds. The SMVOC tube preparation laboratory will analyze these samples to demonstrate the resin is free of background contamination and to confirm that efficient surrogate recoveries are achievable.

Two XAD-2 resin traps prepared for the Method 0023A and 0010 sampling trains will be spiked with a sampling surrogate and MS compounds. These samples will be extracted and analyzed for PCDD/PCDF and SVOC/PAH by the XAD-2 trap preparation laboratory to demonstrate the resin is free of background contamination and to confirm that efficient surrogate recoveries are achievable.

#### 6.3.2 Sampling Method for Volatile Organic Compounds Field Blanks

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

#### 6.3.3 Sampling Method for Volatile Organic Compounds Trip Blanks

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

#### 6.3.4 Blank Trains and Reagent Blanks

Blank train samples are the samples recovered from sampling trains that have been assembled and charged with all the required chemical reagents and collection media in the same manner as the sampling trains used to sample the process off-gases. The sampling trains are leak-checked and heated to temperature in a location near the duct. The sampling train remains sealed at the duct location for a period equivalent to the length of time the corresponding sampling train is operated during the test run. The blank train is then recovered in the same way that actual process off-gas sampling trains are recovered. The recovered blank train components are labeled as blank train samples and submitted for analysis with the actual process off-gas train samples. The results of the blank train samples indicate

possible contamination introduced to the samples by reagents, glassware, sampling environment, and sampling recovery. The blank train samples for the sampling trains used during this SPT will be collected as summarized in Table 6-1. For this SPT, the following blank trains will be collected:

Two Method 0023A PCDD/PCDF blank trains (one for each test condition)

Two Method 0010 SVOC/PAH blank trains (one for each test condition)

Two Method 29 metals blank trains (one for each test condition).

Reagent blanks are samples of the reagent source solvents, solutions, and other media used in sampling. Reagent blank samples for all sampling trains as summarized in Table 6-1.

# 7.0 SAMPLE HANDLING, TRACEABILITY, AND HOLDING TIMES

## 7.1 SAMPLE CUSTODY AND SECURITY

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

During the test, once the samples are transferred from the sampling technician to the sample custodian, sample custody becomes the responsibility of the sample custodian until the samples arrive at the analytical laboratory. When overnight couriers are utilized, the air bill will serve to document the transfer of custody from the sample custodian to the courier. The courier's air bill becomes part of the COC record. Upon transfer of the samples from the courier to the analytical laboratory, sample custody will be maintained by the analytical laboratory performing the analyses.

Samples for organic analysis (SMVOC, Method 0010, Method 0023A, etc.) will be kept on ice ($4 \pm 2^{\circ}C$) and shipped to the analytical laboratory in sealed, insulated shipping containers. All ice used for shipping samples shall be double-bagged in Ziplock® bags to prevent water leakage during shipping. "Blue ice" also may be used to chill samples. Samples not requiring chilling (particulate, chloride, properties, etc.) may be shipped in sealed shipping containers without ice.

## 7.2 SAMPLE IDENTIFICATION

In consultation with the test manager, the laboratory analysis coordinator will prepare the master sample list (see Figure 7-1) for the SPT. Once the master sample list is set, the alpha-numeric sample numbers will be assigned to every sample. The sample numbers are used to track individual samples from collection through analysis. From the master sample list, the analytical laboratory will prepare the prelabeled EPA Class III clean sample containers for field use, and the Tenax®, Anasorb®, and XAD-2 resins according to the specifications of the methods as described in the SPT plan. These items will be shipped to the test site in sealed transport containers. The sampling contractor will provide the sampling reagents for field use.

**Figure 7-1. Example Master Sample List**

| Parameter | Sample Name/Matrix | Sample Containers | Preservation | Maximum Holding Time |
|---|---|---|---|---|
| Stack gas<br>VOCs & TICs | Stack gas SMVOC tubes | Glass culture tube in foam cushioned plastic tube | Chill to 4°C | 14 days |
| | Stack gas SMVOC condensate | Glass VOA vials with Teflon-lined lids or septum lids | Chill to 4°C | 14 days |
| Stack gas<br>PCDD/PCDFs | Stack gas Method 0023A filter | Glass petri dish | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| | Stack gas Method 0023A sorbent tube | Standard cartridge wrapped in aluminum foil and sealed in plastic bag | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| | Stack gas Method 0023A solvents | Amber glass Boston round bottles with Teflon-lined lid | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| Stack gas SVOCs & TICs and PAHS | Stack gas Method 0010 filter | Glass petri dish | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| | Stack gas Method 0010 sorbent tube | Standard cartridge wrapped in aluminum foil and sealed in plastic bag | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| | Stack gas Method 0010 solvents | Amber glass Boston round bottles with Teflon-lined lid | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| Stack gas metals | Method 29 filter | Glass or plastic petri dish | NA | 180 days/28 days for Hg |
| | Method 29 liquids | Amber glass Boston round bottles with Teflon-lined lid | NA | 180 days/28 days for Hg |
| Stack gas | Method 26A | Glass or plastic petri dish | NA | 180 days |
| | Method 26A | Amber glass Boston round bottles with Teflon-lined lid | NA | 180 days |
| Stack gas chloride | Method 26A | Amber glass Boston round bottles with Teflon-lined lid or polyethylene bottles | NA | 28 days |

Reference: Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January, 1990 and promulgated method

### 7.3 PROCESS SAMPLE COLLECTION FORMS

While a process sample is being taken in the field, the sampling technician will complete a field sampling record. An example field sampling record is presented as Figure 7-2. The field sampling record will be filled out in its entirety for every sample. This will provide information to be used in the final report. The sampling technician shall provide the completed field sampling record to the sample custodian with the associated sample.

### 7.4 SAMPLE COLLECTION FORMS

While sampling is being performed, the sampling technician will complete a sampling record. Figure 7-3 presents an example field sampling record for isokinetic sampling. The sampling record will be completed in its entirety for every sampling train. This will provide information necessary to perform the emissions calculations. The sampling technician shall provide the completed sampling record to the sampling coordinator at the completion of each sampling run.

### 7.5 SAMPLE LABELING

An example sample label format is presented in Figure 7-4. Each sample container will be labeled to show the source of the sample as INTEC, the project identification, sampler's initials, laboratory to which the sample will be shipped, the unique alphanumeric sample number, date and time, sample description, test number, and run number. If a single sample requires multiple containers, the number of the container and the total number of containers will be noted on the label. Project samples will be tracked by the assigned unique alphanumeric sample numbers. The sample number will appear on the sample label, the RFA, and the COC.

### 7.6 SAMPLE COLLECTION CHECKLIST

The master sample list identifies every sample by the assigned unique alphanumeric sample numbers (refer to Figure 7-1), and the corresponding analytical test(s) required. As field samples are acquired and routed through the sample custodian, the samples will be checked off against the master list to ensure that all the appropriate samples have been collected.

### 7.7 REQUEST FOR ANALYSIS AND CHAIN OF CUSTODY

Collected samples will be shipped from the site to the laboratory in sealed containers with COC and RFA forms. Example RFA and COC forms are presented as Figures 7-5 and 7-6, respectively. The sampling technician and sample custodian will complete the COC and RFA forms for every sample. Some samples may consist of several subsamples. Each individual component of the sample will be listed separately on the RFA or COC with its own unique alphanumeric sample identification number. The samples will be preserved as needed, and will remain in the possession of the sample custodian. The sample custodian will secure the samples in a location accessible only to authorized personnel until custody is transferred to a courier for delivery to the laboratory.

**Figure 7-2. Example Process Sampling Record**

| | | | |
|---|---|---|---|
| **Client:** | INTEC | | |
| **Location:** | Idaho National Laboratory | **Sheet No.:** | 1 of 1 |
| **Program:** | CPT | **Run Start:** | 0800 |
| **Stream:** | Simulant Feed | **Run End:** | 1335 |
| **Sampler:** | CHV | **Sample Frequency:** | One grab sample prior to test |
| **Date:** | 18-Nov-10 | **Sample Type:** | Grab |
| **Test No.:** | 1 | **Equip/Container:** | 4 oz. |
| **Run No.:** | 1 | **Source:** | Tap on feed line |

| Sample No. | Sample Time | Comment | Sample No. | Sample Time | Comment |
|---|---|---|---|---|---|
| 1 | 0800 | Run Start 0800 | 26 | : | |
| 2 | 0830 | | 27 | : | |
| 3 | 0900 | | 28 | : | |
| 4 | 0930 | | 29 | : | |
| 5 | 1000 | | 30 | : | |
| 6 | 1030 | | 31 | : | |
| 7 | 1100 | | 32 | : | |
| 8 | 1130 | | 33 | : | |
| 9 | 1200 | | 34 | : | |
| 10 | 1230 | | 35 | : | |
| 11 | 1330 | | 36 | : | |
| 12 | 1330 | Run End 1335 | 37 | : | |
| 13 | : | | 38 | : | |
| 14 | : | | 39 | : | |
| 15 | : | | 40 | : | |
| 16 | : | | 41 | : | |
| 17 | : | | 42 | : | |
| 18 | : | | 43 | : | |
| 19 | : | | 44 | : | |
| 20 | : | | 45 | : | |
| 21 | : | | 46 | : | |
| 22 | : | | 47 | : | |
| 23 | : | | 48 | : | |
| 24 | : | | 49 | : | |
| 25 | : | | 50 | : | |

**Total No. of Grabs:**
**Comments/Notes:**
Samples were prepared prior to the start of the test from a representative grab sample.
The balance of the sample was discarded.
Two 4 oz. for Archive

**Figure 7-3. Example Sampling Record**

**Isokinetic Stack Sample Data Collection Sheet**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Contract No. | 10905 | Method: | M29 (Metals) | | | Page 1 of 1 | |
| Facility | INTEC | Initial Leak Rate ($ft^3$ @ in.Hg) | | <0.010 @ 10" | | Operator | MJK |
| Source | IWTU | Final Leak Rate ($ft^3$ @ in.Hg) | | <0.010 @ 10" | | Pitot No. | K-004 |
| Date | 18-Nov-10 | Start Time | 0800 | Meter No. | 1442 | PTCF | 0.84 |
| Condition No. | 1 | End Time | 1015 | DGMCF | 1.004 | Init. Pitot Leak Check | 0.0 |
| Run No. | 1 | Duration (min) | 120 | $\Delta$ H @ | 1.645 | Final Pitot Leak Check | 0.0 |
| Stat. Press. (in. $H_2O$) | -0.94 | Bar. Press. (in.Hg) | 29.73 | Nozzle Dia.( ") | 0.251 | Kf | 2.3 |

| Point No. | Time (24 Hr) | Volume ($ft^3$) | $\Delta$ P (in. $H_2O$) | $\Delta$ H (in. $H_2O$) | Temperatures (°F) Flue Gas | Probe | Filter | Impingers | Meter in | Meter Out | Cond. Exit | Vacuum (in. Hg) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A-1 | 0800 | 704.25 | 0.78 | 1.79 | 294 | 225 | 258 | 68 | 103 | 105 | NA | 6.0 |
| A-1 | 0805 | 707.88 | 0.78 | 1.79 | 294 | 260 | 257 | 67 | 105 | 104 | | 6.0 |
| A-2 | 0810 | 711.50 | 0.74 | 0.17 | 294 | 257 | 261 | 66 | 106 | 104 | | 6.0 |
| A-2 | 0815 | 714.96 | 0.74 | 1.7 | 294 | 261 | 258 | 66 | 107 | 104 | | 6.0 |
| A-3 | 0820 | 718.09 | 0.68 | 1.56 | 292 | 258 | 257 | 64 | 108 | 104 | | 6.0 |
| A-3 | 0825 | 721.50 | 0.68 | 1.56 | 292 | 257 | 258 | 63 | 109 | 104 | | 6.0 |
| A-4 | 0830 | 725.36 | 0.77 | 1.77 | 292 | 258 | 258 | 67 | 104 | 104 | | 6.0 |
| A-4 | 0835 | 729.35 | 0.77 | 1.77 | 292 | 258 | 256 | 67 | 109 | 103 | | 6.0 |
| A-5 | 0840 | 733.20 | 0.77 | 1.7 | 293 | 258 | 259 | 61 | 110 | 104 | | 6.0 |
| A-5 | 0845 | 737.10 | 0.77 | 1.7 | 289 | 256 | 258 | 61 | 108 | 104 | | 6.0 |
| A-6 | 0850 | 740.67 | 0.74 | 1.43 | 285 | 259 | 255 | 62 | 106 | 105 | | 6.0 |
| A-6 | 0855 | 744.12 | 0.74 | 1.43 | 290 | 258 | 255 | 62 | 105 | 103 | | 6.0 |
| STOP | 0900 | 747.21 | | | | | | | | | | |
| B-1 | 0915 | 747.42 | 0.62 | 1.77 | 292 | 255 | 259 | 66 | 96 | 95 | | 7.0 |
| B-1 | 0920 | 750.94 | 0.62 | 1.77 | 292 | 259 | 257 | 65 | 96 | 95 | | 7.0 |
| B-2 | 0925 | 754.76 | 0.77 | 1.43 | 293 | 257 | 257 | 62 | 99 | 95 | | 7.0 |
| B-2 | 0930 | 758.21 | 0.77 | 1.29 | 289 | 257 | 258 | 62 | 102 | 95 | | 6.0 |
| B-3 | 0935 | 761.52 | 0.62 | 1.04 | 285 | 258 | 257 | 62 | 104 | 97 | | 6.0 |
| B-3 | 0940 | 764.52 | 0.56 | 1.1 | 290 | 257 | 253 | 61 | 106 | 99 | | 7.0 |
| B-4 | 0945 | 767.79 | 0.45 | 1.5 | 296 | 257 | 257 | 63 | 109 | 100 | | 6.0 |
| B-4 | 0950 | 767.92 | 0.56 | 1.5 | 296 | 253 | 258 | 64 | 111 | 101 | | 6.0 |
| B-5 | 0955 | 774.51 | 0.45 | 0.99 | 296 | 257 | 259 | 61 | 112 | 103 | | 6.0 |
| B-5 | 1000 | 777.52 | 0.44 | 0.94 | 297 | 258 | 258 | 60 | 112 | 104 | | 5.5 |
| B-6 | 1005 | 780.47 | 0.65 | 0.85 | 297 | 259 | 258 | 61 | 113 | 105 | | 5.5 |
| B-6 | 1010 | 783.17 | 0.65 | 0.85 | 294 | 258 | 257 | 61 | 112 | 105 | | 5.5 |
| END | 1015 | 785.94 | | | | | | | | | | |

Comments:

**Figure 7-4 Example Sample Label Format**

**INTEC IWTU Comprehensive Performance Test**
**Idaho National Laboratory**
**Focus Project No. 010905**

| | | | |
|---|---|---|---|
| **Sample Type:** | Method 29 Filter | **Sample No.:** | **K-3014** |
| **Test No.** ____1___ | **Run No.** ____1___ | **Container(s):** | ____ of ____ |
| **Analysis Required:** | Metals: Sb, As, Ba, Be, Cd, total Cr, Co, Pb, Mn, Hg, Ni, Se, Ag, Tl, & Zn | | |
| **Analysis Laboratory:** | Analytical Laboratory | | |
| **Date:** | 18-Nov-10 | **Initials:** | MJK |
| **Time:** | 1415 | **Preservation:** | NA |

**Figure 7-5. Example Request for Analysis**

**Request for Analysis/Chain of Custody No. 007**
**INTEC Comprehensive Performance Test**
**Idaho National Laboratory**

**Idaho National Laboratory**
**Focus Project No. 040809**

**Laboratory Lot No.** _H-XXXXXXXX_____
**Laboratory Project No.** ___XXXXXXX____

| | |
|---|---|
| **Project Description:** | IWTU CPT Program |
| **Client Project No.:** | 040809 |
| **Test America Project No:** | XXXXXXX |
| **Client Project Mgr:** | Anthony Eicher<br>865-694-7517 |
| **Test America Contact:** | Laboratory Contact<br>XXX-XXX-XXXX |
| **Test America Project Mgr:** | Laboratory Project Manager<br>XXX-XXX-XXXX |

**Laboratory Deliverable Requirements**

Analytical Due Date: 21 days from lab receipt

Data Package Due Date: 30 days from lab receipt

**Analytical Testing QC Requirements:**
MS - Matrix Spike
MSD - Matrix spike duplicate
PDS - Post-digestion spike
DUP - Duplicate
BT- Blank train

**Laboratory Destination:**
Laboratory Destination
Laboratory Name
Laboratory Address
Laboratory Address
XXX-XXX-XXXX
**Courier:** Hand deliver

**Project Deliverables:**

| **Holding Time Requirements:** | |
|---|---|
| ICAP Metals | 180 days to analysis |
| | |

| Field Sample No. | Test No. | Run No. | Sample Collection Date/ Time | Sample Container | Sample Description | Analysis Specifications | Project QC Requirements |
|---|---|---|---|---|---|---|---|
| K- 3014 | 1 | 1 | 18-Sep-09 1415 | 250 mL amber bottle | Method 29 Train Particulate Filter | Combine this sample with the corresponding nitric acid probe rinse, Sample No. K-3013. Perform Method 29 digestion and analyze via Method 6020 for: Sb, As, Ba, Be, Cd, total Cr, Co, Pb, Mn, Hg, Ni, Se, Ag, Tl, & Zn. | 0 |
| K- 3015 | 1 | 1 | 18-Sep-09 1415 | 1-liter amber | Method 29 Train $HNO_3$ Probe Rinse | Combine this sample with the corresponding filter, Sample No. K-3012. Perform Method 29 digestion and analyze via Method 6010B or 6020 for: Sb, As, Ba, Be, Cd, total Cr, Co, Pb, Mn, Hg, Ni, Se, Ag, Tl, & Zn. | |
| K- 3016 | 1 | 1 | 18-Sep-09 1415 | 250 mL amber bottle | Method 29 Train 5% $HNO_3$/10% $H_2O_2$ Impingers | Perform Method 29 digestion and analyze via Method 6020 for: Sb, As, Ba, Be, Cd, total Cr, Co, Pb, Mn, Hg, Ni, Se, Ag, Tl, & Zn. | MS/MSD |

**Figure 7-6. Example Chain of Custody**

**Request for Analysis/Chain of Custody No. 007**
**INTEC Comprehensive Performance Test**
**Idaho National Laboratory**
**Focus Project No. 010905**

**Sample Receipt Log and Condition of the Samples Upon Receipt**

Please fill in the following information: Comments

(Please write "NONE" if no comment is applicable.)

(1) Record the identities of any samples that were listed on the Request for Analysis form but were not found in the sample shipment _____

(2) Record the sample shipping cooler temperature of all coolers transporting samples listed on the Request for Analysis form. _____

(3) Record any apparent sample loss or breakage.

(4) Record any unidentified samples transported with this shipment of samples. _____

(5) Indicate if all samples were received according to the project's required specifications (i.e, no non-conformances). _____

**Custody Transfer**

Relinquished by: _____
Name Company Date/Time

Accepted by: _____
Name Company Date/Time

Relinquished by: _____
Name Company Date/Time

Accepted by: _____
Name Company Date/Time

Relinquished by: _____
Name Company Date/Time

Accepted by: _____
Name Company Date/Time

### 7.8 SAMPLE SHIPMENT

Field samples may be transported directly to the analytical laboratory by the test management or sampling contractor. If the samples are shipped by overnight courier, e.g. Federal Express, an individual trained in U.S. Department of Transportation and International Air Transport Association regulations will package the samples to ensure compliance with the applicable portions of these regulations.

Prior to shipping any samples, the sample custodian will verify the condition of the samples: sample temperatures for organic analyses samples, condition of all containers, level of sample within all containers (to be marked on the outside of the container), and type of packing material used. RFAs and COCs will be checked to verify there is a RFA and COC for every sample being shipped. Before shipping the samples from the site, the sample custodian shall make a photocopy set of all the RFAs and COCs to remain with the test coordinator.

### 7.9 SAMPLE DELIVERY

Upon receipt of samples at the laboratory, the receiver will accept custody for the shipment by an exchange of signatures with the delivering agent. The shipping containers will be opened by the laboratory analysis coordinator (or designee) and inspected. The container contents will be verified against the accompanying RFA and COC. Any damage to the contents of the shipping container or deviations from the original shipment documents will be noted on the COC. A labeled temperature blank (labeled volatile-organic analysis vial or sample bottle with water) will be shipped in every container with samples for organic analysis solely for the purpose of determining sample temperatures. The laboratory analysis coordinator or designee will, immediately upon opening the sample packaging, open the temperature blank and measure the temperature of the water inside the temperature blank using a thermometer. This temperature will be recorded on the COCs and any applicable laboratory documentation (sample receipt log).

Individual samples will be sorted and directed to the respective laboratory section responsible for the analyses noted on the RFA. Samples will be secured in a location accessible only to authorized personnel. Samples for organic analysis shall be secured in refrigerated sample storage. The COC forms are used specifically to track the samples. To provide specific instructions to the analysts, the RFAs will accompany the respective COCs.

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

### 7.10 SAMPLE PRESERVATION

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

**Table 7-1. Sample Containers, Preservation, and Holding Times**

| Parameter | Sample Name/Matrix | Sample Containers | Preservation | Maximum Holding Time |
|---|---|---|---|---|
| Gas<br>VOCs & TICs | Gas SMVOC tubes<br>Gas SMVOC condensate | Glass culture tube in foam cushioned plastic tube<br>Glass VOA vials with Teflon-lined lids or septum lids | Chill to 4°C<br>Chill to 4°C | 14 days<br>14 days |
| Gas<br>PCDD/PCDFs | Gas Method 0023A filter | Glass petri dish | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| | Gas Method 0023A sorbent tube | Standard cartridge wrapped in aluminum foil and sealed in plastic bag | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| | Gas Method 0023A solvents | Amber glass Boston round bottles with Teflon-lined lid | Chill to 4°C | 30 days until extraction<br>45 days after extraction |
| Gas SVOCs & TICs and PAHS | Gas Method 0010 filter | Glass petri dish | Chill to 4°C | 30 14 days until extraction<br>45 40 days after extraction |
| | Gas Method 0010 sorbent tube | Standard cartridge wrapped in aluminum foil and sealed in plastic bag | Chill to 4°C | 30 14 days until extraction<br>45 40 days after extraction |
| | Gas Method 0010 solvents | Amber glass Boston round bottles with Teflon-lined lid | Chill to 4°C | 30 14 days until extraction<br>45 40 days after extraction |
| Gas metals | Method 29 filter | Glass or plastic petri dish | NA | 180 days/28 days for Hg |
| | Method 29 liquids | Amber glass Boston round bottles with Teflon-lined lid | NA | 180 days/28 days for Hg |
| Gas | Method 26A | Glass or plastic petri dish | NA | 180 days |
| | Method 26A | Amber glass Boston round bottles with Teflon-lined lid | NA | 180 days |
| Gas chloride | Method 26A | Amber glass Boston round bottles with Teflon-lined lid or polyethylene bottles | NA | 28 days |

Reference: Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January, 1990 and promulgated method

The onsite location of the SMVOC sample holding area is of special importance. The SMVOC tubes will be stored in a clean area away from where all other process off-gas and process samples are handled both before and after sampling. High concentration volatile organic samples (recovered elements of other sampling trains, waste feed samples, etc.) must be segregated to prevent inadvertent contamination of the SMVOC samples. The SMVOC tube sets and SMVOC condensate samples will be preserved after sampling by placing them on double bagged ice or blue ice in a clean, preferably previously unused, dedicated sample cooler. To preclude contamination from solvents and process samples, all SMVOC samples will be shipped in dedicated shipping containers separate from all other test samples.

