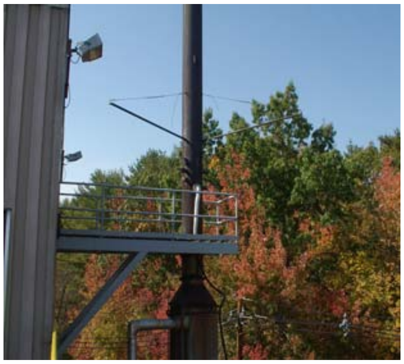
Prepared for:
Bostik, Inc. [U.S. EPA ID# MAD 001 039 767]
211 Boston Street
Middleton, MA



Trial Burn Plan / MACT CPT Plan for the Polyester Burner Unit – Revision 2

Volume II of III

ENSR Corporation
January 31, 2008
Document No.: 00963-044-200

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Volume II of III

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GLOSSARY OF TERMS AND ACRONYMNS

acfm	actual cubic feet per minute
Ag	silver
As	arsenic
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
AWFCO	automatic waste feed cut-off
Ba	barium
Be	beryllium
BIF	boiler(s) and industrial furnace(s)
CAA	Clean Air Act
CARB	California Air Resources Board
Cd	cadmium
CEMS	continuous emission monitoring system
cfh	cubic feet per hour
Cl_2	chlorine (gas)
CMS	continuous monitoring system
CO	carbon monoxide
CO_2	carbon dioxide
CPT	comprehensive performance test
Cr	chromium
CVAAS	cold vapor atomic absorption spectroscopy
DCS/DAS	data control system / data acquisition system
DI	deionized (water)
DOT	Department of Transportation
DRE	destruction and removal efficiency
dscfm	dry standard cubic feet per minute
EPA	Environmental Protection Agency (U.S.)
GC/MS	gas chromatography/mass spectrometry
HAPs	hazardous air pollutants
HCl	hydrogen chloride (gas) or hydrochloric acid
Hg	mercury
HOCs	hazardous organic constituents
HRA	hourly rolling average
HRGC/HRMS	high resolution gas chromatography / high resolution mass spectrometry
HWC	hazardous waste combustor
ICAP	inductively coupled argon plasma
ICP-MS	inductively coupled plasma mass spectrometry
LCS	laboratory control sample
LDAR	leak detection and repair
LSC	laboratory services coordinator
MACT	maximum achievable control technology

MA DEP	Massachusetts Department of Environmental Protection
MCB	monochlorobenzene
MDL	method detection limit
MEK	methyl ethyl ketone
MOC	management of change
MSDS	material safety data sheet
MS/MSD	matrix spike / matrix spike duplicate
MTEC	maximum theoretical emission concentration
ND	non-detect
NDIR	non-dispersive infrared
O&M	operation and maintenance
OPL	operating parameter limit
O_2	oxygen
PAHs	polycyclic aromatic hydrocarbons
Pb	lead
PCDDs	polychlorinated dibenzo-p-dioxins
PCDFs	polychlorinated dibenzofurans
PET	performance evaluation test
PHA	process hazard analysis
PICs	products of incomplete combustion
P&ID	process and instrumentation diagram
PM	particulate matter
POHC	principal organic hazardous constituent
ppm(v)	part per million (volume basis)
psia	pounds per square inch absolute
QAO	quality assurance officer
QAPP	quality assurance project plan
QA/QC	quality assurance/quality control
RA	risk assessment or rolling average
RCRA	Resource Conservation and Recovery Act
RPD	relative percent difference
RRF	relative response factor
RSD	relative standard deviation
Sb	antimony
scfm	standard cubic feet per minute
SLRA	screening level risk assessment
S/N	signal-to-noise ratio
SOP	standard operating procedure
SPCCs	system performance check compounds
SSMP	startup, shutdown and malfunction plan
SVOCs	semivolatile organic compounds
SW	Struthers-Wells
TBP	trial burn plan
TICs	tentatively identified compounds

Tl	thallium
UDRI	University of Dayton Research Institute
VOA	volatile organic analysis
VOCs	volatile organic compounds
VOST	volatile organic sampling train
w.c.	water column