XAD-2 traps and other train sample components for the Method 0023A PCDD/PCDF and Method 0010 SVOC and PAH trains will be preserved after sampling by placing them in a cooler on double bagged ice or blue ice. Solvent samples from these sampling trains may be stored and shipped with the XAD-2 traps.

For nonorganic analysis process off-gas samples (particulate, chloride, etc.), sample preservatives (if applicable, refer to Table 7-1) will be used as required by the target analyte. These samples will be stored in dedicated sample packaging or coolers. These samples do not require chilling.

## 8.0 SPECIFIC CALIBRATION PROCEDURES AND FREQUENCY

### 8.1 GENERAL

Equipment and instruments used to generate data for determining compliance with performance requirements or to establish quantitative allowable operating limits will be calibrated according to the manufacturer's instructions, prior to or during the test or both, as necessary. The calibration procedures are separated into groups according to the personnel who will perform them. The process instruments will be calibrated by INTEC operations personnel. Sampling equipment will be calibrated by the sampling contractor. Analytical instruments will be calibrated by the contracted laboratory personnel. The calibration procedures for process instrumentation process off-gas sampling, and laboratory analytical instruments systems are described in the following subsections.

### 8.2 PROCESS INSTRUMENTATION

The parameter continuous monitoring system (CMS) (thermocouples, flow meters, pressure transducers, etc.) will be calibrated in accordance with INTEC SOPs within 30 days of the start of testing.

The CEMS (installed carbon monoxide and oxygen monitors) will undergo a relative accuracy test audit within 90 days prior to the test. The relative accuracy test audit will include calibration drift tests, response time tests, calibration error tests, and relative accuracy tests per the appendix to 40 CFR 63, Subpart EEE. The installed carbon monoxide and oxygen CEMS will be calibrated daily before testing. The zero and span checks will be considered a verification of the data quality from these monitors.

CMS and CEMS data will be reported in 1-minute intervals and will be archived in the CMS data acquisition system.

### 8.3 SAMPLING EQUIPMENT

Sampling equipment is calibrated according to the criteria specified in the reference method being used. In addition, the guidelines set forth in the *Quality Assurance Handbook for Air Pollution Measurement Systems*, Volume III, "Stationary Source Specific Methods" (EPA-600/4-77-027b) will be followed. Dry gas meters, orifices, nozzles, pitot tubes, etc. are calibrated in accordance with this document. The range of the calibration is specified for all environmental measurements to encompass the range of probable experimental values. This approach ensures that all results are based upon interpolative analyses rather than extrapolative analyses.

Calibrations are designed to include, where practical, at least three measurement points evenly spaced over the range. This practice minimizes the probability of making false assumptions of calibration linearity. In addition, it is common practice to select, when practical, at least one calibration value approximating the levels anticipated in the actual measurement. Typically, calibration frequency is dictated by the need to demonstrate the stability of the calibration value over the course of measurements. Calibrations are made both pre- and post-test to demonstrate stability.

Following the SPT, calibrations are checked on all relevant items of sampling equipment to ensure the validity of data collected in the field. New items that require calibration are calibrated before initial field use. Equipment whose calibration status may change with use or time is inspected in the field before testing begins and again upon return from each field use. When an item of equipment is found to be out of calibration, it is repaired and recalibrated or retired from service. All equipment is periodically recalibrated in full, regardless of the outcome of these regular inspections.

Data obtained during calibrations are recorded on standardized forms, which are checked for completeness and accuracy by management. Data reduction and subsequent calculations are performed using standard procedures, and are computerized, where appropriate. Calculations are checked at least twice for accuracy. Copies of calibration forms are included in the test or project reports.

Emissions sampling equipment requiring calibration include pitot tubes, pressure gauges, thermometers, dry gas meters, and barometers. The following sections elaborate on the calibration procedures for these specific equipment items.

#### 8.3.1 Pitot Tubes

All Type S pitot tubes, whether separate or attached to a sampling probe, are inspected in accordance with the geometry standards in EPA Method 2.

For Type S pitot tubes with a $D_t$ between 3/16 and 3/8 inches, the pitot tube may be calibrated according to the procedure outlined in Sections 10.1.2 through 10.1.5 of Method 2 before and after the test, or a baseline (isolated tube) coefficient value of 0.84 may be assigned.

All Type S pitot tubes greater than 3/8 inches are calibrated over an eight-point range with a wind tunnel, and a calibration coefficient is calculated for each pitot tube. The acceptance limits are listed in Table 8-1.

#### 8.3.2 Differential Pressure Gauges

Some meter consoles are equipped with 10-inch water column inclined-vertical manometers. Fluid manometers do not require calibration other than leak checks. Manometers are leak-checked in the field prior to each test series.

#### 8.3.3 Digital Temperature Indicator

One digital temperature indicator is used to determine the flue gas temperature, probe temperature, oven temperature, "train temperature," and dry gas meter temperature. The digital temperature indicator is calibrated over a seven-point range (32–450°F) using an ASTM mercury-in-glass thermometer as a reference. The calibration is acceptable if the agreement is within ±2% or 2°F from 50–180°F.

#### 8.3.4 Dry Gas Meter and Orifice

A calibrated wet test meter is used to calibrate the dry gas meter and orifice. The full calibration procedure is used to obtain the calibration factor of the dry gas meter. Full calibrations are performed using a calibrated wet test meter as a reference standard.

**Table 8-1. Sampling Equipment Calibration Requirements**
Page 1 of 3

| Sampling Equipment | Acceptance Criteria | Measurement Frequency | Action If Criteria Are Not Met |
|---|---|---|---|
| Volumetric Flow Measurements | | | |
| Type S pitot tube inspection | All dimension specifications met | Calibrate prior to test and visually inspect after each field test | Use pitot tubes that meet face opening specifications; repair or replace as required. |
| Type S pitot tube calibration | ± 3% for volumetric flows >1,000 fpm<br>± 5–6% for volumetric flows >600 and <1,000 fpm | Refer to Section 10.0 of Method 2: If $D_t$ is between 0.48 and 0.95 cm (3/16 and 3/8 in.), and if $P_A$ and $P_B$ are equal and between 1.05 and 1.50 $D_t$, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 10.1.2 through 10.1.5 of Method 2 before and after the test, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube with pitot tube inspection before and after the test | Check for blockage. If blockage is significant, recalculate calibration coefficient. |
| Barometers | ± 0.1 inches Hg (± 2.5 mm Hg) of mercury-in-glass barometer | Calibrate initially versus mercury-in-glass barometer; check before and after field test | Adjust to agree with a certified barometer. |
| Process off-gas temperature measurement system | Capable of measuring within ± 2°F (± 1°C) of mercury-in-glass thermometer | Calibrate prior to test and after each field use | Adjust to agree with Hg bulb thermometer; construct calibration curve, correct readings. |
| Pressure sensors (excludes inclined manometer) | Agree within ± 5% of inclined manometers | Prior to and after field use | Adjust to agree with Hg bulb thermometer; construct calibration curve, correct readings. |
| Wet test meter | $Y_{mi} = Y_m \pm 0.030$ Y (before test)<br>$Y_{mf}/Y_{mi}$ is 0.95 to 1.05 (after test) | Calibration prior to test<br>Check calibration after test | Before test: Adjust until specifications are met.<br>After test: Recalculate calibration coefficient. |

**Table 8-1. Sampling Equipment Calibration Requirements**
Page 2 of 3

| Sampling Equipment | Acceptance Criteria | Measurement Frequency | Action If Criteria Are Not Met |
|---|---|---|---|
| Volumetric Flow Measurements | | | |
| Dry gas meters | $Y_i = Y \pm 0.02\ Y$ (before test)<br>$Y_{mf}/Y_{mi}$ is 0.95 to 1.05 (after test) | Calibration versus wet test meter initially, and when post-check exceeds $\Delta Y \pm 0.05$ | Before test: Repair or replace and then calibrate.<br>After test: Recalculate calibration coefficient. |
| Assembled isokinetic sampling train leakage | 0.02 cubic feet per minute (cfm) (0.00057 $m^3$/min) at vacuum of $\geq$ 10 inches Hg (250 mm Hg) before the start of sampling, and $\geq$ maximum vacuum value recorded during sampling run for post-sampling leak checks | Just prior to start of first sampling traverse (required); after first sampling traverse (recommended); after moving sampling train from first traverse port to second traverse port (recommended); end of second sampling traverse (required). | Before sampling or at post port change: Isolate and repair leak point(s); repeat leak check.<br>End of first traverse or end of second traverse: Determine leak rate; If <4% of sampling rate, correct sample volume per procedures in Section 6.3 of Method 5.<br>If >4% of sampling rate, invalid test sample; discard sample and repeat sampling run for invalid train. |
| Tedlar bags | Any water manometer displacement after 10 minutes when inflated to 2 to 4 inches (5 to 10 cm) water column pressure | Every bag | Discard bag. |
| Orsat analyzer leak check | Using bag or calibration gas, bring liquid level in each pipette to the reference mark and close stopcock. Burette meniscus changes < 0.2 mL after 4 minutes. | Before and after test | Replace leaking tubing or stopcock valves, or defective hose tubing clamps. |
| Viability of Orsat analyzer solutions | Using oxygen and carbon dioxide calibration gases, conduct Orsat analyses; $\pm$0.5% of calibration standard | Before and after test | Replace spent absorbing solution with fresh solution. |
| Analytical balance (for moisture) | ± 1 mg of Class-S weights | Check with Class-S weights upon receipt and daily | Adjust or repair. |

**Table 8-1. Sampling Equipment Calibration Requirements**
Page 3 of 3

| Sampling Equipment | Acceptance Criteria | Measurement Frequency | Action If Criteria Are Not Met |
|---|---|---|---|
| Sampling Train Heating Systems and Thermocouples | | | |
| Probe heating system (isokinetic sampling trains) | Capable of maintaining 248° ± 25°F (120°C ± 14°C) at a flow of 0.75 cfm (21.2 L/min) | Calibrate initially by APTD-0576(11) or use published calibration curves | Repair, or replace, and then verify the calibration. |
| Probe nozzle (isokinetic sampling trains) | Average of three ID measurements of nozzle within 0.001 inches (0.0025 mm); difference between high and low 0.002 inches (0.0050 mm) | Use a micrometer to measure to nearest 0.025 mm (0.001 in.); check before and after field test | Recalibrate, reshape, and sharpen when nozzle becomes nicked, dented, or corroded. |
| Probe heating system Sampling Method for Volatile Organics Compounds (SMVOC) Train | Maintained at a temperature >130°C (>266°F) | Periodic check during sampling | Immediately increase the SMVOC system to the proper temperature. |
| Thermocouples (process off-gas meters and final impinger) | Impinger thermocouple ± 1°C (2°F) [Method 5]; dry gas thermocouple ± 3°C (5.4°F) [Method 0010]; thermocouple within ± 1°C (± 2°F) of absolute temperature. | Calibrate prior to test against a mercury thermometer | Adjust; determine a correction factor or reject. |

Sources:
*Quality Assurance Handbook for Air Pollution Measurement Systems*, Volume III, "Stationary Source Specific Methods," EPA-600/R-94/038c, EPA, September 1994.
*Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment,* APTD-0576, U.S. EPA, Office of Air Programs, March 1972.
*Construction of Isokinetic Source Sampling Equipment*, APTD-0581, U.S. EPA, Office of Air Programs, April 1971.
Method 0031, "Sampling Method for Volatile Organics Compounds," and Method 0010, "Modified Method 5 Sampling Train," *Test Method for Evaluating Solid Waste Physical/Chemical Methods*, SW-846, EPA, Office of Solid Waste, Update III, December 1997.
"Determination of Particulate Emissions from Stationary Sources," *Test Methods and Procedures, New Source Performance Standards,* 40 CFR 60, Appendix A, EPA.

#### 8.3.4.1 Dry Gas Meter

Each metering system is fully calibrated at the time of purchase and quarterly. Upon request, a post-test calibration can be performed after each field use. If the calibration factor deviates by less than 5% from the initial value, the test data are acceptable. If it deviates by more than 5%, the meter is recalibrated, and the meter coefficient (initial or recalibrated) that yields the lowest sample volume for the test runs is used.

EPA Method 5 requires another full calibration anytime the post-test calibration check indicates that the calibration factor has changed by more than 5%. Standard practice is to recalibrate the dry gas meter quarterly and check the orifice calibration during and after each field use.

#### 8.3.4.2 Orifice

An orifice calibration factor is calculated for each of the 18 flow settings during a full calibration. The arithmetic average of the values obtained during the calibration is used.

### 8.3.5 Barometer

Each field barometer is adjusted before each test series to agree within ±0.1 inches of a reference aneroid barometer. The reference barometer is checked against the weather station pressure value (corrected for elevation difference) reported by the National Weather Service or mercury barometer. This information is obtained by calling the weather line or the nearest airport.

## 8.4 LABORATORY ANALYTICAL EQUIPMENT

The laboratory instruments will be calibrated as specified by the appropriate method before analyzing the test samples. The laboratory instrument calibration procedures are based on instructions in the referenced analytical methods and are summarized, along with other routine QC checks, in Table 8-2. The calibrations performed and the results will be reported as appropriate to ensure the quality of data in the laboratory sample analysis report.

**Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions**

Page 1 of 4

| Parameter/Method | Quality Control Check | Method of Determination | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| Volatile Organic Compounds & TICs by GC/MS (SW846 8260B) | Initial calibration | 3 - 5 standards bracketing expected concentrations | Prior to sample analysis | Variability of average RRF less than or equal to 20% RSD for CCCs. SPCCs (chlorobenzene and 1,1,2,2-tetrachloroethane) will be ≥ 0.3 and SPCCs (chloroethane, 1,1-dichloroethane and bromoform) will be ≥ 0.1 | Recalibrate |
| | Continuing calibration | Midlevel standard | Prior to sample analysis, then every 12 hours or after sample set | RRF for CCCs within 25% difference of the initial calibration average RRF. SPCCs (chlorobenzene and 1,1,2,2-tetrachloroethane) will be ≥ 0.3 and SPCCs (chloroethane, 1,1-dichloroethane and | Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check. |
| | Consistency in chromatography | For MS methods, monitor internal standard retention time and area. For non-MS methods, monitor retention time windows for componds of interest. | Every sample, standard, and blank | Retention time within 30 seconds of last calibration check. Area within -50 to +100% of last calibration check | Perform calibration standard check. Reanalyze sample if possible, or flag data. |
| | Calibration check | Analysis of independent calibration check standard | In association with each initial calibration | Within 3 std. deviations of historical mean (laboratory specific) | Recalibrate and recheck. |
| | Method blank | Analysis of blank | Analyze one with each analytical batch | Result less than method detection limit | Flag data and discuss in case narrative. |

**Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions**
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| Parameter/Method | Quality Control Check | Method of Determination | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| Semivolatile Organic Compounds & TICs and PAHs by GC/MS (SW846 8270C) | Initial calibration | 5 standards bracketing expected concentrations. Critical level should be at least 10 times higher than lowest standard. | Prior to sample analysis | Variability of average RRF less than or equal to 30% RSD for CCCs. SPCCs will be greater than or equal to 0.05. | Recalibrate |
| | Continuing calibration | Midlevel standard | Prior to sample analysis, then every 12 hours or after sample set | RRF for CCCs within 20% difference of the initial calibration average RRF. SPCCs will be greater than or equal to 0.05. | Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check. |
| | Consistency in chromatography | For MS methods, monitor internal standard retention time and area. For non-MS methods, monitor retention time windows for componds of interest. | Every sample, standard, and blank | Retention time within 30 seconds of last calibration check. Area within -50 to +100% of last calibration check | Perform calibration standard check. Reanalyze sample if possible, or flag data. |
| | Calibration check | Analysis of independent calibration check standard | In association with each initial calibration | Within 3 std. deviations of historical mean (laboratory specific) | Recalibrate and recheck. |
| | Method blank | Analysis of blank | Analyze one with each analytical batch | Result less than method detection limit | Flag data and discuss in case narrative. |

**Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions**
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| Parameter/Method | Quality Control Check | Method of Determination | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| PCDD/PCDF by High Resolution GC/High Resolution MS (SW846 8290) | Initial Calibration | All five high resolution concentration calibration solutions must be used for the initial calibration | Prior to sample analysis | PCDD/PCDF: The %RSD for the mean RRF from the each of the unlabeled standards must not exceed ± 20%, and those for the labeled reference compounds must not exceed ± 30%. | Recalibrate |
| | Continuing Calibration | Midlevel standard | At the beginning and end of each 12 hour shift | PCDD/PCDF: RFs must be within ± 20% of the initial calibration mean RRF for unlabeled standards and ± 30% for labeled standards | Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check. |
| | Retention time window verification and GC column performance | Monitor retention times | Start of each 12 hour shift | PCDD/PCDF: Compliance with Section 8.2.1 of Method 8290 | Correct according to method |
| | Method Blank | Analysis of blanks | Analyze one with each analytical batch | Results less than method detection limit | Flag data as discussed in case narrative |
| Metals by ICP or CVAA (SW846 6010B ICP and CVAA by SW846 7240) | Initial calibration | Multiple standards (CVAA) or 1 standard (ICP) and a calibration blank, bracketing the expected concentrations. Critical level should be at least twice the lowest calibration standard. | Prior to sample analysis | Correlation coefficient of linear plot >0.995 (CVAA). Not applicable for ICP. | Recalibrate |
| | Reagent blank | Analysis of blank | After every 10 samples and at end of analysis | Less than instrument detection limit (IDL) | Reanalyze if greater than the reporting limit and discuss in case narrative if greater than the IDL |
| | Calibration check | Analysis of independent calibration check standard | Once after initial calibration | 90 - 110% of theoretical value | Reanalyze and recalibrate, if necessary |
| | Post digestion spike | Analysis of post digest spike, spiked at 2 to 5 times the original sample value | One sample per test for ICP | 85 - 115% of theoretical value | Flag data; discuss in case narrative |

**Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions**
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| Parameter/Method | Quality Control Check | Method of Determination | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| Metals by ICP or CVAA (SW846 6010B ICP and CVAA by SW846 7240) Cont. | Calibration accuracy (ICP) | Reanalysis of high level standard | After every initial calibration | 90 - 110% of theoretical value | Recalibrate and recheck |
| | Interference check (ICP) | Analysis of interference sample | After every initial calibration and at the end of each run | 80 - 120% of theoretical value | Recalibrate and recheck |
| | Continuing calibration | Midlevel standard and blank | Beginning and end of each analysis period and after every 10 samples | ICP – Midlevel standard 90 - 110% of theoretical value; blank <50% of lowest calibration standard or within 3 SD of average blank. | Identify and correct problems; reanalyze samples run since last acceptable continuing calibration check. |
| Particulate and Gravimetric (EPA Method 26A) | Calibration check | Calibration of balance with standardized weights | Prior to analysis, between each group of sample weighings, and at the end of each day. | 99 – 101% of theoretical value | Recalibrate and recheck sample weights. |
| Chloride by Ion Chromatography (EPA Method 26A) | Initial Calibration | 4 standards bracketing expected concentrations Note: | Prior to sample analysis | Linear correlation coefficient >0.995 | Recalibrate |
| | Retention time check for ion identification | Determine average retention time for ions of interest or relative retention time of several ions for every calibration curve | Prior to sample analysis | Average Retention Time -Sample identification is positive if results are within retention time window of standards<br>Relative Retention Time – Sample identification is positive if results are within 3 SD of average RRT | Ions of interest are not present if criteria are not met. |
| | Control check sample | Midlevel independent standard analyzed in duplicate | Beginning and end of each analysis period and after every 10 samples | 90 – 110% of theoretical value | Repeat calibration check. If second check fails criteria, regenerate analytical system and reanalyze all samples since last acceptable calibration check. |
| | Reagent blank (ICB and CCBs) | Analysis of blanks | Immediately following the ICV and following each CCV. | Less than 1 mg/L | Contamination source must be found and corrected. All samples analyzed since the last acceptable CCB must be reanalyzed. |
| | Reagent blank (ICB and CCBs) | Analysis of blanks | Immediately following the ICV and following each CCV. | Less than 1 mg/L | Contamination source must be found and corrected. All samples analyzed since the last acceptable CCB must be reanalyzed. |

## 9.0 ANALYTICAL PROCEDURES

Analytical procedures and methods are summarized in Table 9-1. Individual analytical methods are described in detail in Appendix B of the SPT plan and are incorporated here by reference. The following is a list of the analytical reference methods for the procedures presented in Table 9-1:

- *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition, November 1986 and updates
- *Sampling and Analysis Methods for Hazardous Waste Incineration*, EPA 600/8-84-020, March 1993
- *Annual Book of ASTM Standards*, American Society for Testing and Materials, Philadelphia, Pennsylvania, Annual Series
- "Test Methods and Procedures, New Source Performance Standards," 40 CFR 60, Appendix A, December 2009
- *Methods for Chemical Analysis of Water and Wastes*, EPA 600/4-79-020, March 1993
- *Guidance for Total Organics*, EPA/600/R-96/036, March 1996
- "Quality Assurance Procedures for Continuous Emissions Monitors Used for Hazardous Waste Combustors," 40 CFR 63, Appendix to Subpart EEE, February 2010.

Table 9-2 presents the volatile organics target compounds for the Method 0031 samples.

Table 9-3 presents the semivolatile organics target compounds for the Method 0010 samples.

Table 9-4 presents the PAH target compounds for the Method 0010 samples.

Table 9-5 presents the PCDD/PCDF target analytes for the Method 0023A samples.

Table 9-6 presents the metals target analytes for the Method 29 samples.

The specifics of the sampling and analytical methodologies followed during the test by the selected sampling and analytical contractors include the referenced nationally recognized methods (ASTM, SW846, 40 CFR 60, Appendix A, etc.) or the contractor-specific SOPs or both.

During sampling and analysis, situations may still arise that require modifying the specific sampling or analytical procedures included or referenced in the SPT plan or this QAPjP. The laboratory SOPs, which include a number of procedures for special circumstances, will be followed. If the laboratory finds it necessary to make adjustments to the analysis methods, the changes will be documented following the corrective action procedures in Section 14.0 of the QAPjP. Any such changes must be approved by the test manager and QAO.

**Table 9-1. Summary of Analytical Procedures and Methods**

| Analysis | Sample Name | Sample Matrix | Analytical Procedure | Reference Method |
|---|---|---|---|---|
| Volatile Target Compounds and TICs | SMVOC Tubes | Tenax – Tenax – Anasorb | Thermal desorption, purge & trap, GC/MS | SW846, 5041A & 8260B |
| | SMVOC condensate | Aqueous liquid | Purge & trap, GC/MS | SW846 8260B |
| Semivolatile Target Compounds & TICs and PAHs | Method 0010 train particulate filter and front half solvent rinses | Particulate filter & front half methylene chloride/acetone solvent rinses | Soxhlet extraction, GC/MS | SW 846 3542 & 8270C |
| | Method 0010 train XAD-2 resin and back half solvent rinses | XAD-2 resin and back half methylene chloride/acetone solvent rinses | Soxhlet extraction, GC/MS | SW 846 3542 & 8270C |
| | Method 0010 train condensate | Aqueous liquid | Liquid/liquid extraction, GC/MS | SW 846 3542 & 8270C |
| PCDD/PCDFs | Method 0023A train particulate filter, XAD-2 resin, and methylene chloride, acetone and toluene solvent rinses | Particulate filter, XAD-2 resin and methylene chloride/acetone solvent rinses | Soxhlet extraction, HRGC/HRMS | EPA 0023A & SW846 8290 |
| Metals | Method 29 filter and nitric acid proble rinses | Particulate filter and front half 0.1N nitric acid rinses | Acid digestion, ICP, CVAA | EPA Method 29; SW846 3050, 6010B, 6020A, 7470 |
| | Method 29 acidified peroxide impinger solutions | 5% nitric acid/10% hydrogen peroxide | Acid digestion, ICP, CVAA | EPA Method 29; SW846 3050, 6010B, 6020A, 7470 |
| | Method 29 empty impinger rinse | 0.1N nitric acid rinses | Acid digestion, CVAA | EPA Method 29, 7470 |
| | Method 29 acidified permanganate impinger solutions | 4% potassium permanganate impinger solution/10% sulfuric acid | Acid digestion, CVAA | EPA Method 29, 7470 |
| | Method 29 acidified permanganate impinger hydrochloric acid rinses | 8N hydrochloric acid | Acid digestion, CVAA | EPA Method 29, 7470 |
| Particulate | Method 26A filter and acetone probe rinses | Particulate filter and acetone probe rinses | Gravimetric | EPA Method 5 |
| Hydrogen Chloride (HCl) | Method 26A sulfuric acid impinger solutions | 0.1 N nitric acid rinses | Ion chromatography | EPA Method 26A; SW846 9057 |
| Chlorine ($Cl_2$) | Method 26A sodium hydroxide impinger solutions | 0.1N sodium hydroxide | Ion chromatography | EPA Method 26A; SW846 9057 |

ASTM American Society for Testing and Materials
EPA Method New Source Performance Standards, Test Methods and Procedures, Appendix A, 40 CFR 60.
SW846 Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates.

**Table 9-2. Target Volatile Organic Compounds**

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| Volatile Compound | CAS Number |
|---|---|
| Acetone | 67-64-1 |
| Acetonitrile | 75-05-8 |
| Acrylonitrile | 107-13-1 |
| Benzene | 71-43-2 |
| Bromobenzene | 108-86-1 |
| Bromochloromethane | 74-97-5 |
| Bromodichloromethane | 75-27-4 |
| Bromoform | 75-25-2 |
| Bromomethane | 74-83-9 |
| 2-Butanone (MEK) | 78-93-3 |
| n-Butylbenzene | 104-51-8 |
| sec-Butylbenzene | 135-98-8 |
| tert-butylbenzene | 98-06-6 |
| Carbon Disulfide | 75-15-0 |
| Carbon tetrachloride | 56-23-3 |
| Chlorobenzene | 108-90-7 |
| Chlorodibromomethane | 96-12-8 |
| Chloroethane | 75-00-3 |
| Chloroform | 67-66-3 |
| Chloromethane | 74-87-3 |
| 2-Chlorotoluene | 95-49-8 |
| 4-Chlorotoluene | 106-43-4 |
| 1,2-Dibromo-3-chloropropane | 96-12-8 |
| 1,2-Dibromoethane | 106-93-4 |
| Dibromomethane | 74-95-3 |
| 1,2-Dichlorobenzene | 95-50-1 |
| 1,3-Dichlorobenzene | 541-73-1 |
| 1,4-Dichlorobenzene | 106-46-7 |
| Dichlorodifluoromethane | 75-71-8 |
| 1,1-Dichloroethane | 75-34-3 |
| 1,2-Dichloroethane | 107-06-2 |
| 1,1-Dichloroethene | 75-35-4 |
| cis-1,2-Dichloroethene | 156-59-2 |
| trans-1,2-Dichloroethene | 156-60-5 |
| 1,2-Dichloropropane | 78-87-5 |
| 1,3-Dichloropropane | 142-28-9 |
| 2,2-Dichloropropane | 594-20-7 |
| 1,1-Dichloropropene | 563-58-6 |
| cis-1,3-Dichloropropene | 10061-01-5 |
| trans-1.3-Dichloropropene | 10061-02-6 |
| Ethylbenzene | 100-41-4 |
| Hexachlorobutadiene | 87-68-3 |
| 2-Hexanone | 591-78-6 |
| Iodomethane | 74-88-4 |
| Isopropylbenzene | 98-82-8 |
| p-Isopropyltoluene | 99-87-6 |

**Table 9-2. Target Volatile Organic Compounds**

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| Volatile Compound | CAS Number |
|---|---|
| Methylene chloride | 75-09-2 |
| 4-Methyl-2-pentanone (MIBK) | 108-10-1 |
| n-Propylbenzene | 103-65-1 |
| Styrene | 100-42-5 |
| 1,1,1,2-Tetrachloroethane | 630-20-6 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 |
| Tetrachloroethene | 127-18-4 |
| Toluene | 108-88-3 |
| 1,2,3-Trichlorobenzene | 87-61-6 |
| 1,2,4-Trichlorobenzene | 120-82-1 |
| Trichloroethane, 1,1,1- | 71-55-6 |
| Trichloroethane,1,1,2- | 79-00-5 |
| Trichloroethene | 79-01-6 |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | 76-13-1 |
| Trichlorofluoromethane | 75-69-4 |
| 1,2,3-Trichloropropane | 96-18-4 |
| 1,2,4-Trimethylbenzene | 95-63-6 |
| 1,3,5-Trimethylbenzene | 108-67-8 |
| Vinyl acetate | 108-05-4 |
| Vinyl chloride | 75-01-4 |
| m & p-Xylene | 179601-23-1 |
| o-Xylene | 95-47-6 |
| Xylenes (total) | 1330-20-7 |

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**Table 9-3. Target Semivolatile Compounds**

Page 1 of 2

| Semivolatile Compound | CAS Number |
|---|---|
| Acetophenone | 98-86-2 |
| Aniline | 62-53-3 |
| Benzaldehyde | 100-52-7 |
| Benzidine | 92-87-5 |
| Benzoic acid | 65-85-0 |
| Benzonitrile | 100-47-0 |
| Benzyl alcohol | 100-51-6 |
| bis(2-Chloroethoxy) methane | 111-91-1 |
| bis(2-Chloroethyl) ether | 111-44-4 |
| bis(2-ethylhexyl) phthalate | 117-81-7 |
| 4-Bromophenyl-phenylether | 101-55-3 |
| Butyl benzyl phthalate | 85-68-7 |
| Carbazole | 132-32-1 |
| 4-Chloro-3-methylphenol | 59-50-7 |
| 4-Chloroaniline | 106-47-8 |
| 2-Chlorophenol | 95-57-8 |
| 4-Chlorophenyl-phenylether | 7005-72-3 |
| Dibenzofuran | 132-64-9 |
| 1,2-Dichlorobenzene | 95-50-1 |
| 1,3-Dichlorobenzene | 541-73-1 |
| 1,4-Dichlorobenzene | 106-46-7 |
| 3,3'-Dichlorobenzidine | 91-94-1 |
| 2,4-Dichlorophenol | 120-83-2 |
| Diethyl phthalate | 84-66-2 |
| Dimethyl phthalate | 131-11-3 |
| 2,4-Dimethylphenol | 105-67-9 |
| Di-n-butyl phthalate | 84-74-2 |
| 4,6-Dinitro-2-methylphenol | 534-52-1 |
| 1,3-Dinitrobenzene | 99-65-0 |
| 2,4-Dinitrophenol | 51-28-5 |
| 2,4-Dinitrotoluene | 121-14-2 |
| 2,6-Dinitrotoluene | 606-20-2 |
| Di-n-octylphthalate | 117-84-0 |
| Diphenylamine | 122-39-4 |
| 1,2-Diphenylhydrazine | 122-66-7 |
| Hexachlorobenzene | 118-74-1 |
| Hexachlorobutadiene | 87-68-3 |
| Hexachlorocyclopentadiene | 77-47-4 |
| Hexachloroethane | 118-74-1 |
| Isophorone | 78-59-1 |
| 2-Methylphenol (o-Cresol) | 95-48-7 |
| 3 & 4-Methylphenol (m- & p-Cresol) | 65794-96-9 |

**Table 9-3. Target Semivolatile Compounds**

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| Semivolatile Compound | CAS Number |
|---|---|
| 2-Nitroaniline | 88-74-4 |
| 3-Nitroaniline | 99-09-2 |
| 4-Nitroaniline | 100-01-6 |
| Nitrobenzene | 98-95-3 |
| 2-Nitrophenol | 88-75-5 |
| 4-Nitrophenol | 100-02-7 |
| N-Nitrosodimethylamine | 62-75-9 |
| N-Nitroso-di-n-propylamine | 621-64-7 |
| N-Nitrosodiphenylamine | 86-30-6 |
| 2,2'-Oxybis (1-Chloropropane) | 108-60-1 |
| Pentachlorobenzene | 608-93-5 |
| Pentachloronitrobenzene | 82-68-8 |
| Pentachlorophenol | 87-86-5 |
| Phenol | 180-95-2 |
| Pyridine | 110-86-1 |
| 1,2,4-Trichlorobenzene | 120-82-1 |
| 2,4,5-Trichlorophenol | 95-95-4 |
| 2,4,6-Trichlorophenol | 88-06-2 |
| 1,2,4,5-Tetrachlorobenzene | 95-94-3 |

CAS = Chemical Abstract Service

**Table 9-4. Target Polyaromatic Hydrocarbon Compounds**

| PAH Compound | CAS Number |
|---|---|
| Acenaphthene | 83-32-9 |
| Acenaphthylene | 208-96-8 |
| Anthracene | 120-12-7 |
| Benzo(a)anthracene | 56-55-3 |
| Benzo(a)pyrene | 50-32-8 |
| Benzo(b)fluoranthene | 205-99-2 |
| Benzo(e)pyrene | 192-97-8 |
| Benzo(ghi)perylene | 191-24-2 |
| Benzo(k)fluoranthene | 207-08-9 |
| 2-Chloronaphthalene | 91-58-7 |
| Chrysene | 218-01-9 |
| Dibenz(a,h)anthracene | 53-70-3 |
| Fluoranthene | 206-44-0 |
| Fluorene | 86-73-7 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 |
| 2-Methylnaphthlene | 91-57-6 |
| Naphthalene | 91-20-3 |
| Perylene | 198-55-0 |
| Phenanthrene | 85-01-8 |
| Pyrene | 129-00-0 |

CAS = Chemical Abstract Service

**Table 9-5. Target Dioxin/Furan Compounds**

| Dioxin/Furan Compound | CAS Number |
|---|---|
| 2,3,7,8-TCDD | 1746-01-6 |
| Total TCDD | 41903-57-5 |
| 2,3,7,8-TCDF | 51207-31-9 |
| Total TCDF | 30402-14-3 |
| 1,2,3,7,8-PeCDD | 40321-76-4 |
| Total PeCDD | 36088-22-9 |
| 1,2,3,7,8-PeCDF | 57117-41-6 |
| 2,3,4,7,8-PeCDF | 57117-31-4 |
| Total PeCDF | 30402-15-4 |
| 1,2,3,6,7,8-HxCDD | 57653-85-7 |
| 1,2,3,4,7,8-HxCDD | 39227-28-6 |
| 1,2,3,7,8,9-HxCDD | 19408-74-3 |
| Total HxCDD | 34465-46-8 |
| 1,2,3,6,7,8-HxCDF | 57117-44-9 |
| 1,2,3,4,7,8-HxCDF | 70648-26-9 |
| 1,2,3,7,8,9-HxCDF | 72918-21-9 |
| 2,3,4,6,7,8-HxCDF | 60851-34-5 |
| Total HxCDF | 55684-94-1 |
| 1,2,3,4,6,7,8-HpCDD | 35822-39-4 |
| Total HpCDD | 37871-00-4 |
| 1,2,3,4,6,7,8-HpCDF | 67562-39-4 |
| 1,2,3,4,7,8,9-HpCDF | 55673-89-7 |
| Total HpCDF | 38998-75-3 |
| Total OCDD | 3268-87-9 |
| Total OCDF | 39001-02-0 |

CAS = Chemical Abstract Service

**Table 9-6. Target Metal Analytes**

| Metal | CAS Number |
|---|---|
| Antimony | 7440-36-0 |
| Arsenic | 7440-38-2 |
| Barium | 7440-39-3 |
| Beryllium | 7440-41-7 |
| Cadmium | 7440-43-9 |
| Chromium | 7440-47-3 |
| Cobalt | 7440-48-4 |
| Lead | 7439-92-1 |
| Manganese | 7439-96-5 |
| Mercury | 7439-97-6 |
| Nickel | 7440-02-0 |
| Selenium | 7782-49-2 |
| Silver | 7440-22-4 |
| Thallium | 7440-28-0 |
| Zinc | 7440-66-6 |

CAS = Chemical Abstract Service

## 10.0 SPECIFIC INTERNAL QUALITY CONTROL CHECKS

### 10.1 DEFINITIONS

The various types of QA/QC checks that may be performed as part of the SPT for both sampling and analysis are defined below. One or more of these QA/QC checks are associated with each measurement system in order to assess the compliance of the data to the DQOs established in Section 5.0. Table 10-1 summarizes all the sample analyses and their associated internal QC checks associated with this SPT.

**Audit Sample**—A field or alternate laboratory-prepared blank spike submitted to the analytical laboratory to assess accuracy or potential sample degradation.

**Blank, Field**—A sampling train or sampling component that is set up in the field but is not used for test sampling. The field blank is used to assess background contamination that may affect the representativeness of the field samples.

**Blank, Media**—A sample of unused sampling media analyzed to ensure the media are uncontaminated. This type of sample may also be referred to as a "reagent blank" (see below).

**Blank, Method**—A sample of unused media that is prepared and analyzed in the analytical laboratory to assess background contamination that may exist in the laboratory, on glassware, or in the analytical system.

**Blank, Reagent**—A sample of unused reagent(s) used to demonstrate the absence of contamination in the reagents.

**Blank, Spike**—A laboratory-prepared sample of blank media that is spiked with a known amount of target analyte(s) used to assess the accuracy of the analytical method.

**Blank, System**—An aliquot of uncontaminated reagent used to clean out the analytical system after high level samples have been analyzed or before analysis begins.

**Blank, Trip**—An unused sample component that is shipped to the field with the sampling equipment and media and/or returned to the laboratory without having been exposed to field conditions. If contamination is encountered in the field blank(s), the trip blank is analyzed to assess whether or not the contamination originates in the field, is inherent in the equipment or media, or results from exposure during shipping and handling.

**Breakthrough Check**—The result of the analysis of a secondary component (i.e., sorbent tube) in a sampling train is compared to the result of the primary component to assess whether or not the primary component has successfully captured the target analytes. If the result of the secondary component analysis is high compared to the primary component analysis, it is possible that the analytical results may be artificially low.

**Calibration Check**—A standard solution from a source other than the calibration standards used to verify the integrity of an instrument's calibration.

**Calibration Standards**—High purity compounds or mixtures of compounds used to adjust the response of an analytical instrument. The laboratory will use traceable standards and submit standard preparation logs as part of the deliverables package.

**Contingency Sample**—An archived portion of a field sample from the same location as other field samples that is collected and held in case of breakage or QA/QC failure during

the handling or analysis of the primary sample. This type of sample is sometimes referred to as an "archive sample."

**Continuing Calibration Verification**—A mid-point standard from the same calibration source as the initial calibration solution analyzed periodically to verify that calibration conditions have not drifted from the initial calibration.

**Duplicate Analysis**—A sample that is split in the laboratory and prepared and analyzed twice. The results of the two analyses are compared as a measure of precision.

**Duplicate Injection**—A second analysis of a single sample preparation. This QC test may be used to assess analytical QC failures, matrix interferences, or as a measure of analytical system precision.

**Initial Calibration**—A series of analyses of solutions, that have known concentrations, used to establish the correspondence between the amount of an analyte present in the solution and the instrument's response across the expected analytical range of the samples. Initial calibrations also establish retention time windows for identification purposes in chromatographic methods.

**Internal Standard Recovery**—Internal standards are nontarget spikes added to samples for quantitation purposes. The percent recovery of the internal standards is checked to assess whether or not significant matrix interferences may affect the accuracy and precision of analytical results.

**Performance Evaluation (PE) Sample**—See Audit Sample.

**Proficiency Test**—A series of blank spikes analyzed in the test laboratory to demonstrate an analyst's ability to successfully perform the method with acceptable precision and accuracy.

**Replicate**—One of a series of identical samples or splits of a single sample used to assess precision.

**Serial Dilution**—The result of the analysis of a highly contaminated sample, run undiluted, is compared to the results for the same sample after serial dilution. The two results are expected to match to within method-specified criteria. This test is a measure of the linearity of ICP calibration and the analysis technique.

**Spike, Field**—See Audit Sample.

**Spike, Matrix**—Spike of the known or controlled amount of an actual target analyte to an actual sample matrix that is then analyzed for that analyte. The percent recovery of the spiked analyte provides a measure of the matrix bias.

**Surrogate**—Nontarget or isotopically labeled analyte spiked into field samples as a measure of method efficiency and accuracy.

### 10.2 SPECIFIC QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

A variety of QC checks are required both in the field and in the laboratory to ensure collected samples accurately represent the field conditions under study, to assess compliance with the DQOs, and to assess biases in the measurement system.

#### 10.2.1 Field Activities

To ensure the representativeness of samples collected during the SPT and to ensure integrity of field measurements, a variety of QC checks and controls will be implemented throughout the sampling program. The following checks and controls will be implemented:

- Standard forms or standard field notebooks or both will be used to document field activities and data collection. The data collection forms and field notebooks will be reviewed routinely by senior staff for accuracy, completeness, and internal consistency.

- The strict adherence to detailed operating procedures as documented in the various project controlling documents and related SOPs will be enforced by experienced senior technical staff.

- Project personnel will be selected based on appropriate levels of training and experience and will receive site-specific training prior to working onsite. The site-specific training will include health and safety requirements; security requirements; briefings on overall project goals, objectives, and schedules; and specific technical training related to their assigned tasks.

- Routine calibration will be performed on measurement systems and sampling equipment, including metering systems, thermocouples, barometers, rotameters, and pitot tubes. Guidance related to equipment calibration is provided in *Quality Assurance Handbook for Air Pollution Measurement Systems*, Volume III, "Stationary Source Specific Methods and Quality Assurance/Quality Control Procedures for Hazardous Waste Incinerators," Appendix A. The detailed specifications, acceptance criteria, and corrective action requirements are presented in Section 8.0 of this QAPjP. All calibrations will be documented and the documentation maintained in the project files.

- Leak checks will be performed according to method specifications before and after sampling.

- Field QC samples will be routinely submitted, including audit (PE) samples, field blanks, media blanks, reagent blanks, trip blanks, and contingency samples. The frequency of submittal for these field QC samples and other field samples are provided in Tables 5-1 and 8-2.

- Field audits and surveillances will be performed periodically by the QAO to assess conformance to specifications. If nonconforming conditions are noted, the corrective action provisions of the QA plan will be invoked.

#### 10.2.2 Laboratory Activities

Standard laboratory QA procedures, required of each laboratory, provide discussions related to QA/QC checks and controls within the laboratory. Specific DQOs, calibration requirements, acceptance criteria, and corrective action requirements for this SPT are presented in Table 5-1 and Table 8-2 of this QAPjP.

In addition to the previously referenced requirements, the laboratory will provide for QC of sampling media and sample collection equipment. Sorbents used in the organic sampling trains will be prepared according to method specifications. Samples of the prepared media will be tested according to the intended method of use. The results of these tests will be retained in the laboratory's files for future reference.

**Table 10-1. Summary of System Performance Test Analyses**

| Analysis | Sample Matrix | Field Samples[a] | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency [(a)] | QC Analyses | Total Analyses[(b)] |
|---|---|---|---|---|---|---|---|---|---|
| | | Test | Field QC | | | | | | |
| Metals by ICP | Analytical system QC | NA | NA | Digestion (SW-846 Method 3010A) | ICP (SW-846 Method 6020) | LCS | One per batch/ matrix specific | 2 or more | 6 |
| | | | | | | Serial dilution | One per batch/ matrix specific | 2 or more | |
| | | | | | | Method blank | One per batch/ matrix specific | 2 or more | |
| Mercury by CVAA | Analytical system QC | NA | NA | CVAA (SW-846 Method 7470A) | CVAA (SW-846 Method 7470A) | LCS | One per batch matrix specific | 2 or more | 4 |
| | | | | | | Method blank | One per batch/ matrix specific | 2 or more | |
| Target VOCs and TICs by SW-846 Method 0031 (SMVOC) | SMVOC resin tubes | 16 | — | NA (Note: Only 3 of the 4 tube sets from each test run will be analyzed; the fourth set is a back-up in case of tube breakage.) | Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); each tube in each tube pair is analyzed separately | Surrogate spikes | Every analysis[(c)] | 24 | 24 |
| | SMVOC condensate | 8 | — | NA | Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); each tube in each tube pair is analyzed separately | Surrogate spikes | Every analysis[(c)] | 16 | 16 |
| | SMVOC field blank resin tubes | — | 8 | NA | Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); tube pair is analyzed as a single sample | Surrogate spikes | Every analysis[(c)] | 16 | 16 |
| | SVOC trip blank resin tubes | — | 1 | NA | Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); tube pair is analyzed as a single sample | Surrogate spikes | Every analysis[(c)] | 2 | 2 |
| Target VOCs and TICs by SW-846 Method 0031 (SMVOC) (cont) | SMVOC condensate trip/field blank | — | 2 | NA | Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); each tube in each tube pair | Surrogate spikes | Every analysis[(c)] | 4 | 4 |

| Analysis | Sample Matrix | Field Samples[a] | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency [a] | QC Analyses | Total Analyses[b] |
|---|---|---|---|---|---|---|---|---|---|
| | | Test | Field QC | | | | | | |
| | | | | | is analyzed separately | | | | |
| | SMVOC audit resin tubes (if provided) | — | 4 | NA | Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); each tube in each the pair is analyzed separately | Surrogate spikes | Every analysis[c] | 8 | 8 |
| | Spiked resin blank tube pairs | — | 2 | NA | Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); tube pair is analyzed as a single sample | Surrogate spikes | Every analysis[c] | 4 | 4 |
| | Analytical system QC | NA | NA | NA | Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); each tube in each tube pair is analyzed separately | LCS | 1 per condensate | 1 or more | 1 |
| | | | | | | Method blank | 1 per analytical run | 2 or more | 2 |
| PCDD/PCDFs by EPA Method 0023A | Method 0023A filter, XAD-2 resin, and front half and back half solvent rinses | 8 | 2-blank train | Soxhlet extraction (EPA Method 0023A) | HRGC/HRMS for PCDD/PCDFs (SW-846 Method 8290) | PCDD/PCDF pre-sampling surrogate spikes | Every XAD-2 resin tube before sampling | 8 | 8 |
| | | | | | | PCDD/PCDF isotope dilution internal standard spike | Every analysis[c] | 8 | |
| | | | | | | PCDD/PCDF recovery standard spike | Every analysis[c] | 8 | |
| | Method 0023A acetone reagent blank | — | 1 | NA | HRGC/HRMS for PCDD/PCDFs (SW-846 Method 8290) | PCDD/PCDF isotope dilution internal standard spike | Every analysis[c] | 1 | 1 |
| | | | | | | PCDD/PCDF recovery standard spike | Every sample | 1 | |
| | Method 0023A methylene chloride reagent blank | — | 1 | NA | HRGC/HRMS for PCDD/PDDRs (SW-846 Method 8290) | PCDD/PCDF isotope dilution internal standard spike | Every analysis[c] | 1 | 1 |