1.0 Introduction

1.1 Facility Overview

Bostik, Inc. operates a chemical manufacturing facility, located in Middleton, Massachusetts (U.S. EPA ID# MAD 001 039 767). A pumpable-liquid hazardous waste is generated from the plant's polyester and direct solvation resin manufacturing units. The hazardous waste is currently burned in a vertically-fired process heater, referred to as the polyester burner unit. This unit meets the United States Environmental Protection Agency (U.S. EPA) definition of a boiler in 40 CFR 260.10. General facility information is provided below:

Owner:	Bostik, Inc.
Address:	211 Boston Street Middleton, MA 01949-2128
Telephone Number:	(978) 777-0100
U.S. EPA ID #.	MAD 001 039 767
Facility Contact:	Mr. Dan Welch, Health Safety Environment & Quality Manager Phone No.: (978)-750-7402 e-mail: dan.welch@bostik-us.com

The major business of the Bostik facility is the manufacture of polyester resins for adhesive applications. The Bostik plant manufactures polyester resins in two manufacturing units at the facility; the Polyester and Direct Solvation departments. A waste byproduct, known as polyester distillate, is produced during the manufacture of these resins. This waste byproduct is hard-piped to four (4) distillate storage tanks designated as DT-1, T-9, T-1 and T-2.

A single liquid hazardous waste stream that is generated onsite is burned in the polyester burner unit. This unit provides energy for the thermal requirements associated with the polyester resin manufacturing processes (i.e., to preheat the reactor feed material). The polyester burner unit is a Struthers-Wells (SW) vertically-fired process heater. The unit burns natural gas as a start-up fuel and Resource Conservation and Recovery Act (RCRA) hazardous waste as the primary fuel. Because the unit burns RCRA hazardous waste, it is regulated by 40 CFR Part 63, Subpart EEE: National Emission Standards for Hazardous Air Pollutants from Hazardous Waste Combustors.

1.2 Regulatory Background and Compliance History

Bostik became subject to 40 CFR Part, 266 Subpart H, commonly referred to as the boiler and industrial furnace (BIF) Rule, when the regulation was promulgated in 1991. Since that time, Bostik has operated under the interim status provisions of the rule and has performed tri-annual compliance recertifications ever since in compliance with 40 CFR 266.103(d). The unit currently operates under the January 2003 Recertification of Compliance that is based on an Adjusted Tier 1 scenario for metals and chlorine provided in 40 CFR 266.106(e) and 107(e).

This Trial Burn Plan (TBP) is being submitted to U.S. EPA Region 1 as part of a RCRA Part B Permit Application. Bostik originally submitted a complete Part B permit application and TBP in July 1995. That application was never acted upon and EPA subsequently requested an updated permit application in a letter dated May 31, 2006. The “call-in” letter was received by Bostik on June 6, 2006 and the deadline for submittal was 120 days from receipt. EPA subsequently granted an extension for overall document submission until December 8, 2006. The three-volume document (Revision 0) was initially submitted on December 8, 2006. A revised document (Revision 1 dated June 12, 2007) was submitted in response to general comments provided by EPA via e-mail and conference call on April 12, 2007. The current revision (Revision 2) is being submitted in response to comments from EPA dated November 28, 2007.

The regulated hazardous waste combustor (HWC) at the plant operates in accordance with the Recertification of Compliance that was submitted to U.S. EPA Region 1 in January 2003. As described in the 2003 Recertification of Compliance, the unit currently operates under the Adjusted Tier 1 scenario for metals and hydrogen chloride/chlorine (HCl/Cl_2), as provided in 40 CFR 266.106(e) and 107(e). Previous compliance activities have therefore been limited to a demonstration that the unit complies with the carbon monoxide (CO) emission standard found in 40 CFR 266.104(b) and the particulate matter (PM) emission standard outlined in 40 CFR 266.105, while establishing a maximum hazardous waste feed rate and demonstrating that the metal and chlorine feed rates comply with the Adjusted Tier 1 limits.

Due to the fact that Bostik would be going through the process of submitting a TBP and conducting a trial burn in the near future, EPA Region 1 granted a waiver / time extension for the subsequent Recertification of Compliance that would have been due by January 2006.

With respect to a Title V Operating Permit under the Clean Air Act (CAA), as previously discussed during the March 2, 2006 meeting at EPA Region 1, Bostik anticipates the need to obtain a CAA Title V Operating Permit from the Massachusetts Department of Environmental Protection (MA DEP) as noted at 40 CFR 63.1200(a)(2). The schedule for the expected request from MA DEP and the subsequent submittal has not been specified at this time.