| Analysis | Sample Matrix | Field Samples[a] | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency [a] | QC Analyses | Total Analyses[b] |
|---|---|---|---|---|---|---|---|---|---|
| | | Test | Field QC | | | | | | |
| PCDD/PCDFs by EPA Method 0023A (cont) | | | | | | PCDD/PCDF recovery standard spike | Every analysis[c] | 1 | |
| | Method 0023A toluene reagent blank | — | 1 | NA | HRGC/HRMS for PCDD/PDDRs (SW-846 Method 8290) | PCDD/PCDF isotope dilution internal standard spike | Every analysis[c] | 1 | 1 |
| | | | | | | PCDD/PCDF recovery standard spike | Every analysis[c] | 1 | |
| | Method 0023A spiked XAD-2 resin blank | — | 2 | Soxhlet extraction (SW-846 Method 0023A) | HRGC/HRMS for PCDD/PDDRs (SW-846 Method 8290) | PCDD/PCDF pre-sampling surrogate spikes | Every XAD-2 resin tube before sampling | 2 | 2 |
| | | | | | | PCDD/PCDF isotope dilution internal standard spike | Every analysis[c] | 2 | |
| | | | | | | PCDD/PCDF recovery standard spike | Every analysis[c] | 2 | |
| | Method 0023A spiked XAD-2 resin | — | 1 | Soxhlet extraction (EPA Method 0023A) | HRGC/HRMS for PCDD/PDDRs (SW-846 Method 8290) | PCDD/PCDF pre-sample surrogate spikes | Every XAD-2 resin | 1 | 1 |
| | | | | | | PCDD/PCDF isotope dilution internal standard spike | Every analysis[c] | 1 | |
| | | | | | | PCDD/PCDF recovery standard spike | Every analysis[c] | 1 | |
| | Analytical system QC | NA | | Soxhlet extraction (EPA Method 0023A) | HRGC/HRMS for PCDD/PDDFs (SW-846 Method 8290) | Method blank | 1 per analytical batch | 1 or more | 3 |
| | | | | | | Blank spike | 2 per analytical batch | 2 | 6 |
| SVOCs & TICs and PAH SW-846 | Method 0010 front half | 8 | 2-blank train | Soxhlet extraction with MeC12 (SW-846 | GC/MS (SW-846 Method 8270B) | Semivolatile surrogate spikes | Every sample | 10 | 10 |

| Analysis | Sample Matrix | Field Samples[a] | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency [(a)] | QC Analyses | Total Analyses[(b)] |
|---|---|---|---|---|---|---|---|---|---|
| | | Test | Field QC | | | | | | |
| Method 0010 | composite; filter and front half solvent rinses | | | Methods 0010, 3542, 8270B) | | Semivolate internal standard surrogate spikes | Every sample | 10 | |
| | Method 0010 back half composite; XAD-2 and back half solvent rinses | 8 | 2-blank train | Soxhlet extraction with MeC12 (SW-846 Methods 0010, 3542, 8270B) | GC/MS (SW-846 Method 8270B) | Semivolatile surrogate spikes | Every sample | 10 | 10 |
| | | | | | | Semivolatile internal standard surrogate spikes | Every sample | 10 | |
| | | | | | | $^{13}$C-labeled pre-sampling surrogate spikes | Every XAD-2 resin before sampling | 10 | |
| | Condensate impinger | 8 | 2-blank train | Separatory funnel acid/base liquid-liquid extraction (SW-846 Methods 0010, 3542, 8270B) | | Semivolatile surrogate spikes | Every sample | 10 | 10 |
| | | | | | | Semivolatile internal standard surrogate spikes | Every sample | 10 | |
| | Method 0010 methylene chloride reagent blank | — | 1 | NA | GC/MS (SW-846 Method 8270B) | Semivolatile surrogate spikes | Every sample | 1 | 1 |
| | | | | | | Semivolatile internal standard surrogate spikes | Every sample | 1 | |
| | Method 0010 acetone reagent blank | — | 1 | NA | GC/MS (SW-846 Method 8270B) | Semivolatile surrogate spikes | Every sample | 1 | 1 |
| | | | | | | Semivolatile internal standard surrogate spikes | Every sample | 1 | |
| | Analytical system QC | NA | | Soxhlet extraction with MeCl2 (SW-846 Methods 0010, 3542, 8270B) | GC/MS (SW-846 Method 8270B | Method blank | 1 per analytical batch | 1 or more | 3 |
| | | | | | | Blank spike | 2 per analytical batch | 2 | 6 |
| Metals by Method 29 | Method 29 front half: filter, and nitric acid probe and front half filter holder rinses | 8 | 2 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | PDS[(d)] | One per test | 2 | 12 |

| Analysis | Sample Matrix | Field Samples[a] | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency [(a)] | QC Analyses | Total Analyses[(b)] |
|---|---|---|---|---|---|---|---|---|---|
| | | Test | Field QC | | | | | | |
| | Method 29 10% $HNO_3$/5% $H_2O_2$ impinger contents and rinses | 8 | 2 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | PDS[(d)] | One per test | 2 | 12 |
| Metals by Method 29 (cont) | Method 29 $KMnO_4$/5% $H_2SO_4$ impinger content and rinses | 8 | 2 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | PDS[(d)] | One per test | 2 | 12 |
| | Method 29 HCl rinse | 8 | 2 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | PDS[(d)] | One per test | 2 | 12 |
| | Method 29 filter reagent blank | — | 1 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | Reagent blank | One for test program | 1 | 1 |
| | Method 29 $HNO_3$ reagent blank | — | 1 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | Reagent blank | One for test program | 1 | 1 |
| | Method 29 10% $HNO_3$/5% $H_2O_2$ reagent blank | — | 1 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | Reagent blank | One for test program | 1 | 1 |
| | Method 29 $KMnO_4$/$H_2SO_4$ reagent blank | — | 1 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | Reagent blank | One for test program | 1 | 1 |
| | Method 20 HCl reagent blank | — | 1 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | Reagent blank | One for test program | 1 | 1 |
| | Spike filter audit sample for metals | — | 1 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 | Audit sample | One for test program | 1 | 1 |

| Analysis | Sample Matrix | Field Samples[a] | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency [a] | QC Analyses | Total Analyses[b] |
|---|---|---|---|---|---|---|---|---|---|
| | | Test | Field QC | | | | | | |
| | | | | | Method 7470) for Hg | | | | |
| | Liquid audit sample for metals | — | 1 | Method 29 | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | Audit sample | One for test program | 1 | 1 |
| Metals by Method 29 (cont) | Analytical system QC | NA | NA | NA | ICP (SW-846 Method 6010B or 6020A) CVAAS (SW-846 Method 7470) for Hg | LCS | One per batch/ matrix specific | 1 or more | 1 |
| | | | | | | Serial dilution | One per batch/ matrix specific | 1 or more | 1 |
| | | | | | | Method blank | One per batch/ matrix specific | 1 or more | 1 |
| Particulate by Method 26A | Method 26A particulate filter | 8 | — | Dessiccate to constant mass | Gravimetric (Method 5) | Replicate weighing to constant weight | Every sample | 8 | 8 |
| | Method 26A probe and filter holder acetone rinses | 8 | — | Evaporate/desiccate to constant mass | Gravimetric (Method 5) | Replicate weighing to constant weight | Every sample | 8 | 8 |
| | Method 26A filter reagent blank | — | 1 | Dessiccate to constant mass | Gravimetric (Method 5) | Replicate weighing to constant weight | Every sample | 1 | 1 |
| | Method 26A acetone reagent blank | — | 1 | Evaporate/desiccate to constant mass | Gravimetric (Method 5) | Replicate weighing to constant weight | Every sample | 1 | 1 |
| HCl by Method 26A | Method 26A $H_2SO_4$ impingers | 8 | — | NA | Ion chromatrography (EPA Method 26A) | Duplicate | One per test | 1 | 11 |
| | | | | | | MS/MSD analyzed in duplicate[d] | 1 per batch (assuming all samples batched together) | 2 | |
| | Method 26A $H_2SO_4$ reagent blank | — | 1 | NA | Ion chromatrography (EPA Method 26A) | Reagent blank | One for test program | 1 | 1 |
| Cl2 by Method 26A | Method 26A NaOH impingers | 8 | — | NA | Ion chromatrography (EPA Method 26A) | Duplicate | One per test | 1 | 11 |

| Analysis | Sample Matrix | Field Samples[a] | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency [(a)] | QC Analyses | Total Analyses[(b)] |
|---|---|---|---|---|---|---|---|---|---|
| | | Test | Field QC | | | | | | |
| | | | | | | MS/MSD analyzed in duplicate[d] | 1 per batch (assuming all samples batched together) | 2 | |
| | Method 26A NaOH reagent blank | — | 1 | NA | Ion chromatrography (EPA Method 26A) | Reagent blank | One for test program | 1 | 1 |
| Cl ion by Method 26A | Method 26A deionized water reagent | — | 1 | NA | Ion chromatrography (EPA Method 26A) | Reagent blank | One for test program | 1 | 1 |
| Cl ion chromatography | Liquid audit sample | — | 2 | NA | Ion chromatrography (EPA Method 26A) | Duplicate | One for test program | 2 | 4 |
| **TOTAL** | | | | | | | | | **29** |

Notes:
[a] The test condition is comprised of three replicate sampling runs.
[b] Total laboratory analyses includes field sample analyses and laboratory QC analyses.

[d] MS = Matrix and
MSD = Matrix spike duplicate
PDS = Post-digestion and
[e] Refer to Table 5-1 notes for the matrix spike compounds

| | |
|---|---|
| CVAA | cold vapor atomic absorption |
| EPA Method | *New Source performance Standards*, Test Methods and Procedures, Appendix A 40 CFR 60 |
| GC | gas chromatography |
| HR | high resolution |
| IC | ion chromatography |
| ICP | inductively coupled argon plasma spectroscopy |
| MS | mass spectroscopy |
| NA | not applicable |
| PAH | polyaromatic hydrocarbon |
| PCDD/PCDF | polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans |
| QC | quality control |
| SVOC | semivolatile organic compound |
| SW846 | *Test Methods for Evaluating Solid Waste*, Third Edition, November 1986, and Updates |
| TIC | tentatively identified compound |
| VOC | volatile organic compound |

# 11.0 DATA REDUCTION, DATA VALIDATION, AND DATA REPORTING

## 11.1 DATA REDUCTION

This section of the QAPjP describes the approach that will be used to report, review, and reduce the field and laboratory data. The raw data include field samples and sampling documentation, sample tracking documentation, laboratory preparation documentation, and raw analytical data. The analytical results from the laboratory are assembled into complete analytical data packages. The analytical data packages include the analytical results and their defensible backup data. The analytical data are evaluated for compliance with the project DQOs. Data determined to meet the analytical requirements will be used to calculate the unit performance and emissions.

## 11.2 ORGANIC ANALYSES

Organic analyses will be conducted using gas chromatography techniques. Although the principles of operation and specific methods of calibration differ according to the analyte-specific methods, the general data reduction scheme is the same for all these tests. The individual methods should be consulted for details.

Depending on the specific method, analytical instrumentation is calibrated using 3 to 5 points covering the expected analytical range. The GC/MS methods use an internal standard technique. For GC/MS calibrations, a response factor (RF) is calculated using the following formula:

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}$$

Where

$RF$ = response factor

$A_s$ = response for the analyte

$A_{is}$ = response for the internal standard

$C_s$ = concentration of the analyte

$C_{is}$ = concentration of the internal standard.

The response factors must agree to within method-specified criteria for the percent relative standard deviation (%RSD). The formula for %RSD follows:

$$\%\,\mathrm{RSD} = \frac{\sigma}{\mathrm{avg\ RF}} \times 100$$

Where

$\%RSD$ = percent relative standard deviation

$\sigma$ = standard deviation of the calibration factors

$avg\ RF$ = average response factor.

When samples are analyzed, the area of the peak produced by a given analyte is compared to the RF to arrive at an analytical result according to the following formula:

$$M_x = A_x \times RF$$

Where

$M_x$ = mass of analyte in the sample

$A_x$ = response of the analyte in the sample

$RF$ =response factor.

Samples containing a concentration of an analyte exceeding the concentration the instrument is calibrated for will be diluted, if such a dilution is practical given the sample preparation method. In that case, $M_x$ is multiplied by the dilution factor to arrive at the final result. If, under the circumstances of the method, a dilution is not possible, the analytical result must be considered estimated.

To arrive at a concentration in the gas sample, the mass of any subsamples must be added together and then compared to the volume of gas sampled according to the following formula:

$$C_x = \frac{(M_1 + M_2 + .. M_n)}{V}$$

Where

$Cx$ = concentration of the analyte in the gas sample

$Mn$ = result (mass) for each component in the sampling train

$V$ = volume of gas sampled.

### 11.3 METALS ANALYSES

The analysis of metals also begins with an instrument calibration composed of 2 to 6 points, depending on the specific analytical method. For ICP for nonmercury (non-Hg) metals analyses and cold vapor atomic adsorption spectroscopy for mercury (Hg) analyses, instruments are profiled and calibrated according to the instrument manufacturer's instructions. A calibration blank and a QC check standard then are analyzed to ensure appropriate instrument response. A percent recovery (%R) is calculated according to the following formula:

Where

$$\%R = \frac{Found}{True} \times 100$$

$\%R$ = percent recovery

$Found$ = result of the analysis

$True$ = expected result.

The %R is expected to be within method specifications before analysis can begin. The calibration is verified periodically according to method specifications using the same technique.

Atomic absorption instruments are calibrated using 3 to 5 points. A linear regression is performed, and a correlation coefficient is calculated to assess linearity of the curve. It is beyond the scope of this document to provide a detailed explanation of the statistics supporting linear regression and the calculation of the correlation coefficient. Any standard statistics textbook can be referenced for additional information. Calibration checks are performed as above and periodically verified.

Analytical results for metals are read directly from the instrument in terms of concentration. Dilution factors must be used as discussed previously if applicable. To combine the results of the subsamples of a metals sampling train, the concentration is converted back to mass using the following formula:

$$M_{xs} = C_{xs} \times V_{xs}$$

Where

$M_{xs}$ = mass in the subsample

$C_{xs}$ = concentration in the subsample

$V_{xs}$ = volume of the subsample.

The concentration in the gas sample then can be calculated.

### 11.4 ION CHROMATOGRAPHY

Anions, such as chloride, are separated on the ion chromatograph using a system comprising separator columns, guard columns, and eluents. The system is calibrated using a minimum of 3 points, and the calibration is verified with a mid-range standard.

### 11.5 DIRECT READING INSTRUMENTS

Gravimetric, temperature, pressure, flow, and CEMS data are directly read from the measurement instrumentation. The instrumentation will be calibration-checked prior to the test and routinely prior to reading measurements; however, no data reduction beyond formatting into tables is expected

### 11.6 ANALYTICAL DATA PACKAGES

Analytical data packages will be organized in accordance with the laboratory SOPs. The complete analytical data package is a stand-alone deliverable that includes the final analysis results, raw analytical instrument data, initial and continuing calibration data, parameter-specific QC documentation, sample preparation documentation, and records of sample traceability. These data are sufficient for performing independent verification of the final analytical results. Every analytical data package includes the following:

Cover Page—Identifies the laboratory-assigned lot number, project identification, laboratory project manager, and issue date.

Table of Contents—Lists organization of the data package.

Sample Summary—Cross references project sample identifications and laboratory sample identifications.

Analytical/Preparation Methods Summary—Identifies the methods used to prepare and analyze the samples.

Narrative—Summarizes the project-specific information and any pertinent information concerning data quality. The narrative documents sample delivery and condition upon receipt, and any analytical difficulties or anomalies encountered during sample preparation and analysis.

QC Data Association Summary—Compares sample results to the project and laboratory DQOs and association of project samples to laboratory QC.

Analytical Data Report—Summarizes analytical data for all samples and associated QA samples, including as appropriate: data flags, duplicate analysis results, surrogate recovery results, method blank results, laboratory control sample results, and matrix spike/matrix spike duplicate/post-digestion spike (MS/MSD/PDS) results.

COC documentation.

Each analytical data package will include the identification and signature or initials of each analyst who handles the test samples. The laboratory analysis coordinator will certify by signature the contents of each analytical data package.

### 11.6.1 Organic Analyses

The organic analytical data packages will include the following:

Sample Results

Raw Sample Data

Sample Data

Tentatively Identified Compound Data for Compounds by GC/MS (if applicable)

Standards Data

Initial Calibration Summary(s) and Raw Data

Continuing Calibration Summary(s) and Raw Data

Initial Calibration(s) Tuning Raw Data (GC/MS only)

Continuing Calibration(s) Tuning Raw Data (GC/MS only)

Raw QC Data

Method Blank Data

Tentatively Identified Compound Data for Compounds by GC/MS (if applicable)

MS/MSD Data and Evaluation Reports

Laboratory Control Standard Data and Evaluation Reports

Miscellaneous Data

Sample Data Review Checklist

Calibration Data Review Checklists

Run Logs

Extraction Sheets

Sample Results

Example Calculations.

### 11.6.2 Metals Analyses

The metals analytical data packages will include the following:

Sample Results

Quality Control Reports

Method Blank Report

Sample Duplicate Report

MS/MSD/PDS Data and Evaluation Report

Laboratory Control Sample Data and Evaluation Report

Quality Control Summaries

Initial Calibration Verification and Blank Results

Continuing Calibration Verification and Blank Results

Interference Check Sample

PDS Sample Recovery

Serial Dilutions Documentation for ICP

Instrument Detection Limits

ICP Correction Factors

ICP Linear Ranges

Raw Data

Data Review Checklist

Sample, Standards, and Quality Control Data

Digestion/Extraction Sheets

Preparation Log

Analysis Run Log.

### 11.6.3 Other Inorganic Analyses

The data packages for other inorganic analyses will include the following:

Sample Results

Quality Control Summary

Method Blank Report

Sample Duplicate report

MS/MSD Data and Evaluation Report(s)

Laboratory Control Sample Data and Evaluation Report

Raw Data

Data Review Checklist

Sample, Standards, and Quality Control Data

Distillation, Extraction, and Sample Preparation Sheets

Standards Preparation Logs

Run Logs.

## 11.7 DATA VALIDATION

The results of all sample analysis and all QA/QC sample analysis (100% of the laboratory data) will be compared to the specifications in Tables 5-1 and 8-2. The following data validation criteria prepared by the U.S. EPA Data Review Work Group will be followed as applicable to the individual methods used:

*Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review*, EPA, 2001

*Analytical Operations/Data Quality Center National Functional Guidelines for Chlorinated Dioxin/Furan Review*, EPA, 2002

*Contract Laboratory National Functional Guidelines for Inorganic Data Review*, EPA, 2004.

Any sample data associated with a QC check that fails to meet the target criteria established in these tables will be flagged in the final report, and an assessment of the impact, if any, of missing the target DQO will be provided. Additional guidance is in the analytical methods and *Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration* (EPA/625/6-89/023).

The MDL and the RDL or laboratory-specified RL will be reported for all nonisotope dilution method compounds. Results reported between the MDL and the RDL or RL will be flagged as estimated. Non-

detects for the isotope dilution methods will be determined using the SW-846 Method 8290 definition of an EDL without the use of empirical factors or other mathematical manipulations specific to the laboratory. The laboratory must include with each data package any calculations or statistical methods sample dilution corrections. The laboratory must maintain on file documentation for the reported detection limits.

Particular attention will be paid to the results of blank data. Analytical data will not be routinely corrected for contamination. They will be, however, evaluated on a case-by-case basis for possible blank correction with prior approval of DEQ. Samples associated with contaminated blanks will be flagged so that this information may be assessed in the final report.

The output from the data validation process will be a summary comparison of the QA/QC results to the specified DQOs, a review and discussion of any deficiencies identified in the data assessments of laboratory performance, and overall precision, accuracy, representativeness, and completeness of the data set.

Detailed procedures for the internal laboratory review of data are in the laboratory's QA manuals and related SOPs.

## 11.8 DATA REPORTING

### 11.8.1 Project Reporting Format

The outline for the final SPT report is presented in Figure 11-1.

**Figure 11-1. Example System Performance Test Report Outline**

- EXECUTIVE SUMMARY
- SYSTEM PERFORMANCE TEST SUMMARY
  - Engineering Description
    - General Description
    - Waste Feed Systems
    - Solid Product Handling System
    - Off-gas Treatment System
    - Process Continuous Monitoring System
    - Continuous Emissions Monitoring System
    - Automatic Waste Feed Cutoff System
  - Summary of Test Plan and Objectives
  - Test Implementation Summary
    - Deviations from the Test Plan
- PROCESS OPERATIONS
  - Process Operating Conditions
  - Feed Material Characteristics
  - Effluent Characteristics
- COMPLIANCE RESULTS
  - Organic Emissions
  - Particulate Emissions
  - Hydrogen Chloride and Chlorine Emissions
  - Metals Emissions
  - Process Off-gas Carbon Monoxide and Total Hydrocarbons
  - Process Off-gas Nitrogen Oxides
  - Dioxin and Furan Emissions
  - CEMS Performance Evaluation Results
- QUALITY ASSURANCE/QUALITY CONTROL RESULTS
  - QA/QC Activities and Implementation
    - QA Surveillance
    - Sample Collection
    - Sample Analysis
    - Process Instrumentation
    - Emissions Sampling Equipment
    - Laboratory Analytical Instrumentation
  - Audits and Data Validation
  - Calculations
  - Conclusions
- RISK EVALUATION
- PROPOSED PERMIT OPERATING CONDITIONS
  - Development of Operating Limits
  - Specific Control Parameters
- APPENDICES
  - A. Process Operating Data
  - B. Test Manager's Log
  - C. Process Instrument Calibration Data
  - D. Continuous Emissions Monitoring Data
  - F. Sampling Report
  - G. List of Samples
  - H. Analytical Report
  - I. Calculations
  - J. Documentation to Support Metals Extrapolation
  - K. Data Validation Report
  - L. Corrective Action Requests
  - M. CEMS Performance Evaluation Report

### 11.8.2 Detection Limit Definitions

The detection limit for each project target analyte will be derived according to the referenced analytical method and the laboratory's SOPs. The laboratory will provide detection limits for each analyte. A number of types of detection limits are associated nonisotope dilution analytical methods. The *Human Health Risk Assessment Protocol* (EPA, Office of Solid Waste, July 1998) defines the following detection limits:

**Instrument Detection Limit**—The smallest signal above background that an instrument can reliably detect, but not quantify. Also, commonly described as a function of the signal-to-noise ratio.

**Method Detection Limit (MDL)**—The minimum concentration of a substance that can be measured (using nonisotope dilution methods) and reported with 99% confidence that the analyte concentration is greater than zero, and is determined from analysis of a sample in a specific matrix type containing the analyte. The MDL is considered the lowest level at which a compound can be reliably detected. The MDL is based on statistical analyses of laboratory data. In practice, the MDLs are determined on analytical reagents (e.g., water) and not on the matrix of concern. MDLs for a given method are laboratory and compound specific. To determine the MDL as specified in 40 CFR Part 136, Appendix A, at least seven replicate samples with a concentration of the compound of interest near the estimated MDL are analyzed. The standard deviation among these analyses is calculated and multiplied by 3.14. The result of the calculation becomes the MDL. The factor of 3.14 is based on a t-test with six degrees of freedom and provides a 99% confidence that the analyte can be detected at this concentration.

**Reliable Detection Limit (RDL)**—The detection level recommended by the National Environmental Research Laboratory in Cincinnati, Ohio. The RDL is defined as 2.63 times the MDL. The RDL is a total of eight standard deviations above the MDL development test data (3.14 × 2.63).

**Reporting Limit (RL)**—The laboratory-determined detection, normally the lowest calibration standard.

The risk assessment protocol recommends that nonisotope dilution methods quantify non-detects using the method MDL and the derived RDL.

Component 2 of the EPA Region 6 *Hazardous Waste Combustion Unit Permitting Manual* uses the term SQL. The SQL is the matrix-specific MDL. The SQL may be equal to the laboratory-specific MDL or some factor greater than (e.g., 5–10 times) the laboratory-specific MDL that takes into consideration matrix-specific interferences or sample serial dilutions.

Isotope dilution methods quantify non-detects using EDLs as defined in SW-846 Method 8290, as follows:

> EDL - "The sample specific estimated concentration of a given analyte required to produce a signal with a peak height of 2.5 times the background signal level."

The laboratory will provide the MDL or SQL for each nonisotope dilution analyte on the analytical certificate. The laboratory will maintain documentation and validation for the reported MDL. If matrix interference(s) occurs or serial dilutions are necessary, the sample-specific MDL (SQL) will be reported. If a sample-specific MDL (SQL) is applicable, the analytical data package shall include the documentation of the serial dilutions or other measures taken to determine the detection limit. EDLs are sample specific and analyte specific, and will be provided on the analytical certificate for each sample and each analyte.

### 11.8.3 Detection Limits and Data Reduction

Analytical results for all analyses, except Method 0023A samples for PCDD/PCDFs, that are reported as non-detect in compliance demonstration samples will be handled in the following manner. The analytical result will be reported as non-detect, and the appropriate detection limit will be shown. In subsequent calculations, the following rules will apply for data reporting and performance demonstrations:

- For analyses of single-component samples (e.g., feed or residue samples) that are reported as non-detect, the values will be accompanied by a "less than" ("<") and/or a data flag that denotes a non-detect result was obtained

- For analyses of single-component samples (e.g., feed or residue samples) that are reported as non-detect, any subsequent calculations using detection limit values will be accompanied by "<".

- For analyses of multi-component samples (e.g., Method 0023A), where multiple analytical results must be combined or summed for use in subsequent calculations and the calculations involve the use of one or more non-detect values in combination with one or more detectable values, the final calculated values will also include "<".

- For analyses of multi-component samples where all of the multiple analytical results are non-detect values, the final calculated values will include a data flag such as "ND" or "U" with the "<" to indicate that the target analyte was non-detect in all of the relevant sample fractions.

- Where destruction and removal efficiency, system removal efficiency, or similar performance measurements are calculated using emissions rates reported with "<" or non-detect-flagged values, the resulting performance measurement will be accompanied by a "greater than" (">") sign.

For Method 0023A sampling train PCDD/PCDFs results, any dioxin/furan congener that is non-detect will be counted as zero in calculating and reporting the 2,3,7,8-tetrachlorodibenzo-p-dioxin toxicity equivalents, corrected to 7% oxygen. For the front-half and back-half separate analyses of the Method 0023A samples, if a fraction is non-detect for a specific congener, the specific congener's EDL will be reported. In subsequent compliance calculations, the congener concentration for a fraction that is reported as non-detect will be counted as zero for that fraction.

### 11.8.4 Other Quality Control Data Reporting

Other QC data that are included in the analytical reports and may be discussed or commented on in the final SPT reports are:

Sample holding times

Sample surrogate recovery results

Duplicate results

MS/MSD results

Blank train results

Reagent blank results (if analyzed)

Trip and field blank results

RPD and RSD evaluations of accuracy or precision.

These data will be evaluated against the target DQOs. Any data that fall outside of the DQO limits will be flagged, footnoted, and assessed relative to the performance and emissions testing objectives. The data from the field blanks and trip blanks will be used to assess possible contamination from field handling and transport of the samples to the laboratory. The reagent blank and blank train results will be used to assess contamination resulting from reagents used, sample handling, and sample recovery procedures. Any blank results that may have impacted performance and emissions testing results or evaluations will be flagged, footnoted, and discussed in the test reports.

#### 11.8.5 Final Case Files

At a minimum, the following documents will be retained upon completion of the SPT in the final case file, which must be maintained at the INTEC facility for at least three years:

- All legal documents and orders
- All field documents, including those used for preliminary field activities
- Copies of all analytical data
- Copies of the final report and background documents
- All correspondence relating to the project as well as corrective action requests.

## 12.0 ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES

### 12.1 SAMPLING EQUIPMENT

All equipment used in emission testing measuring systems must be maintained in good operating order. To achieve this objective, a routine preventive maintenance program is necessary. Procedures used in this SPT follow those of *Maintenance Calibration and Operation of Isokinetic Source Sampling Equipment* (Publication No. APTD-05-76) and Volume III of the *Quality Assurance Handbook for Air Pollution Measurement Systems*.

The potential impact of equipment malfunction on data completeness is minimized through two complementary approaches. First, an equipment maintenance program is part of routine operations. The maintenance program's strengths include:

- Trained technicians experienced in the details of equipment maintenance and fabrication
- Adequate spare parts inventory
- Availability of tools and specialized equipment.

The second approach is based upon equipment redundancy. Backup equipment, spare parts, and tools are included on the materials transported to the field for each sampling task. This approach allows the sampling team to respond to equipment breakage or malfunction in a timely fashion, minimizing the quantity of lost data.

For field equipment, preventive maintenance schedules are based on results of routine inspections and on accumulated experience. At a minimum, equipment will be inspected prior to the beginning of and at the conclusion of each test. A record of each inspection (Figure 12-1) will be kept as part of the final case file. Maintenance schedules for CEMs follow manufacturer's recommendations.

**Figure 12-1. Example Equipment Inspection Record Form**

**Idaho Nuclear Technology and Engineering Center**

Date/Time of Inspection: _____

Equipment Inspected:

1) _____

Condition: Good See Problems Section Below

2) _____

Condition: Good See Problems Section Below

3) _____

Condition: Good See Problems Section Below

4) _____

Condition: Good See Problems Section Below

5) _____

Condition: Good See Problems Section Below

Problems Noted:

_____

_____

_____

_____

Action Taken

_____

_____

_____

Inspector's Signature_____

An effective preventive maintenance program is necessary to ensure data quality. Each item of field test equipment is assigned a unique, permanent identification number. Each item of equipment returning from the field is inspected before it is returned to storage. During these inspections, items are cleaned, repaired, reconditioned and recalibrated, where necessary. Each item of equipment transported to the field for this SPT is inspected again before being packed to detect equipment problems that may originate during storage. This minimizes lost time on the job site due to equipment failure. Occasional equipment failure in the field is unavoidable despite the most rigorous inspection and maintenance procedures. For this reason, adequate spare parts are kept in a central location so the sampling contractor can quickly respond to the job site with replacement equipment for all critical sampling train components.

### 12.2 LABORATORY INSTRUMENTS

The laboratories perform regular maintenance on all analytical instruments. An inventory of replacement parts is kept to prevent downtime. Manufacturers' service representatives also are contracted, as required, for major instrument repairs.

Preventive and routine maintenance is covered in each of the laboratories' QA manuals and SOPs or in accordance with manufacturer's recommendations (i.e., instrument manuals). Daily maintenance (such as replacement of injector septa, etc.) is covered in instrument SOPs. Inoperative equipment is tagged as nonusable until repaired. Logbooks are maintained for each instrument to record usage, maintenance, and repairs.

### 12.3 PROCESS INSTRUMENTS

Onsite personnel perform regular maintenance on all process instrumentation. Routine and preventive maintenance programs are used. Where appropriate, manufacturers' recommendations for maintenance of process instruments are followed. Operators conduct daily reviews of process instrumentation by noting suspicious or inconsistent readings. Maintenance logs are used to record the frequency and type of repairs necessary for process instruments. Process instruments used to demonstrate compliance with operating limits will be calibrated prior to the test. Records of these calibrations will be included in the final SPT report.

## 13.0 ASSESSMENT PROCEDURES FOR ACCURACY, PRECISION, AND COMPLETENESS

The QA activities implemented in this study will provide a basis for assessing the accuracy and precision of the analytical measurements. Section 5.0 discusses the QA activities that will generate the accuracy and precision data for each sample type. The generalized forms of the equations that will be used to calculate accuracy and precision are presented in the following subsections.

### 13.1 ACCURACY

When a reference standard material is used in the analysis, percent accuracy (A) will be calculated as follows:

$$A = \frac{\text{Found concentration}}{\text{True concentration}} \times 100$$

Percent analyte recovery (R) will be calculated as follows:

$$R = \frac{X - N}{S} \times 100$$

Where

X = the experimentally determined value

N = the amount of native material in the sample

S = the amount of spiked material of the species being measured.

Recoveries are used to determine accuracy when standards are not available or are not appropriate for a given matrix.

### 13.2 PRECISION

When less than three analyses of the same parameter are available, precision will be calculated as an RPD from the average of replicate measurements according to:

$$\text{RPD} = \frac{(X_1 - X_2)}{\text{Average } X} \times 100$$

Where

$X_1$ and $X_2$ = the highest and lowest results of replicate measurements.

Where three or more analyses of the same parameter are available, the precision will be determined as the RSD according to:

$$\text{RSD} = \frac{\text{Standard deviation}}{\text{Average X}} \times 100$$

### 13.3 COMPLETENESS

Completeness of data generated from a test program is usually calculated as follows:

$$\%\,\text{Completeness} = \frac{\text{Valid data}}{\text{Expected data}} \times 100$$

Data completeness is defined in Section 5.0 of this QAPjP as the percentage of valid data collected from the total number of valid tests conducted. Three valid test runs, at each test condition, are required for the test to be completed. If an individual sample from a test run is lost or broken, the data for that individual analytical parameter may not be 100% complete. This, however, may not invalidate the test run. The completeness objective for this SPT is to generate sufficient data for the regulatory agencies to judge the performance of the system.

# 14.0 AUDIT PROCEDURES, CORRECTIVE ACTION, AND QUALITY ASSURANCE REPORTING

## 14.1 PERFORMANCE AND SYSTEM AUDITS

This section presents information related to the procedures used by the QA staff to assess conformance of the project staff to the specifications contained in the relevant project controlling documents. Further, auditing may be used to assess the ability of subcontractors to successfully perform the work.

### 14.1.1 Field Audits

The QAO assigned to the project will continuously evaluate the operations at the site to ensure that work is being performed in accordance with the various project controlling documents and associated SOPs. Checklists appropriate to the activities may be used. The field audit will cover, but not necessarily limited to, such areas as:

- Conformance to SOPs
- Completeness and accuracy of documentation
- COC procedures
- Compliance with Health and Safety requirements.

### 14.1.2 Performance Evaluations

The QAO or the regulatory agency or both may submit PE samples (referred to elsewhere as "audit samples") to the laboratory. PE samples will be submitted for analysis to demonstrate analytical performance on an as-required basis.

A performance audit sample for PCDD/PCDFs will be requested from the EPA Region Office.

### 14.1.3 Laboratory Audits

INTEC or its appointed representative may choose to audit the laboratories at any time during the project on an as-required basis to assess the laboratory's ability to successfully perform the work and to ensure mutual agreement between INTEC and the laboratory with regard to the scope of work, QA/QC requirements, and deliverable requirements. Reasonable notice will be provided to the laboratory prior to any onsite inspection.

## 14.2 CORRECTIVE ACTION

The following procedures have been established to ensure that nonconforming conditions, such as malfunctions, deficiencies, deviations, and errors, are promptly investigated, documented, evaluated, and corrected. Every person employed in the SPT is expected to function as a QC inspector to ensure the quality of the final product. Quality, as it relates to this SPT, is defined as "performing the work according to the agreed upon specifications contained in the SPT plan and relevant SOPs or causing the specification to be changed *in a controlled manner*." Each individual is encouraged to identify any condition adverse to the successful completion of the work or any modification to the specifications that might result in a better end product. These improvements might be framed in terms of higher quality, greater safety, greater efficiency, and lower cost.

### 14.2.1 Field

When a nonconforming condition or an opportunity for improvement is noted at the site or contractor location, the corrective action provisions of this plan will be invoked to identify the condition and

recommend corrective action. Condition identification, cause, reference documents, and the corrective action planned to be taken will be documented and reported, at a minimum, to the employee's immediate supervisor.

A Corrective Action Request, as shown in Figure 14-1, should be used to identify the adverse condition or opportunity for improvement, reference document(s), and recommended corrective action(s). The Corrective Action Request is directed to the test coordinator. The test coordinator affixes their signature and the date to the corrective action block that states the cause of the condition(s) and corrective action(s) to be taken. The test coordinator is responsible for first notifying the regulatory agency representative of any problems or deviations from the QAPjP or the SPT plan identified in the Corrective Action Request. The test coordinator then forwards the requested response to the QAO for follow-up and filing. The QAO maintains the log for status control of Corrective Action Requests and responses, confirms the adequacy of the intended corrective action(s), and verifies its implementation. The QAO will issue and distribute copies of completed Corrective Action Requests to the originator, test manager, SPT team lead, and the involved contractor(s) if any. Corrective Action Requests are transmitted to the project file for future reference and are incorporated into the test reports.

### 14.2.2 Laboratory

The laboratories' QA manuals and the related SOPs contain detailed discussions of corrective actions to be taken if established criteria fail during laboratory analysis. The laboratory has the responsibility to immediately notify the test coordinator or QAO or both when any analytical QC nonconformance occurs so a mutually acceptable course of action can be pursued.

## 14.3 QUALITY ASSURANCE OFFICER REPORT

The QAO will provide a written report to be included as a section to the final test reports. The QAO report will address:

- Overview of activities and significant events related to QA/QC
- Summary of audit results
- Review of corrective action request status
- Laboratory QA/QC reports
- Data validation results
- Summary of significant changes in procedures or QA/QC programs
- Recommendations.

**Figure 14-1. Example Corrective Action Request Form**

**Corrective Action Request**

Number:
Date:
File Name:
Client: Idaho Nuclear Technology and Engineering Center

**REQUEST**

To:

You are hereby requested to take corrective actions indicated below and as otherwise determined by you (A) to resolve the noted condition and (B) to prevent it from recurring. Your written response is to be returned to the project Quality Assurance Officer or other responsible manager by _____.

**Condition:**

**Reference Documents:**

**Recommended Corrective Actions:**

| Originator | Date | Approval | Date | Approval | Date |
|---|---|---|---|---|---|

**RESPONSE**

**Cause of Condition:**

**Resolution:**

**Prevention:**

**Affected Documents:**

| | |
|---|---|
| Test Manager | Date |
| Quality Assurance Officer | Date |
| QAO Follow-up | Date |
| STP Team Lead | Date |

# Appendix D

# EDF-9483, IWTU System Performance Test: Risk Evaluation for Organic Compounds Emissions

Document ID: EDF-9483
Revision ID: 2
Effective Date: 08/25/11

**Engineering Design File**

# IWTU System Performance Test: Risk Evaluation for Organic Compounds Emissions

Idaho Cleanup Project

CH2M ◆ WG Idaho, LLC is the Idaho Cleanup Project contractor for the U.S. Department of Energy

431.02
02/20/2009
Rev. 19
(Use with MCP-2374 or MCP-2059)

**ENGINEERING DESIGN FILE**

EDF-9483
Revision 2
Page 1 of 10

EDF No.: 9483 EDF Rev. No.: 2 Project No.: 25051 FCF and/or FDC No.: N/A

1. Title: IWTU System Performance Test: Risk Evaluation for Organic Compounds Emissions
2. Index Codes: Building/Type NA SSC ID NA Site Area INTEC
3. Formal Calculation? ☐ Yes (MCP-2374) ☒ No (MCP-2059) Quality Level: 3 QLD Number: 3477

*Formal analyses and calculations require engineering management approval.*

*4. NPH PC or SDC: or ☒ N/A SSC Safety Category: or ☒ N/A

*5. (a) Affects Safety Basis: ☐ Yes ☒ No (b) Affects SNF/HLW: ☐ Yes ☒ No

6. Summary:

A simplified risk assessment for potential organic compound releases from the Integrated Waste Treatment Unit (IWTU) was performed. This risk assessment incorporates available data on organic compounds potentially present in IWTU offgas, a previous risk assessment for the New Waste Calcining Facility, and the Environmental Protection Agency's "RAIS Contaminated Media (Risk) Calculator." Releases of organic compounds from the IWTU are expected to be small to unmeasurable.

This revision (2) updates the risk assessment using the most recent Environmental Protection Agency risk factors. Noncarcinogenic risk increased but is still four orders of magnitude below the target hazard index of 0.25. Potential carcinogenic risk from the project decreased.

7. Signatures: (See instructions for significance of signatures. Add or delete signatories as needed.)

| Name (typed or printed) / Signature and Date | Signatory Role | Organization / Discipline |
|---|---|---|
| Chris Staley<br>Chris Staley 8-23-2011 | Author | Environmental Programs<br>Design Agent |
| James Tkachyk<br>James W. Tkachyk 8/24/11 | Technical Checker | Environmental Project Support<br>Technical Checker |
| Kirk Nielsen<br>KN 8/23/11 | Reviewer | Environmental Programs<br>Requestor (if applicable) |
| Pamela Lilburn<br>Pamela F Lilburn 8-23-11 | Reviewer | Environmental Programs<br>Technical Editor |
| Steve Birrer<br>Steve Birrer 8-25-11 | Reviewer | Environmental Programs<br>Department Manager |
| Roger Wilhelmsen<br>by Steve Birrer 8-25-11 | Document Owner | Environmental Programs<br>Document Owner |

431.02
02/20/2009
Rev. 19
(Use with MCP-2374 or MCP-2059)

**ENGINEERING DESIGN FILE**

EDF-9483
Revision 2
Page 2 of 10

EDF No.: 9483 EDF Rev. No.: 2 Project No.: 25051 FCF and/or FDC No.: N/A

| | | | |
|---|---|---|---|
| 8. | Does document contain sensitive unclassified information?<br>If Yes, what category: | ☐ Yes | ☒ No |
| 9. | Will document be externally distributed? | ☒ Yes | ☐ No |
| | | | |
| 10. | Registered Professional Engineer's Stamp (if required) | | ☐ N/A |

| Registered Professional Engineer Stamp | | |
|---|---|---|
| | | This Engineering Design File was prepared under the direction of the Registered Professional Engineer as indicated by the stamp and signature provided on this page. The Professional Engineer is registered in the State of Idaho to practice _____ Engineering. |

* Not required for commercial level calculations.

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# IWTU System Performance Test: Risk Evaluation for Organic Compounds Emissions

## 1. INTRODUCTION

A simplified risk assessment for potential organic compound releases from the Integrated Waste Treatment Unit (IWTU) was performed. This risk assessment incorporates available data on organic compounds potentially present in IWTU offgas, a previous risk assessment for the New Waste Calcining Facility (NWCF) (Abbott et al. 2000), and the Environmental Protection Agency's (EPA's) "RAIS Contaminated Media (Risk) Calculator" (EPA 2011).

This revision (2) updates the risk assessment using the most recent EPA risk factors. Revisions incorporate changes to or additions of risk factors for several of the chemicals. Acetone, N-nitrosodimethylamine, and hexachlorocyclopentadiene previously had no associated noncancer risk factors for the inhalation pathway. EPA has since added risk factors for these chemicals. EPA also revised downward cancer risk factors for both the inhalation and ingestion pathways, and added a noncancer risk factor for ingestion for N-nitrosodimethylamine.

## 2. SOURCE TERM

Emissions rates for potential organic compounds in the IWTU offgas were derived from several sources (see Table 1):

A. Several compounds are known to be in the tank waste to be processed by IWTU (Swenson 2005). For this risk analysis, the compounds are assumed to pass through the treatment process unaltered.

B. Two compounds were measured in the Hazen test (Olson 2006) of the IWTU process; for this risk analysis, these compounds are assumed to be in the same concentrations in the IWTU offgas.

C. Three compounds dominated the risk for the NWCF offgas (Boardman et al. 2001); these compounds are assumed released from IWTU in proportion to the two facilities' waste feed rates.

Table 1 summarizes IWTU release rates for organic compounds. In Table 1, the release rate calculated for bis-(2-ethylhexyl) phthalate was higher from the NWCF-scaled offgas, and the higher value is used in the risk analysis for conservatism.

Table 1. Organic compounds evaluated for potential risk from Integrated Waste Treatment Unit emissions.

| Organic Compound | Reason of Interest | Concentration in Waste or Offgas | Conversion | Emission Rate From IWTU Stack (g/s) |
|---|---|---|---|---|
| Acetone | In tank waste | 33 µg/L | See Footnote a | 7.29E-06 |
| Bromomethane | | 59 µg/L | | 1.30E-05 |
| Chloromethane | | 75 µg/L | | 1.66E-05 |
| Bis-(2-ethylhexyl) phthalate | | 46 µg/L | | 1.02E-05 |
| Di-n-octyl phthalate | | 20 µg/L | | 4.42E-06 |
| Tri-n-butyl phosphate | | 44 µg/L | | 9.71E-06 |
| Di-n-butyl phthalate | In Hazen emissions | 37.7 µg/dscm | See Footnote b | 1.87E-05 |
| N-nitrosodimethylamine | | 8.7 µg/dscm | | 4.31E-06 |
| Benzo(a)pyrene | Risk driver in NWCF offgas | 5.30E-05 g/s | See Footnote c | 6.87E-05 |
| Bis-(2-ethylhexyl) phthalate | | 9.80E-04 g/s | | 1.27E-03 |
| Hexachlorocyclopentadiene | | 5.50E-04 g/s | | 7.13E-04 |

a. Concentration in feed (µg/L) × feed rate (13.25 L/min) × (1g/1E+6 µg) × (1 min/60 sec);
assumes all organics exit system unreacted.

b. Hazen (Olson 2006) offgas concentration (µg/dscm) × IWTU offgas flow rate (29.7 dscm/min) × (1g/1E+6 µg) × (1 min/60 sec);
assumes offgas concentrations will be the same for Hazen and IWTU.

c. (IWTU waste feed rate [210 gal/hr]) ÷ (NWCF waste feed rate [162 gal/hr]) × (emission rate from NWCF stack);
assumes concentrations are the same in IWTU offgas and NWCF offgas.

NWCF New Waste Calcining Facility

## 3. AIR DISPERSION AND DEPOSITION MODELING

Air modeling using EPA's AERMOD dispersion model (EPA 2004) was performed for the 2009 IWTU air permit revision. However, deposition was not modeled at that time, nor was the available version of the AERMOD code able to model deposition. Air and deposition modeling for the NWCF screening-level risk assessment (Abbott et al. 2000) was performed using the older ISCST3 code (since replaced by AERMOD). The two models produce slightly different results in modeled air concentrations. Nonetheless, the results using the older code are valid (because deposition is directly proportional to air concentration) to derive a deposition rate for IWTU using relative air concentrations and deposition rate for NWCF. Table 2 summarizes the calculation and results.

Table 2. Derivation of deposition rate for the Integrated Waste Treatment Unit.

| | New Waste Calcining Facility (ISCST3) | Integrated Waste Treatment Unit (AERMOD) |
|---|---|---|
| Annual average unit air concentration ($\mu g/m^3$/lb/hr) | 6.20E-02 | 3.33E-02 |
| Vapor wet deposition rate (second/$m^2$/yr) | 3.70E-04 | 1.99E-04[a] |

a. Calculated as: 3.33E-02 ÷ 6.20E-02 × 3.7E-04.

## 4. RISK ASSESSMENT

EPA's "RAIS Contaminated Media (Risk) Calculator" (EPA 2011) was used to calculate risk from air and soil exposure. Unit concentrations (1 $\mu g/m^3$ for air and 1 mg/kg for soil) were input for all compounds, and these unit risks then scaled to the calculated air and soil concentrations for each compound. Default values for risk parameters were used, except that the "Boise Climatic Zone" (climate most similar to Idaho Falls from drop-down list) was selected. Tables 3 and 4 summarize the calculations and results. Note that risks are added for all release scenarios, a highly conservative calculation. Shaded table cells indicate changes from Revision 1.