With regard to compliance with the maximum achievable control technology (MACT) regulations (Subpart EEE) promulgated on October 12, 2005 (see Section 1.3 below), Bostik has completed the preliminary notifications required by this rule. A Notice of Applicability was sent to EPA on April 10, 2006. Notice of a Public Meeting to address both the new MACT rule and the Part B renewal process was posted in the printed and broadcast media on May 4, 2007. The public meeting was held on June 7, 2007 and the final notice of intent to comply (NIC) was submitted to EPA on June 14, 2007.

1.3 Applicable Performance Standards

1.3.1 RCRA Performance Standards

As stated above, Bostik currently operates under the interim status provisions of RCRA and is in the process of updating the Part B application and negotiating a final RCRA permit. Performance standards that are currently in effect under interim status are identified below:

- Particulate matter emission standard [40 CFR 266.105] – 0.08 gr/dscf corrected to 7% oxygen
- Carbon monoxide emission standard [40 CFR 266.104(b)(1)] – 100 ppm corrected to 7% oxygen

1.3.2 MACT Performance Standards

The MACT rule for hazardous waste combustors (HWCs) promulgated on October 12, 2005, was effective on December 12, 2005 and has a compliance date of October 14, 2008. Bostik fully intends to comply with these regulations and plans to conduct a combined RCRA trial burn and MACT comprehensive performance test (CPT) to demonstrate compliance with all applicable standards and performance criteria. Applicable MACT performance standards are noted below:

- Mercury emission standard [63.1217(a)(2)(i)] – 19 µg/m^3 corrected to 7% oxygen
- Semivolatile metals (SVM - cadmium and lead) emission standard [63.1217(a)(3)(i)] – 150 µg/m^3 corrected to 7% oxygen
- Low volatile metals (LVM – chromium only) emission standard [63.1217(a)(4)(i)] – 370 µg/m^3 corrected to 7% oxygen
- Carbon monoxide emission standard [63.1217(a)(5)(i)] – 100 ppm corrected to 7% oxygen
- Total hydrogen chloride and chlorine emission standard [63.1217(a)(6)(i)] – 31 ppm corrected to 7% oxygen
- Particulate matter emission standard [63.1217(a)(7)] – 80 mg/m^3 corrected to 7% oxygen
- Destruction and removal efficiency [63.1217(c)] – 99.99%

1.4 MACT Rule Integration

1.4.1 Pathways and Options Selected

As outlined under 40 CFR 266.100(b)(3), existing facilities are no longer subject to the standards identified under Part 266 once they have demonstrated compliance with the MACT requirements of Part 63, Subpart EEE, by conducting a CPT and submitting to the Administrator a Notification of Compliance (NOC) under 63.1207(j) and 63.1210(d) documenting compliance with the requirements of Part 63, Subpart EEE. In addition, 63.1217(f) provides an option for area sources (defined under 63.2) to choose either the RCRA standards for non-mercury metals, hydrogen chloride/chlorine and particulate matter or the MACT standards delineated under 63.1217. As an area source for hazardous air pollutants (HAPs), Bostik is electing to comply with the MACT standards for all pollutants.

Additionally, Bostik plans to conduct its initial MACT CPT prior to the October 14, 2008 compliance date and, therefore, the provisions of 63.1207(j)(5) require that an NOC be postmarked within 90 days of completion of the performance test or by the compliance date, whichever is later. Upon postmark of the NOC, Bostik will comply with the operating parameter limits (OPLs) identified in that document. Under this “early compliance” scenario, submittal of a documentation of compliance (DOC) is not required.

Bostik also intends to follow the provisions of 63.1207(m)(2) pursuant to a waiver of performance testing for mercury, SVM, LVM and hydrogen chloride/chlorine by complying with a 12-hr rolling average maximum theoretical emission concentration (MTEC) for each of these parameters. MTEC-related calculations for all parameters are shown in **Table 1-1**. The equations used in Table 1-1 are delineated in the text following this table.