## 5. DISCUSSION

The highest calculated cancer risk, 4E-08, is from the air exposure pathway (see Table 3). That risk is 250 times below the target risk value of 1E-05 and is due to two chemicals, N-nitrosodimethylamine and benzo(a)pyrene. Of these two, only N-nitrosodimethylamine is expected in the IWTU offgas, as indicated by the Hazen tests (Olson 2006). Table 5 lists release rate limits and offgas concentration limits for these two compounds in order to maintain the cancer risk for each at or below 1E-06, providing a margin of safety (because individual compound risks are added) below the 1E-05 target level.

The highest calculated noncarcinogenic hazard, or hazard index, is 3E-05 from the air pathway (see Table 3), four orders of magnitude below the target hazard index of 0.25. Therefore, no noncarcinogenic impacts are expected from the IWTU offgas.

Table 3. Integrated Waste Treatment Unit—Air concentrations, hazard, and cancer risk from air exposure pathway.

| Compound in Waste | Release Rate for IWTU[a] (g/s) | Air Concentration[b] (µg/m$^3$) | Unit Hazard (hazard/µg/m$^3$) | Hazard Quotient (HQ) | Unit Cancer Risk (risk/µg/m$^3$) | Cancer Risk |
|---|---|---|---|---|---|---|
| Acetone | 7.29E-06 | 1.93E-06 | 3.11E-05[c] | 5.99E-11 | — | — |
| Bromomethane | 1.30E-05 | 3.45E-06 | 1.92E-01 | 6.62E-07 | — | — |
| Chloromethane | 1.66E-05 | 4.38E-06 | 1.07E-02 | 4.69E-08 | 7.40E-07 | 3.24E-12 |
| Bis-(2-ethylhexyl)phthalate | 1.02E-05 | 2.69E-06 | — | — | Evaluated below | |
| Di-n-octyl phthalate | 4.42E-06 | 1.17E-06 | — | — | — | — |
| Tri-n-butyl phosphate | 9.71E-06 | 2.57E-06 | — | — | — | — |
| Di-n-butyl phthalate | 1.87E-05 | 4.94E-06 | — | — | — | — |
| N-nitrosodimethylamine | 4.31E-06 | 1.14E-06 | 2.40E+01 | 2.73E-05 | 1.45E-02 | 1.65E-08 |
| Benzo(a)pyrene | 6.87E-05 | 1.82E-05 | — | — | 1.15E-03 | 2.09E-08 |
| Bis-(2-ethylhexyl)phthalate | 1.27E-03 | 3.36E-04 | — | — | 9.86E-07 | 3.31E-10 |
| Hexachlorocyclopentadiene | 7.13E-04 | 1.89E-04 | 4.79E+00 | 9.03E-04 | — | — |
| | | | Total hazard (hazard index) | 3E-05 | Total cancer risk | 4E-08 |

a. See Table 1 for basis of release calculations.

b. Calculated as: Release rate (g/s) 1lb/454 g × 3,600 s/hr × dispersion factor (3.33E-02 µg/m$^3$/lb/hr from Table 2).

c. Shaded cells indicate changes from Revision 1.

Table 4. Integrated Waste Treatment Unit—Soil concentrations, hazard, and cancer risk from soil exposure pathway.

| Compound in Waste | Release Rate for IWTU[a] (g/s) | Soil Concentration (mg/kg)[b] | Unit Hazard (hazard/mg/kg) | Hazard Quotient (HQ) | Unit Cancer Risk (risk/mg/kg) | Cancer Risk |
|---|---|---|---|---|---|---|
| Acetone | 7.29E-06 | 9.66E-08 | 1.42E-05 | 1.37E-12 | — | — |
| Bromomethane | 1.30E-05 | 1.73E-07 | 1.37E-01 | 2.37E-08 | — | — |
| Chloromethane | 1.66E-05 | 2.20E-07 | 8.42E-03 | 1.85E-09 | 6.05E-07 | 1.33E-13 |
| Bis-(2-ethylhexyl)phthalate | 1.02E-05 | 1.35E-07 | Evaluated below | | Evaluated below | |
| Di-n-octyl phthalate | 4.42E-06 | 5.86E-08 | 4.09E-04 | 2.40E-11 | — | — |
| Tri-n-butyl phosphate | 9.71E-06 | 1.29E-07 | 8.18E-05 | 1.05E-11 | 1.90E-08 | 2.45E-15 |
| Di-n-butyl phthalate | 1.87E-05 | 2.47E-07 | 1.64E-04 | 4.06E-11 | — | — |
| N-nitrosodimethylamine | 4.31E-06 | 5.71E-08 | 2.05E+00[c] | 1.17E-07 | 4.43E-04 | 2.53E-11 |
| Benzo(a pyrene | 6.87E-05 | 9.11E-07 | — | — | 6.77E-05 | 6.17E-11 |
| Bis-(2-ethylhexyl)phthalate | 1.27E-03 | 1.68E-05 | 8.18E-04 | 1.38E-08 | 2.88E-08 | 4.85E-13 |
| Hexachlorocyclopentadiene | 7.13E-04 | 9.46E-06 | 2.73E-03 | 2.58E-08 | — | — |
| | | | Total hazard (hazard index) | 2E-07 | Total cancer risk | 9E-11 |

a. See Table 1 for basis of release calculations.

b. Based on one year's deposition and calculated as follows: Release rate (g/s) × deposition rate (1.99E-04 s/m$^2$/yr from Table 2) ÷ soil depth (0.01 m) ×1,000 mg/g × 1 m$^3$/1E-06 cc ÷ soil density.(1.5 g/cc) × 1,000 g/kg = mg/kg soil concentration.

c. Shaded cells indicate changes from Revision 1.

Table 5. Integrated Waste Treatment Unit release rate and offgas concentration limits needed to maintain less than 1E-06 cancer risk for each compound.

| Compound | IWTU Release Rate Limit to Maintain <1E-06 Cancer Risk (g/s) | Concentration Limit in IWTU Offgas to Maintain <1E-06 Cancer Risk [a] (µg/dscm) |
|---|---|---|
| N-nitrosodimethylamine | 2.61E-04[b] | 5.27E+02 |
| Benzo(a)pyrene | 3.29E-03 | 6.64E+03 |

a. Based on 29.7 dscm/min IWTU offgas flow rate.

b. Shaded cells indicate changes from Revision 1.

## 6. CONCLUSION

Releases of organic compounds from IWTU are expected to be small to unmeasurable. The maximum release rates of the two carcinogenic compounds of consequence, N-nitrosodimethylamine and benzo(a)pyrene, are expected to be 60 and 48 times, respectively, lower than levels that would result in a 1E-06 cancer risk.

## 7. REFERENCES

Abbott, M. L., K. N. Keck, R. E. Schindler, R. L. Van Horn, N. L. Hampton, and M. D. Heiser, 2000, *Screening Level Risk Assessment for the New Waste Calcining Facility*, INEL/EXT-97-00686, Revision 5a, Idaho Cleanup Project.

Boardman, R. D., L. J. Young, N. R. Soelberg, and L. A. Matejka, 2001, *NWCF Calciner Emissions Inventory – Final Report For Phase IV Testing*, INEEL/EXT-01-00260, Idaho National Engineering and Environmental Laboratory.

EPA, 2004, *User's Guide for the AMS/EPA Regulatory Model – AERMOD*, EPA-454/B-03-001, U.S. Environmental Protection Agency.

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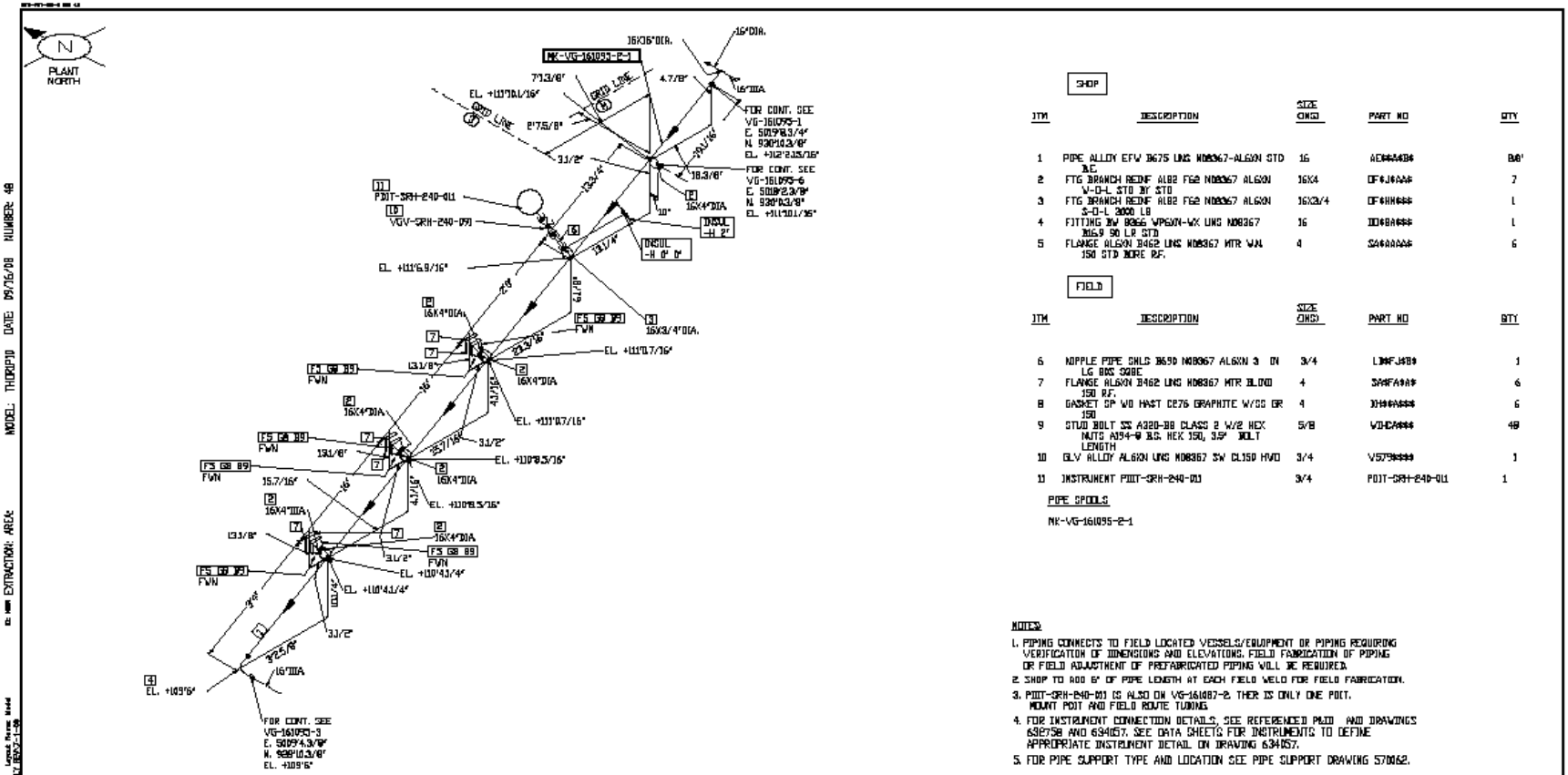
Olson, A., 2006, *Pilot Plant Report for Treating Sodium-Bearing Waste Surrogates Carbonate Flowsheet Integrated Waste Treatment Unit*, TTT-ICPDD-523 RT-ESTD-PMR-001, Rev. 0, Idaho Cleanup Project.

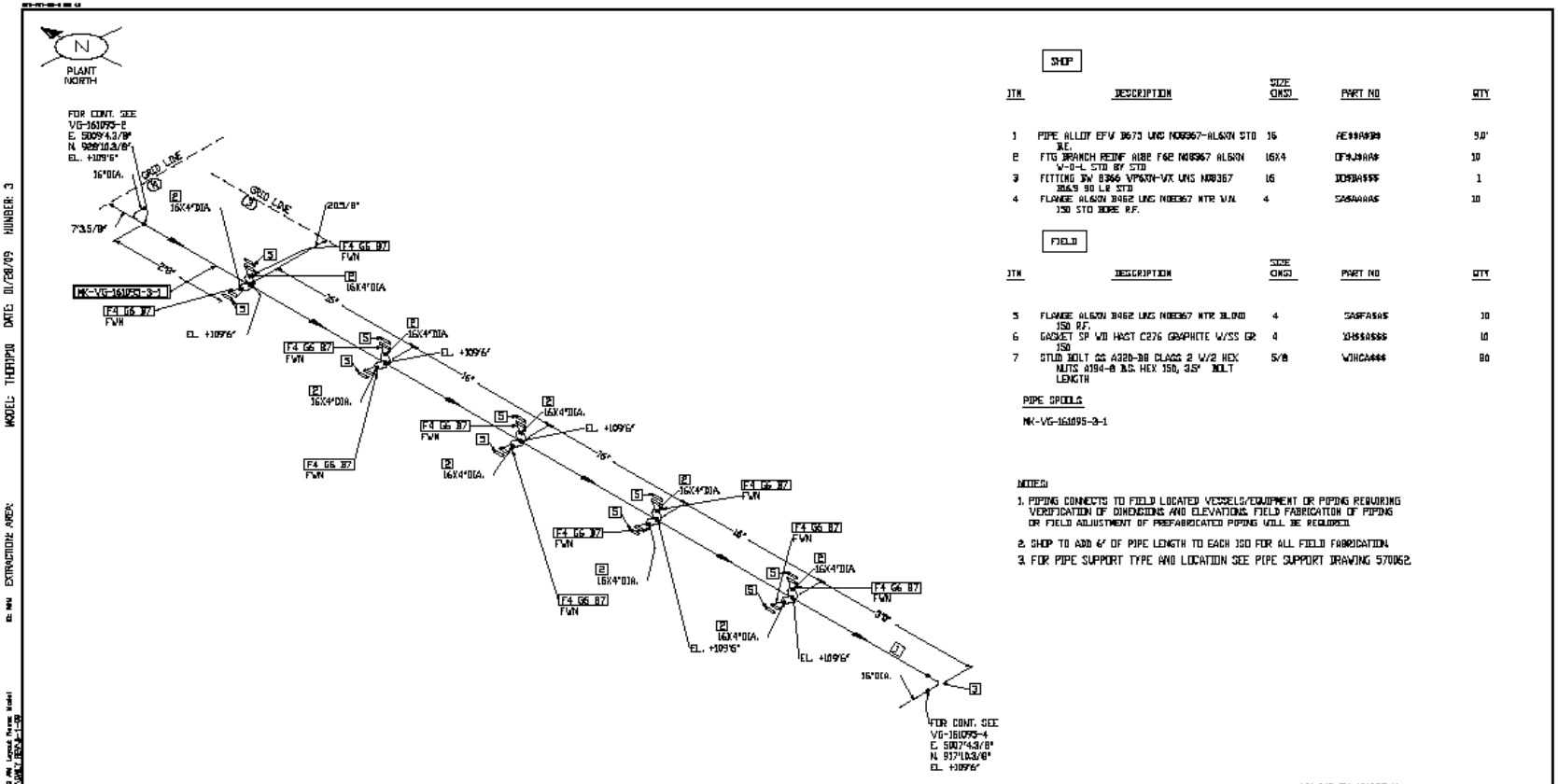
Swenson, M. C., 2005, *Organic Compounds in INTEC Tank Farm Waste*, ICP/EXT-05-00962, Idaho Cleanup Project.

# Appendix E

# Locations of Emissions Sampling Ports

Emissions Test Ports – three on the left and five on the right of the photograph


PLANT NORTH
NK-VG-161095-2-1
FOR CONT. SEE VG-161095-1
FOR CONT. SEE VG-161095-6
FOR CONT. SEE VG-161095-3
PDIT-SRH-240-011
VGV-SRH-240-091
INSUL -H 0" D"
INSUL -H 2"
EL. +111'16.9/16"
EL. +111'07/16"
EL. +110'43/4"
EL. +110'41/4"
EL. +109'6"
16X4"DIA
16X3/4"DIA.
16"DIA.
16"DIA
16X36"DIA.
FWN
SHOP
ITM DESCRIPTION SIZE (INS) PART NO QTY
1 PIPE ALLOY EFW B675 UNS N08367-ALGXN STD B.E. 16 AE6643B# 8.0'
2 FTG BRANCH REDF ALB2 FG2 N08367 ALGXN V-O-L STD BR STD 16X4 DF6J6AA# 7
3 FTG BRANCH REDF ALB2 FG2 N08367 ALGXN S-O-L 2000 LB 16X3/4 DF6HH6## 1
4 FITTING BW B366 WPGWN-WX UNS N08367 B16.9 90 LR STD 16 DD6BA6## 1
5 FLANGE ALGXN B462 UNS N08367 MTR W.N. 150 STD BORE RF. 4 SA6AAAA# 6
FIELD
ITM DESCRIPTION SIZE (INS) PART NO QTY
6 NIPPLE PIPE SMLS B650 N08367 ALGXN 3 IN LG BBS SCHE 3/4 LB#FJ6B# 1
7 FLANGE ALGXN B462 UNS N08367 MTR BLIND 150 RF. 4 SA6FA9A# 6
8 GASKET SP WD HAST CE76 GRAPHITE W/SS GR 150 4 DH6HA6## 6
9 STUD BOLT SS A320-B8 CLASS 2 W/2 HEX NUTS A194-8 B.S. HEX 150, 3.5" BOLT LENGTH 5/8 VDHCA6## 48
10 BLV ALLOY ALGXN UNS N08367 SW CL150 HVD 3/4 V5798### 1
11 INSTRUMENT PDIT-SRH-240-011 3/4 PDIT-SRH-240-011 1
PIPE SPOOLS
NK-VG-161095-2-1
NOTES:
1. PIPING CONNECTS TO FIELD LOCATED VESSELS/EQUIPMENT OR PIPING REQUIRING VERIFICATION OF DIMENSIONS AND ELEVATIONS. FIELD FABRICATION OF PIPING OR FIELD ADJUSTMENT OF PREFABRICATED PIPING WILL BE REQUIRED.
2. SHOP TO ADD 6" OF PIPE LENGTH AT EACH FIELD WELD FOR FIELD FABRICATION.
3. PDIT-SRH-240-011 IS ALSO ON VG-161087-2. THER IS ONLY ONE PDIT. MOUNT PDIT AND FIELD ROUTE TUBING.
4. FOR INSTRUMENT CONNECTION DETAILS, SEE REFERENCED PID AND DRAWINGS 638758 AND 634057. SEE DATA SHEETS FOR INSTRUMENTS TO DEFINE APPROPRIATE INSTRUMENT DETAIL ON DRAWING 634057.
5. FOR PIPE SUPPORT TYPE AND LOCATION SEE PIPE SUPPORT DRAWING 570062.
UNIT/AREA SYSTEM/SERVICE LINE ID SIZE MAT'L SPEC INSUL TYPE INSUL THK TRACING TYPE
SRH VG 161095 4" SK -H 2.00
16" SK -H 2.00
3/4" SK -H 0.00
PLAN NO: N/A
PAID NO: 692799
FILE: P:\2010\DATA\DWG\PIP\VG\VG-161095.THOR.DGN
DSK REF: 50
Richard C. Williams
13847
STATE OF IDAHO
RICHARD C. WILLIAMS
PROJECT NO. 25051
16"-VG-SK-161095-H
REVISION HISTORY
REV DESCRIPTION
6 REMOVED REVISION HISTORY COMPLETELY REVISED PER FCN 01547
SIGNATURES/DATES APPLY TO CURRENT RELEASE ONLY SIGN AND DATE
DESIGN: R. WOODWINE
DRAFTER: K. BRONSON
TECHNICAL CHECK: B. PREUSSNER
ENG GROUP SUPERVISOR: R.L. EASTMAN, P.E.
QUALITY LEVEL: 3
SCALE: NONE
DESIGN LEAD: R. WILLIAMS, P.E.
DESIGN AUTHORITY: D. JOLLY
QUALITY ASSURANCE: M. BARKER
DRAFTING CHECK/ EFFECTIVE DATE: G. MACE
Idaho Cleanup Project
IWTU
VESSEL OFF-GAS
PIPING ISOMETRIC
ISOMETRIC NO.: VG-161095-2
REV 6


N
PLANT NORTH
FOR CONT. SEE VG-161095-2
FOR CONT. SEE VG-161095-4
GRID LINE
16"DIA.
16X4"DIA.
F4 G6 B7 FWN
MK-VG-161095-3-1
EL. +109'6"
SHOP
ITM DESCRIPTION SIZE (INS) PART NO QTY
1 PIPE ALLOY EFW B673 UNS N08367-AL6XN STD BE. 16 AE99A999 9.0'
2 FTG BRANCH REINF A182 F62 N08367 AL6XN W-O-L STD BY STD 16X4 DF9J99A4 10
3 FITTING BW B366 WP6XN-WX UNS N08367 BA.9 90 LR STD 16 DD9BA999 1
4 FLANGE AL6XN B462 UNS N08367 WTR W.N. 150 STD BORE R.F. 4 SA6AAAA5 10
FIELD
ITM DESCRIPTION SIZE (INS) PART NO QTY
5 FLANGE AL6XN B462 UNS N08367 WTR BLIND 150 R.F. 4 SA5FA5A5 10
6 GASKET SP WD HAST C276 GRAPHITE W/SS GR 150 4 XH5AA555 10
7 STUD BOLT SS A320-B8 CLASS 2 W/2 HEX NUTS A194-8 B.S. HEX 150, 3.5" BOLT LENGTH 5/8 W0HCA444 80
PIPE SPOOLS
MK-VG-161095-3-1
NOTES:
1. PIPING CONNECTS TO FIELD LOCATED VESSELS/EQUIPMENT OR PIPING REQUIRING VERIFICATION OF DIMENSIONS AND ELEVATIONS. FIELD FABRICATION OF PIPING OR FIELD ADJUSTMENT OF PREFABRICATED PIPING WILL BE REQUIRED.
2. SHOP TO ADD 6" OF PIPE LENGTH TO EACH ISO FOR ALL FIELD FABRICATION.
3. FOR PIPE SUPPORT TYPE AND LOCATION SEE PIPE SUPPORT DRAWING 570062.
PROJECT NO. 25051
16"-VG-SK-161095-H
UNIT/AREA SYSTEM/SERVICE LINE ID SIZE MAT'L SPEC INSUL TYPE INSUL THK TRACING TYPE
SRH VG 161095 4" SK -H 2.00
16" SK -H 2.00
PLAN NO: N/A
PAND NO: 698799
TASK REF: 30
PROFESSIONAL ENGINEER REGISTERED 10196 STATE OF IDAHO DAVID E. KELLER
REVISION HISTORY
REV DESCRIPTION
0 ISSUED FOR CD-3
1 REROUTED PIPE
2 ADD CONTINUATION LINE NUMBERS
3 DELETED CONTINUATION LINE NUMBERS AND REMOVED NOTE INCORPORATED FCN-00072
4 UPDATE DRAWING
5 COMPLETELY REVISED
SIGNATURES/DATES APPLY TO CURRENT RELEASE ONLY SIGN AND DATE
DESIGN: R. WOODVINE
DRAFTER: K. BRONSON
TECHNICAL CHECK: B. PREUSSNER
ENG GROUP SUPERVISOR: R.L. EASTMAN, P.E.
QUALITY LEVEL: 3 SCALE: NONE
SIGNATURES/DATES APPLY TO CURRENT RELEASE ONLY SIGN AND DATE
DESIGN LEAD: R.C. WILLIAMS
DESIGN AUTHORITY: D. JOLLY
QUALITY ASSURANCE: M. BARKER
DRAFTING CHECK/ EFFECTIVE DATE: G. MACE
Idaho Cleanup Project CH2M-WG
IWTU VESSEL OFF-GAS PIPING ISOMETRIC
ISOMETRIC NO.: VG-161095-3
REV 5

# Appendix F

# Supplemental Sampling Information

AIRTECH Environmental Services Inc.

702 W. 48th Ave. Unit J * Denver, CO 80216
Phone (303) 670-0530 * Fax (303) 670-4130
www.airtechenv.com

March 20, 2012

Kirk Nielsen
IWTU Project Environmental Lead
Environmental Programs
INTEC TR-74 MS 9200

Subject: Idaho Department of Environmental Quality (DEQ) Information Request
Airtech Project No. 3627

Dear Mr. Nielsen;

The information outlined in this revised letter is in response to the request by the State of Idaho DEQ for additional information regarding the System Performance Test (SPT) to be conducted at the Idaho Nuclear Technology and Engineering Center located at the Idaho National Laboratory. The four items being addressed in this transmittal are:

1. A description of the special procedures to be used during the sampling associated with the vertical sampling ports is supplied in Section 1 (attached).
2. A detailed description, of the total hydrocarbons and nitrogen oxides temporary Continuous Emission Monitor System (CEMS) is supplied in Section 2 (attached).
3. Descriptions of the sample recovery procedures including the location of sample recovery and the procedures used to prevent cross contamination of samples are supplied in Section 3 (attached).
4. Copies of the datasheets to be used during the test are supplied in Section 4 (attached).

If you have any questions or require additional information, please feel free to contact me at (303) 670-0530.

Sincerely;
**Airtech Environmental Services Inc.**

Patrick Clark, P.E.

enc.

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*EPA Method 26A Recovery ..... 9*
*EPA Method 29 Recovery ..... 11*
*SW-846 Method 0010 Recovery ..... 13*
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*SW-846 Method 0031 Recovery ..... 17*

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AIRTECH Environmental Services Inc.

## Section 1 – Vertical Port Sampling

All ports will be sealed to prevent duct gases from leaking into the test area. Airtech probes utilize stainless steel tube sheaths which allow for using a gasket to completely seal the probe in the port as shown below:

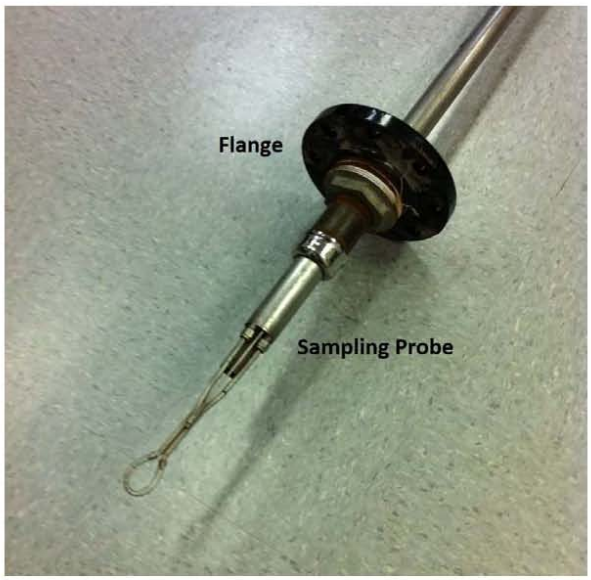



***Figure 1: Sample Probe with Stainless Steel Probe Sheath and Port Seal***

For all isokinetic sample trains, a heated filter box will be mounted on the end of the probe. To connect the back of the filter box to the impingers, a heated flexible Teflon sample line will be used. The impingers will be staged on the ground within approximately 25 feet of the test ports. Prior to testing all sample lines will be cleaned using the steps outlined below:

1.0 Remove any residual solids from the line using a brush

2.0 Soak the line in a hot solution of Alconox non ionic detergent and water

3.0 Scrub the line to ensure that all residual solids are removed

4.0 Rinse the line three (3) times with hot water

5.0 Rinse the line three (3) times with DI water

AIRTECH Environmental Services Inc.

The following table summarizes the additional preparation steps that will be followed as well as the recovery for each line.

| Method | Additional Preparation | Recovery |
|---|---|---|
| 5/26A | No additional preparation<br>Rinse the line three (3) times with ACS grade, or better, acetone and allow to air dry<br>Seal openings with Kimwipes | Rinse three (3) times with DI water and collected into container No. 1 (impinger 1, 2 and 3 contents) |
| 29 | Soak the line in a 10% nitric acid bath for a minimum of four (4) hours<br>Rinse the line three (3) times with deionized (DI) water<br>Rinse the line three (3) times with ACS grade, or better, acetone and allow to air dry<br>Seal openings with Parafilm | Rinse using a total of 100 ml of 0.1 N $HNO_3$ solution and collect in container No. 3 (impinger 1, 2 and 3 contents) |
| 0010 | Rinse the line three (3) times with HPLC grade water<br>Rinse the the line three (3) times with ACS grade dichloromethane<br>Rinse the the line three (3) times with ACS grade acetone<br>Seal openings with aluminum foil | Rinse three (3) times with acetone and dichloromethane solutions and collect in Container No. 5 (back half rinses) |
| 0023A | Rinse the line three (3) times with HPLC grade water<br>Rinse the the line three (3) times with ACS grade dichloromethane<br>Rinse the the line three (3) times with ACS grade acetone<br>Seal openings with aluminum foil | Rinse each piece three (3) times with acetone and collect into container No. 5 (back half rinse)<br>Rinse each piece two (2) times with dichloromethane and collect in Container No. 5 (back half rinse)<br>Rinse each piece two (2) times with toluene and collect in Container No. 6 (back half toluene rinse) – this rinse may be combined with the acetone and methylene chloride rinses and placed into Container No. 5 if sampling for dioxins and furans only |

## Section 2 – Temporary CEMS Description

EPA Methods 3A, 7E, and 25A, will be used to determine the concentration of carbon dioxide, oxygen, nitrogen oxides, and total hydrocarbon from the test location. In EPA Methods 3A, 7E, and 25A, sample gas will be withdrawn from the test location at a constant rate through a stainless steel probe, a glass fiber filter and a Teflon sample line. The probe, filter, and sample line will be operated at a temperature of 250° F to prevent the condensation of moisture. The sample gas will be then split into two separate portions. The first portion will be directed to a pre-conditioning system, and the second portion will be directed to the total hydrocarbon analyzer.

The first portion of the sample gas will pass through a M&C Products compressor gas conditioner to remove moisture and particulate. The clean, dry gas will be then vented to the oxygen, carbon dioxide, and nitrogen oxides analyzers. Results from these analyzers will be determined on a "dry" basis. The second portion of the sample gas will be vented directly to the Flame Ionization Analyzer (FIA). Results from this analyzer will be determined on a "wet" basis. A diagram of the temporary CEMS can be found in Figure 2 on Page 6.

The analyzers that will be used for this project are listed in the table below:

| Parameter | Manufacturer | Model Number | Operating Principle | Units Reported | Range to be used |
|---|---|---|---|---|---|
| Oxygen | API | T803 | Paramagnetic | % | 0 - 16 |
| Carbon Dioxide | API | T803# | Infrared | % | 0 - 16 |
| Nitrogen Oxides | Thermo Environmental | 42IHL | Chemi-luminescence | ppm | 0 - 100 or 0-1,000 |
| Total Hydrocarbons | JUM | 3-300 | Flame Ionization Detector | ppm | 0 - 100 |

Prior to sampling, a calibration error test will be performed on the oxygen, carbon dioxide, and nitrogen oxides analyzers. The zero and high-range calibration gases for each constituent will be introduced directly into each analyzer. Each analyzer will be then adjusted to the appropriate values. The mid-range gas will be then introduced to each analyzer, and the measured values will be recorded. The measured values for each calibration gas will be then compared to the calibration gas values, and the differences must be less than the method requirement of two (2) percent of the span value.

A sample system bias check will be then performed on the oxygen, carbon dioxide, and nitrogen oxides analyzers by introducing the zero and mid-range calibration gases into the sampling system prior to the filter. The gas will be drawn through the entire

sampling system. The measured responses will then be compared to the calibration error test values to determine the bias in response due to the sampling system. The sampling system bias must be less than the method requirement of five (5) percent of the span value.

Prior to sampling, a calibration error test will also be performed on the FIA. The zero and high-range calibration gases will be introduced into the sampling system prior to the filter. The gas will be drawn through the entire sampling system, and the FIA will be adjusted to the appropriate values. The mid and low-range gases will then be introduced to the FIA, and the measured values will be recorded. The measured values for each calibration gas will be then compared to the calibration gas values and the differences must be less than the method requirement of five (5) percent of the actual value.

In addition, the response time will be determined by measuring the time required for each analyzer to reach ninety-five percent (95%) of its high-range calibration gas value.

Prior to sampling a stratification test will be performed as required in Section 8.1.2 of Method 7E. If the gas stream is found to be un-stratified, sampling will be conducted at a single point near the centroid of the duct.

Sampling will be conducted over the course of the test program. One (1) minute rolling averages of one (1) second readings, will be collected using DaisyLab data acquisition software and a Measurement Computing Model USB-1608FS analogue to digital converter.

After each test run the instrument drift for each analyzer will be determined by introducing the zero and mid-range calibration gases into the sampling system prior to the filter. The gas will be drawn through the entire sampling system. The measured responses will then be compared to the values from the previous test run to determine the analyzer drift. For all test runs, the analyzer drift must be less than the method requirement of three (3) percent of the span value.

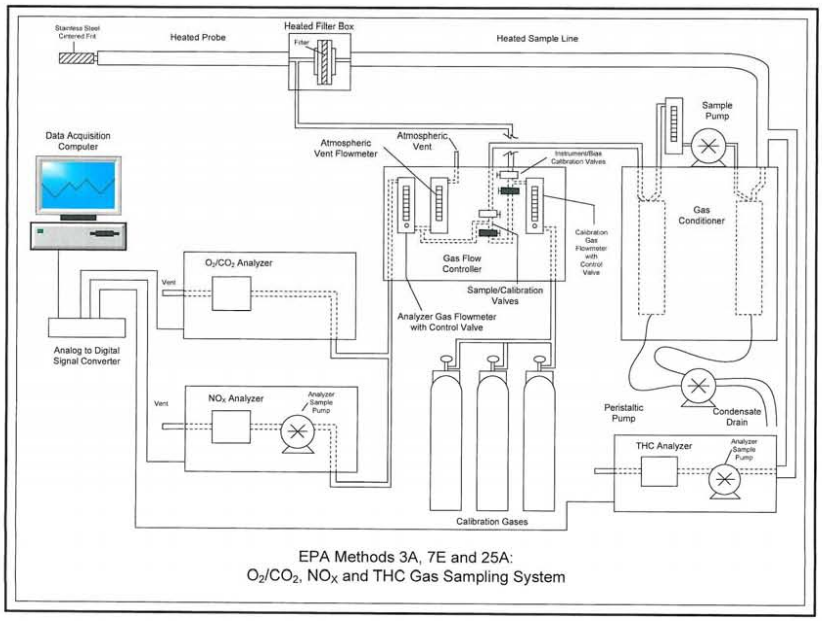



*Figure 2: Diagram of the Temporary CEMS*

## Section 3 – Sample Recovery Procedures

The sample recovery procedures for each method that will be utilized in the SPT can be found on the following pages.

To prevent the reagents used in many of the sampling trains (i.e. acetone, methylene chloride, toluene) from contaminating the samples collected using the Method 0031 Volatile Organic Sampling Train (VOST), two sample recovery trailers will be used. The one trailer will be used for the recovery of all isokinetic sampling trains (Methods 5/26A, 29, 0010, 0023A). The second trailer will be used for the storage of all VOST sampling equipment and adsorbent traps (both used and unused). During sample recovery near the test locations, the VOST sampling train will not be recovered if any solvents are being used in the area (i.e. for probe washing). VOST samples will be shipped to TestAmerica separately from all other samples.

In the trailer used for recovery of the Methods 5/26A, 29, 0010, and 0023A sample trains, separate stations will be set up for each method. The separate, uniquely labeled, and segregated stations are used to prevent cross contamination of samples and reagents. Each method station will have the following minimum components:

- A printed sample recovery diagram for the specified method
- Uniquely labeled squeeze bottles for each reagent used for the specified method
- A complete, unique and segregated set of tweezers / brushes used for recovery of the specified method
- A complete set of reagents used for recovery of the specified method
- Clearly labeled sample bottles used for that specified method

Sample trains will be sealed with Teflon tape when not in use and components will be sealed when being transported between the laboratory and the test locations. All technicians will wear polyethylene or other plastic gloves while recovering field samples.

**EPA Method 5 Recovery**

***Samples to be collected for each train:***

Container No. 1 (Glass Fiber Filter) including loose particulate is collected in a plastic or glass Petri dish.

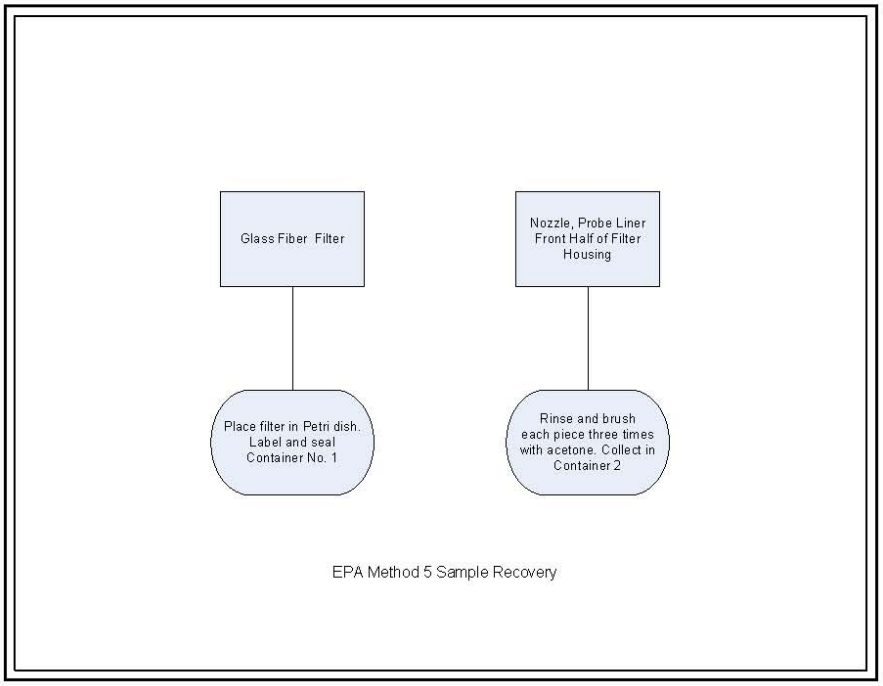
Container No. 2 (F1/2 Acetone Rinse) The front half rinses, consisting of a rinse and brush of the nozzle, probe liner and front half of the filter housing is collected into a glass 250 ml sample jar.

***Reagent blanks to be collected for each test set***

Place at least one glass fiber filter from the same lot as the filters used for sampling in a Petri dish.

Collect 200 ml of acetone into a glass sample jar.

***EPA Method 5 Recovery Scheme***



**EPA Method 26A Recovery**

***Samples to be collected for each train:***

Container No. 1 (Contents of impingers 1 through 3) is collected in a 1000 ml HDPE sample jar.

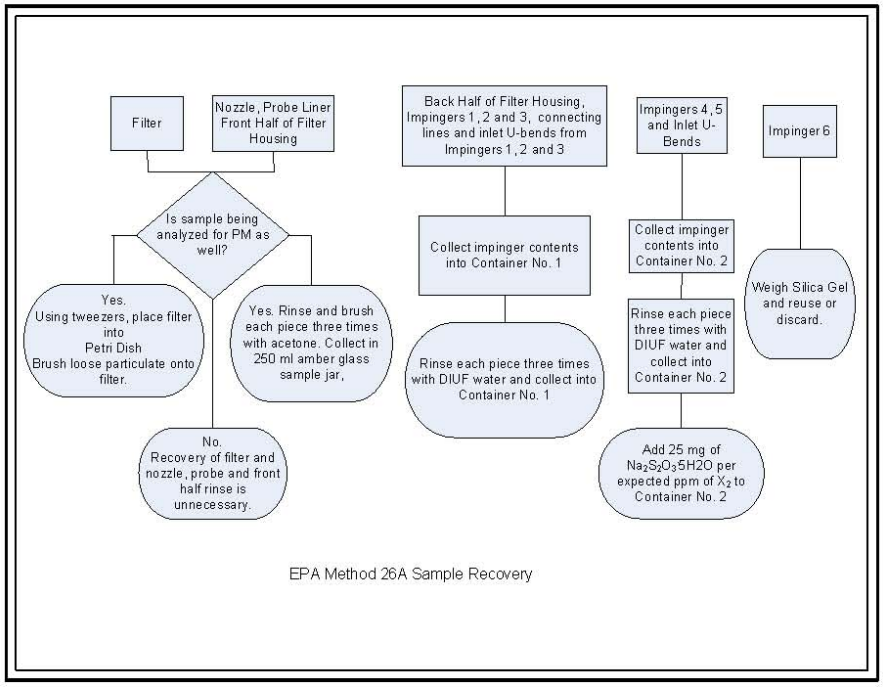
Container No. 2 (Contents of impingers 4 and 5) is collected in a 500 ml HDPE sample jar.

***Reagent blanks to be collected for each test set***

Collect a minimum of 250 ml of the 0.1N $H_2SO_4$ solution in a 500 ml HDPE sample jar. Add enough DIUF water to bring the final volume to that of the field samples.

Collect a minimum of 200 ml of the 0.1N NaOH solution in a 500 ml HDPE sample jar. Add enough DIUF water to bring the final volume to that of the field samples.

***EPA Method 26A Recovery Scheme***



***EPA Method 26A Notes***

- NOTE: If using a Teflon line between the filter and the first impinger, the line must be rinsed in the same manner as impingers 1, 2 and 3 and collected in container 1.
- EPA Method 26A may be combined with EPA Method 5 to additionally determine filterable particulate matter emissions. The types of filter listed above should be used tared.
- Additional impinger may be required to test at high moisture sources.
- **NOTE**: Filter temperature cannot fall below 248°F.

AIRTECH Environmental Services Inc.

**EPA Method 29 Recovery**

***Samples to be collected for each train:***

Container No. 1 (Quartz Fiber Filter): Collect the quartz filter in a 250 ml amber glass sample jar or Petri dish. The Petri dish is required if particulate analysis is being performed (if not use 250 ml glass sample jar).

Container No.2A (F1/2 Acetone Rinse) is collected in a 250 ml amber glass sample jar. Container 2A is only required if particulate analysis is being performed.

Container No. 2 (0.1N $HNO_3$ Rinse) is collected in a 250 ml amber glass sample jar. (A HDPE sample jar may also be used).

Container No. 3 (Contents of Impingers 1-3 and Glassware Rinses) is collected in a 950 ml amber glass sample jar. (A HDPE sample container may also be used).

Container No. 4 (Contents of Impinger 4 and Glassware Rinses) is collected in a 125 ml or 250 ml amber glass sample jar. Container 4 is required because mercury analysis is being performed.

Container No. 5 (Contents of Impingers 5 and 6 and Glassware Rinses) is collected in a 500 ml amber glass sample jar (a vented sample jar is preferred due to pressure buildup in sample). Container 5 is required because mercury analysis is being performed.

Container No. 6 (8N HCl rinse of Impingers 5, 6 and U-bends) is collected in a 250 ml amber glass sample jar. Container 6 is required if mercury analysis because being performed.

***Reagent blanks to be collected for each test set***

Collect a minimum of 300 ml of 0.1 N $HNO_3$ rinse solution into a 500 ml amber glass sample jar (HDPE may also be used).

Collect a minimum of 200 ml of the 5%$HNO_3$/10% $H_2O_2$ solution into a 500 ml amber glass sample jar (HDPE may also be used).

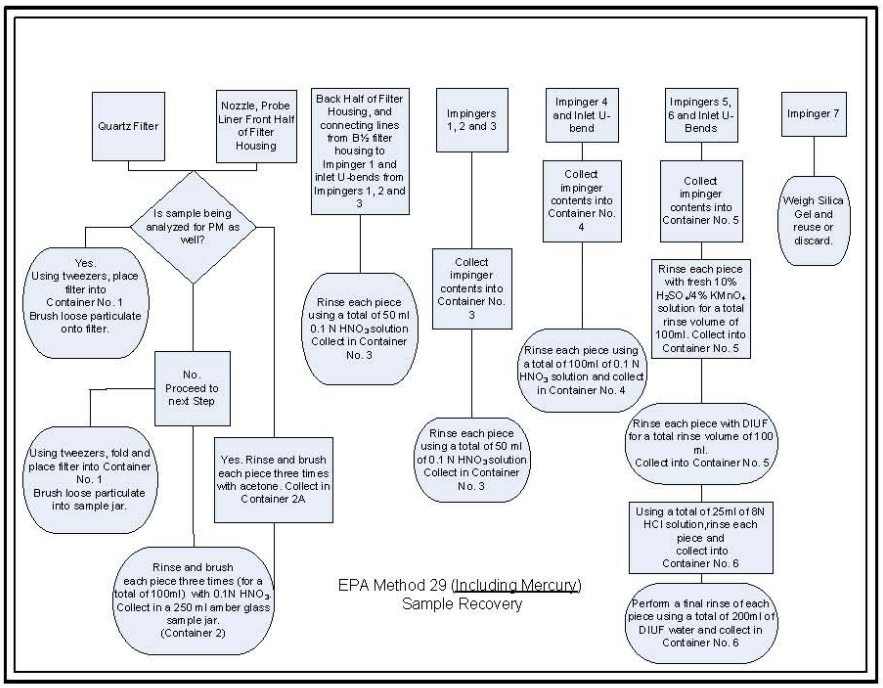
Collect a minimum of 200 ml of 10%$H_2SO_4$/4% $KMnO_4$ mercury absorbing solution into a 500 ml amber glass sample jar. This blank is only required if mercury analysis is performed.

Collect 25 ml of the 8N HCl rinse solution into a 250 ml amber glass sample jar containing 200 ml DIUF water. This blank is only required if mercury analysis is performed.

Place three (3) unused quartz filters into an amber glass sample jar or clean Petri dish.

Field blanks for Method 29 may also be required, depending on the individual project.

***EPA Method 29 Recovery Scheme (Including Mercury)***




***EPA Method 29 Notes***

- NOTE: If using a Teflon line between the filter and the first impinger, the line must be rinsed in the same manner as impingers 1, 2 and 3 (100 ml of 0.1$HNO_3$) and collected in container 3.
- The 10% sulfuric acid/4% potassium permanganate solution must be prepared (acidified) fresh daily.
- This method may be combined with EPA Method 5 to additionally determine filterable particulate matter emissions. The types of filter listed above should be tared.
- There are two options for this sample train, as described previously.
- This method is identical to SW-846 Method 0060 for testing and recovery.
- Rinse volumes must be exact for subsequent blank corrections.
- The back of the filter housing and Impingers 1, 2, and 3 are rinsed with 50 ml of 0.1N $HNO_3$ each and collected in Container 3 for the total 100 ml rinse.

**SW-846 Method 0010 Recovery**

***Samples to be collected for each train***

Container No. 1 (Filter): Place filter in a 250 ml amber glass sample jar or glass Petri dish.

Container No. 2 (XAD Trap): Seal the ends of the trap with Teflon tape, wrap entirely in aluminum foil and place in a large Ziploc bag. Store in cooler on ice or ice packs.

Container No. 3 (F1/2 Rinse) is collected in a 250 ml amber glass sample jar.

Container No. 4 (Impinger Catch) is collected in a 950 ml amber glass sample jar.

Container No. 5 (B1/2 Rinse) is collected in a 250 ml amber glass sample jar.

***Field blanks to be collected for each test set***

The specific project may require a field blank at each location, or one for the whole project. The field blank is performed by assembling a sample train, heating it to temperature and leak checking it. The sample train should be allowed to stand near the location for the same duration as a sample test run. Recovery procedures used for the field blank are identical to those used for an actual test run.

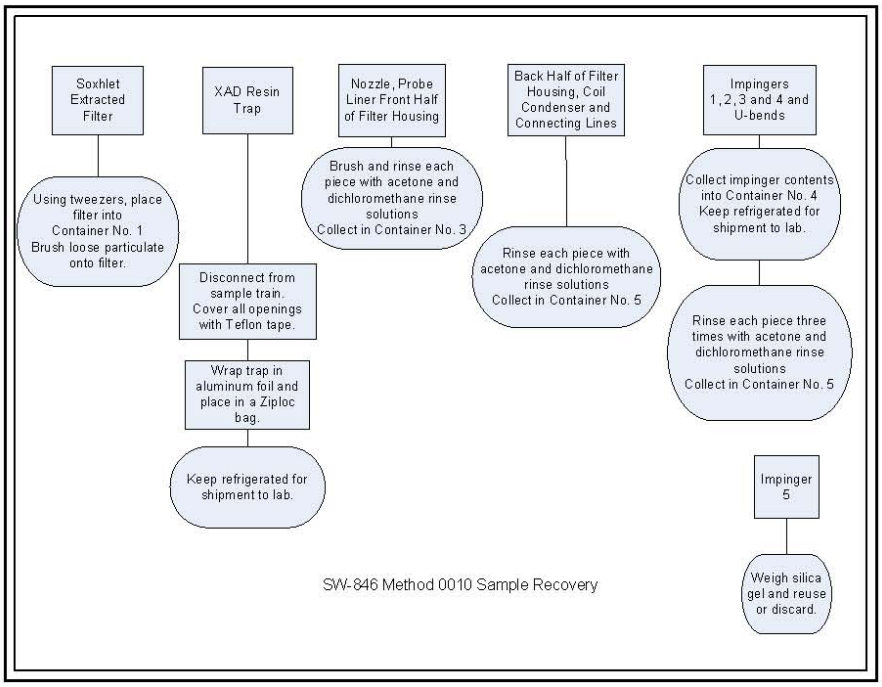
***Reagent blanks to be collected for each test set***

The following reagent blanks should be collected. They are not typically analyzed and only serve as a back up QA/QC check.

Collect a minimum of 200 ml of acetone and dichloromethane rinse solutions into a 250 ml amber glass sample jars.

Collect a minimum of 200 ml of the HPLC water into a 250 ml amber glass sample jar.

***SW-846 Method 0010 Recovery Scheme***




***SW-846 Method 0010 Notes***

- NOTE: If using a heated Teflon line between the filter and the condenser, the line must be rinsed in the same manner as the back half of the filter and condenser and collected in container 5.
- Testing performed by Airtech Environmental Services Inc. will typically require the substitution of a 50% acetone/ 50% dichloromethane for the 50% dichloromethane/50% methanol rinse solution. Recovery is performed in an otherwise identical manner. Separate reagent blanks of acetone and dichloromethane should be collected when using this substitution.
- The 50% acetone/ 50% dichloromethane solution will be separate acetone rinses followed by dichloromethane rinses.

AIRTECH Environmental Services Inc.

**SW-846 Method 0023A Recovery**

***Samples to be collected for each train***

Container No. 1 (Filter): Place filter in a 250 ml amber glass sample jar or glass Petri dish.

Container No. 2 (XAD Trap): Seal the ends of the trap with Teflon tape, wrap entirely in aluminum foil and place in a large Ziploc bag. Store in cooler on ice or ice packs.

Container No. 3 (F1/2 Acetone and Dichloromethane Rinses) are collected in a 250 or 500 ml amber glass sample jar.

Container No. 4 (F1/2 Toluene Rinse) is collected in a 250 ml amber glass sample jar. For dioxins and furans sampling only, this rinse may be combined with the acetone and methylene chloride rinses.

Container No. 5 (B1/2 Acetone and Dichloromethane Rinses) are collected in a 250 ml amber glass sample jar.

Container No. 6 (B1/2 Toluene Rinse) is collected in a 250 ml amber glass sample jar. For dioxins and furans sampling only, this rinse may be combined with the acetone and methylene chloride rinses.

Container No. 7 (Impinger Contents) is collected in a 950 ml amber glass sample jar.

***Field blanks to be collected for each test set***

The specific project may require a field blank at each location, or one for the whole project. The field blank is performed by assembling a sample train, heating it to temperature and leak checking it. The sample train should be allowed to stand near the location for the same duration as a sample test run. Recovery procedures used for the field blank are identical to those used for an actual test run.

***Reagent blanks to be collected for each test set***

The following reagent blanks should be collected. They are not typically analyzed and only serve as a back up QA/QC check.

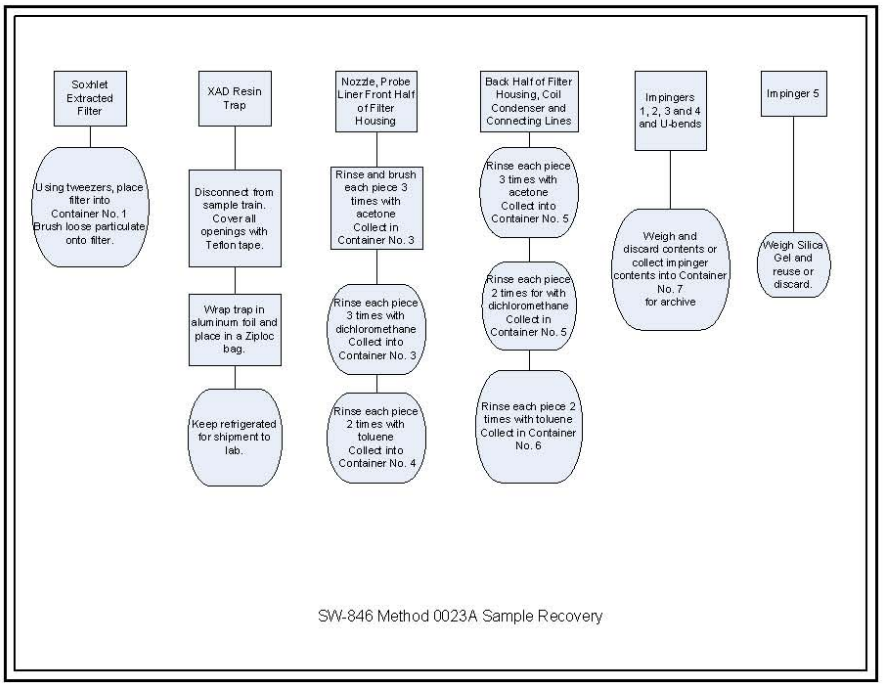
Collect a minimum of 200 ml of acetone into a 250 ml amber glass sample jar.

Collect a minimum of 200 ml of dichloromethane into a 250 ml amber glass sample jar.

Collect a minimum of 200 ml of toluene into a 250 ml amber glass sample jar.

Collect a minimum of 200 ml of HPLC grade water into a 250 ml amber glass sample jar.

***SW-846 Method 0023A Recovery***




***SW-846 Method 0023A Notes***

- NOTE: If using a heated Teflon line between the filter and the condenser, the line must be rinsed in the same manner as the back half of the filter and condenser and collected in containers 5 and 6 as appropriate.
- Make sure to include a thermocouple to monitor the trap temperature when setting up the sample train.
- Store traps on ice or ice packs.
- SW-846 Method 0023A has slight differences in recovery from the very similar EPA Method 23 recovery. In Method 0023A, the applicable glassware is only rinsed, instead of soaked. Method 0023A also has separate back half and front half rinses
- SW-846 Method 0023A may be combined with SW-846 Method 0010 in some instances.

AIRTECH Environmental Services Inc.

**SW-846 Method 0031 Recovery**

***Samples to be collected for each train***

Tube No. 1 (Tenax®-GC trap): Seal the ends of the trap with the original tube fittings and place in the original plastic sleeve. Store the trap in a cooler on ice or ice packs.

Tube No. 2 (Tenax®-GC trap): Seal the ends of the trap with the original tube fittings and place in the original plastic sleeve. Store the trap in a cooler on ice or ice packs.

Tube No. 3 (Anasorb®-747 trap): Seal the ends of the trap with the original tube fittings and place in the original plastic sleeve. Store the trap in a cooler on ice or ice packs.

Container No. 1 (condensate): Place condensate in a 40 or 80 VOA vial and top off with VOC free water supplied by laboratory. Store the sample in a cooler on ice or ice packs.

***Field blanks to be collected for each test set***

The specific project may require a field blank at each location, or one for the whole project. The field blank is performed by assembling a sample train, heating it to temperature and leak checking it. The sample train should be allowed to stand near the location for the same duration as a sample test run. Recovery procedures used for the field blank are identical to those used for an actual test run.

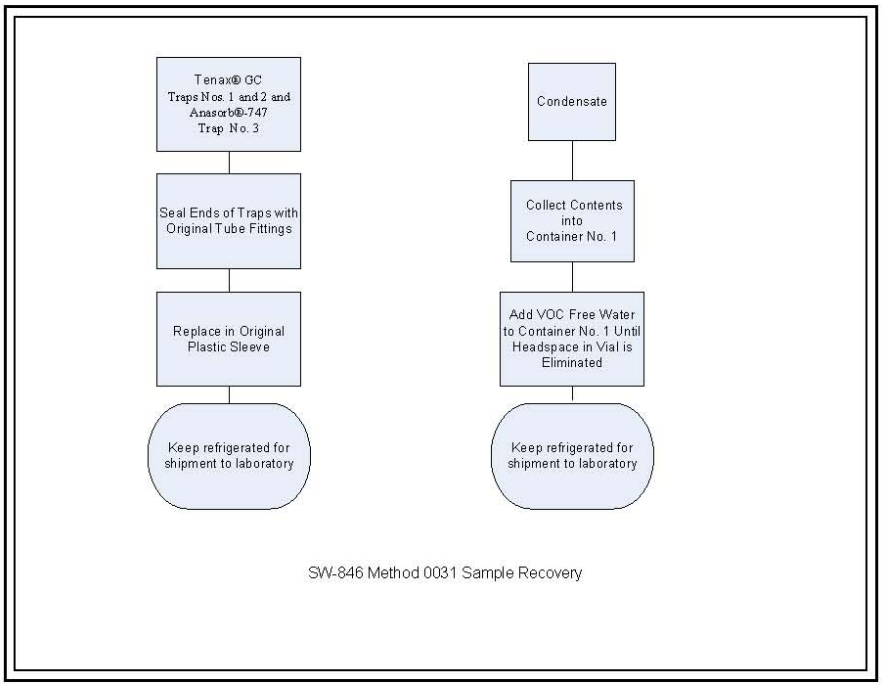
***Trip blanks to be collected for each test set***

The following trip blanks should be collected. These trip blanks should be treated like any other tubes except that the end caps will not be removed during storage at the site. This set of tubes should be analyzed to assess contamination which may occur during storage and shipment.

Collect two (2) Tenax®-GC traps.

Collect one (1) Anasorb®-747 trap.

***SW-846 Method 0031 Recovery***




***SW-846 Method 0031 Notes***

- NOTE: Great care should be taken to prevent cross contamination of the Method 0031 samples with solvents from other test methods.
- Do not use any squeeze bottles for topping of the condensate VOA vial. Pour directly from the VOC free water supplied by the laboratory.

AIRTECH Environmental Services Inc.

## Section 4 - Datasheets

Copies of all datasheets to be completed by Airtech personnel are attached.

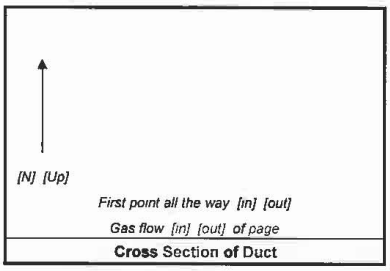
AIRTECH Environmental Services Inc.

## Airtech Environmental Services, Inc.

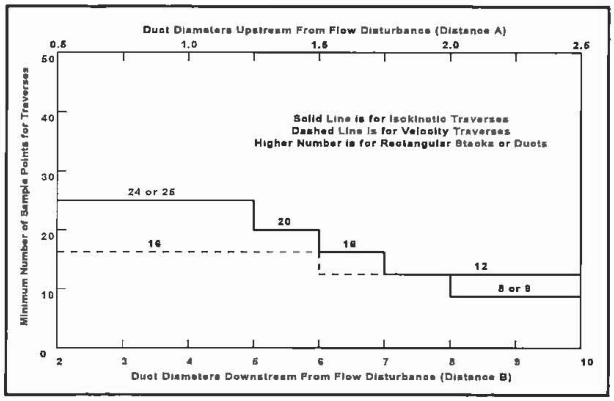
Method 1 Data Sheet

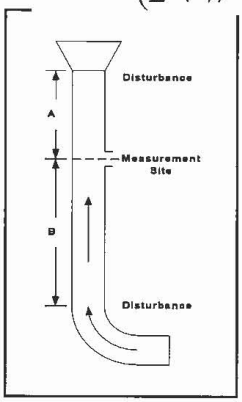
**LOCATION** _____

| | |
|---|---|
| Client | |
| Project No: | |
| Plant | |
| Date | |
| Technician | |
| Duct Diameter (in.) | |
| Port Diameter (in.) | |
| Port Length (in.) | |
| Port Type | |
| Distance A (ft) | |
| Distance B (ft) | |
| Distance A (Duct Diameters) | |
| Distance B (Duct Diameters) | |




For rectangular ducts $$ED = \frac{2LW}{(L+W)}$$



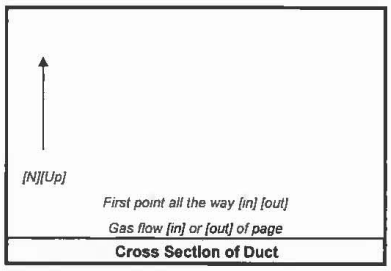




| Location Schematic and Notes | Traverse Point | Distance (in.) |
|---|---|---|
| | 1 | |
| | 2 | |
| | 3 | |
| | 4 | |
| | 5 | |
| | 6 | |
| | 7 | |
| | 8 | |
| | 9 | |
| | 10 | |
| | 11 | |
| | 12 | |
| | 13 | |
| | 14 | |
| *Indicate sample ports, height from grade, types of disturbances, access, unistrut configuration, etc.* | 15 | |
| *Distance to point must include length of port* | 16 | |

**AIRTECH ENVIRONMENTAL SERVICES INC.**
Cyclonic Flow Traverse Data Sheet

| Page | | of | |
|---|---|---|---|

| | | | |
|---|---|---|---|
| Client | | | |
| Plant | | | |
| Location | | | |
| Project Number | | Duct size (in) | |
| Date | | Port Length (in) | |
| Probe ID | | Pitot Cp | |




| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Run Number | | | | Run Number | | | | Run Number | | | |
| Start Time | | | | Start Time | | | | Start Time | | | |
| Stop Time | | | | Stop Time | | | | Stop Time | | | |
| Barometric (inHg) | | | | Barometric (inHg) | | | | Barometric (inHg) | | | |
| Static (in$H_2O$) | | | | Static (in$H_2O$) | | | | Static (in$H_2O$) | | | |
| Probe Operator | | | | Probe Operator | | | | Probe Operator | | | |
| Data Recorder | | | | Data Recorder | | | | Data Recorder | | | |
| Pre Leak Check | | | | Pre Leak Check | | | | Pre Leak Check | | | |
| Post Leak Check | | | | Post Leak Check | | | | Post Leak Check | | | |
| Traverse Point | Pressure $\Delta P$ @ $0^0$ (in $H_2O$) | Angle $\alpha$ (< $20^0$) | Notes | Traverse Point | Pressure $\Delta P$ @ $0^0$ (in $H_2O$) | Angle $\alpha$ (< $20^0$) | Notes | Traverse Point | Pressure $\Delta P$ @ $0^0$ (in $H_2O$) | Angle $\alpha$ (< $20^0$) | Notes |
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| Total | | | | Total | | | | Total | | | |
| Average | | | | Average | | | | Average | | | |

**AIRTECH ENVIRONMENTAL SERVICES INC.**
Impinger Weights Data Sheet

PROJECT NO. _____

| Page | | of | |
|---|---|---|---|

| | | | |
|---|---|---|---|
| Client | | | |
| Plant | | | |
| Location | | | |
| Date | | Unit | |
| Operator | | | |

| | | | | | |
|---|---|---|---|---|---|
| Run No. | | | | | |
| Method No. | | Train ID | | Filter No. | |
| | Contents | Tare with Contents (g) | Final (g) | Total (g) | Notes |
| Impinger No. 1 | | | | | |
| Impinger No. 2 | | | | | |
| Impinger No. 3 | | | | | |
| Impinger No. 4 | | | | | |
| Impinger No. 5 | | | | | |
| Impinger No. 6 | | | | | |
| Impinger No. 7 | | | | | |
| Additional Rinse | | | | | |
| | | | Net Weight (g) | | |

| | | | | | |
|---|---|---|---|---|---|
| Run No. | | | | | |
| Method No. | | Train ID | | Filter No. | |
| | Contents | Tare with Contents (g) | Final (g) | Total (g) | Notes |
| Impinger No. 1 | | | | | |
| Impinger No. 2 | | | | | |
| Impinger No. 3 | | | | | |
| Impinger No. 4 | | | | | |
| Impinger No. 5 | | | | | |
| Impinger No. 6 | | | | | |
| Impinger No. 7 | | | | | |
| Additional Rinse | | | | | |
| | | | Net Weight (g) | | |

| | | | | | |
|---|---|---|---|---|---|
| Run No. | | | | | |
| Method No. | | Train ID | | Filter No. | |
| | Contents | Tare with Contents (g) | Final (g) | Total (g) | Notes |
| Impinger No. 1 | | | | | |
| Impinger No. 2 | | | | | |
| Impinger No. 3 | | | | | |
| Impinger No. 4 | | | | | |
| Impinger No. 5 | | | | | |
| Impinger No. 6 | | | | | |
| Impinger No. 7 | | | | | |
| Additional Rinse | | | | | |
| | | | Net Weight (g) | | |

## AIRTECH ENVIRONMENTAL SERVICES INC.

General Testing Data Sheet

TESTING TYPE: _____

RUN NO. _____ METHOD NO. _____ Page ____ of ____

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Client | | | | | | | | Barometric (InHg) | | Water [ml] [g] | |
| Plant | | | | | | | | Ambient Temp (°F) | | Silica gel (g) | |
| Location | | | | | | | | Static ($InH_2O$) | | Total Vlc | |
| Date | | Project No. | | | | | | Probe ID | | Liner Type | |
| Meter Operator | | | | | | | | Nozzle ID | | Nozzle Dia (in) | |
| Probe Operator | | | | | | | | Filter ID | | | |
| Meter ID | | Yd | | Pitot Cp | | [N] [Up] | | Train ID | | Train Type | |
| ΔH@ | | Kf | | Leak check | | First point all the way [in] [out] | | Duct Dim. (in) | | Port Length (in) | |
| Pre Leak Check | | [cfm] [lpm] @ | | (InHg) | | Gas flow [in] [out] of page | | | | | |
| Post Leak Check | | [cfm] [lpm] @ | | (InHg) | | Cross Section of Duct | | Start Time | | Stop Time | |

| Traverse Point | Min/Point Elapsed Time | Velocity Pressure ΔP ($InH_2O$) | Orifice Setting ΔH ($InH_2O$) | Gas Sample Volume Initial [$f^3$] [l] | Stack Temp (°F) | Probe Temp (°F) | Filter Temp (°F) | Impinger Outlet Temp (°F) | DGM Inlet Temp (°F) | DGM Outlet Temp (°F) | Pump Vacuum (InHg) | Auxiliary Temp (°F) | | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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*Circle correct bracketed [ ] units*
*Train Type denotes impingers, knockouts, etc*

## AIRTECH ENVIRONMENTAL SERVICES INC.

V.O.S.T Meter Data Sheet

Run No. _____ METHOD _____

| Page | | of | |
|---|---|---|---|

| | |
|---|---|
| Client | |
| Plant | |
| Location | |
| Date | |
| Project No. | |
| Probe Operator | |

*[N][Up]*

*Gas flow [in] or [out] of page*

**Cross Section of Duct**

| | |
|---|---|
| Barometric (in. Hg) | |
| Static (in$H_2O$) | |
| Ambient Temp. ($^0$F) | |
| Start Time | |
| Stop Time | |

| Unspiked Train | Meter ID | | Yd | |
|---|---|---|---|---|
| Pre Leak Check | | lpm @ | | (in. Hg) |

| Min/Point / Elapsed Time | Orifice Setting ΔH (in$H_2O$) | Gas Sample Volume Initial [l] | Flow Meter Setting | DGM Inlet Temp ($^0$F) | DGM Outlet Temp ($^0$F) | Pump Vaccum (In Hg) |
|---|---|---|---|---|---|---|
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| Total | | | | | | |
| Average | | | | | | |

| Spiked Train | Meter ID | | Yd | |
|---|---|---|---|---|
| Pre Leak Check | | lpm @ | | (in. Hg) |

| Min/Point / Elapsed Time | Orifice Setting ΔH (in$H_2O$) | Gas Sample Volume Initial [l] | Flow Meter Setting | DGM Inlet Temp ($^0$F) | DGM Outlet Temp ($^0$F) | Pump Vaccum (In Hg) |
|---|---|---|---|---|---|---|
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| Total | | | | | | |
| Average | | | | | | |

**Airtech Environmental Services, Inc**
CEM ANALYZER I D. Data Sheet

Project No. ______________ Page ____ of ____

| | | | |
|---|---|---|---|
| Client | | | |
| Plant | | | |
| Location | | | |
| Date | | Unit | |
| Operator/Tech | | | |

| Analyzer Type | Model | Notes |
|---|---|---|
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| Equipment | Type/Lengths | Used Yes |
|---|---|---|
| Probe | | |
| Filter | | |
| HSL | | |
| Cold Lines | | |
| Gas Conditioner | | |
| DAS Computer | | |
| Flow Panel | | |
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| Cylinder Contents | Cylinder No. | PPM |
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| CEM Schematic and Notes |
|---|
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**Airtech Environmental Services, Inc.**
Nozzle Calibration Form

| | | | |
|---|---|---|---|
| Client | | Job No. | |
| Plant | | | |

| | Nozzle 1 | Nozzle 2 | Nozzle 3 |
|---|---|---|---|
| Date | | | |
| Nozzle ID | | | |
| Operator | | | |
| Test Location | | | |
| Run Number (s) | | | |
| Diameter 1 | | | |
| Diameter 2 | | | |
| Diameter 3 | | | |
| Average | | | |

| | Nozzle 4 | Nozzle 5 | Nozzle 6 |
|---|---|---|---|
| Date | | | |
| Nozzle ID | | | |
| Operator | | | |
| Test Location | | | |
| Run Number (s) | | | |
| Diameter 1 | | | |
| Diameter 2 | | | |
| Diameter 3 | | | |
| Average | | | |

Notes:
Measurements must be made to the nearest 0.001 inches.
Three different diameters should be measured.
The difference between the high and low measurement must be less than 0.004 inches.

Signed _____ Date _____