It is also noted that as an operator of a liquid fuel boiler, Bostik is not subject to a numerical dioxin/furan emission standard under 63.1217. Nevertheless, MACT requires a one-time test for dioxins/furans as stipulated at 63.1207(b)(3). Because Bostik’s HWC is not equipped with a dry particulate removal device, they can elect to comply with either the carbon monoxide (CO) or hydrocarbon (HC) emission standard outlined under 63.1217(a)(5). Bostik is electing to comply with the CO standard as listed previously in Section 1.3.2.

Table 1–1 MTEC Calculations for Bostik's Polyester Burner Unit

Parameter	Units	MACT Standard *	MTEC
Semivolatile Metals (SVM) (Cadmium & Lead)	µg/m³	150.0	149.0
Low Volatile Metals (LVM) (Chromium only)	µg/m³	370.0	162.6
Mercury	µg/m³	19.0	13.5
Total Chlorine (Assume 100% as HCl)	ppm v/v	31.0	▨
	µg/m³	47,146	40,638

Assumptions:

Minimum Stack Gas Flowrate	**1,285**	dscfm
	1,639	wet scfm
Stack Gas Moisture Content	**21.6**	%
Maximum Waste Feed Rate	**652**	lb/hr

Maximum Expected Constituent Concentrations in Waste:

Cadmium	0.10	mg/kg
Lead	1.00	mg/kg
Arsenic	0.50	mg/kg
Beryllium	0.10	mg/kg
Chromium	1.20	mg/kg
Mercury	0.10	mg/kg
Total Chlorine	300	mg/kg
Ash	3,000	mg/kg

Corresponding Constituent Feed Rate Limits at MACT Standard:

Feed Constituent	Constituent Feed Rate (g/hr)	Constituent Feed Rate (g/sec)
Cadmium and Lead (SVM)	0.33	9.10E-05
Chromium (LVM)	0.81	2.24E-04
Mercury	0.041	1.15E-05
Total Chlorine	103	2.85E-02

*** All MACT standards are corrected to 7% oxygen**

(1) MTEC = [(MWFR) x (453.6 g/lb) x (MECC) x 35.314 ft^3/m^3)] / [(60 min/hr) x (MSGF)]

where,

MTEC = maximum theoretical emission concentration, $\mu g/m^3$

MWFR = maximum waste feed rate, lb/hr

MECC = maximum expected constituent concentration in waste feed, mg/kg

MSGF = minimum stack gas flowrate, dscfm

(2) CFRL (for metals) = [(MACT STD) x (MSGF) x (60 min/hr)] / [(35.314 ft^3/m^3) x (1.0 E 06 µg/g)]

where,

CFRL = constituent feed rate limit, g/hr

MACT STD = applicable standard, $\mu g/m^3$

MSGF = minimum stack gas flowrate, dscfm

(3) CFRL (for HCl/Cl_2) = [(MACT STD) x (MSGF) x (36.5 lb/lb-mole) x (60 min/hr) x 453.6 g/lb)] / [(385.3 ft^3/lb-mole) x (1.0 E 06)]

where,

CFRL = constituent feed rate limit, g/hr

MACT STD = applicable standard, ppm

MSGF = minimum stack gas flowrate, dscfm

1.4.2 Notification of Compliance

As noted previously, Bostik plans to conduct its initial MACT CPT prior to the October 14, 2008 compliance date and submit the NOC prior to the compliance date. Examples of the types of information to be provided in the NOC are given in **Table 1-2**.

Table 1–2 Types of Information to be Presented in Bostik's NOC

Facility Information
Facility Name and Location: Bostik, Inc.- 211 Boston Street - Middleton, MA 01949
Contact: Dan Welch –(978)-750-7402 – dan.welch@bostik-us.com
Source Information
Title V Classification: Major Source (HWC MACT Default); Area Source (HAPs)
Affected Source: Polyester Burner Unit (Liquid fuel-fired boiler)
Air Pollution Control: None
Applicability
The Polyester Burner is a liquid fuel-fired boiler and is regulated under 40 CFR Part 63 Subpart EEE (HWC MACT)
Emission Standards
The applicable emission standards (listed in **Section 1.3.2** of this CPT Plan) for the Bostik facility are based on the limits outlined at 40 CFR 63.1217 for liquid fuel-fired boilers. All emission standards (except DRE) are corrected to 7% oxygen.
Compliance Demonstrations
Once the CPT has been completed, Bostik will summarize the test results and show that all emission standards were met and that all operating limits were satisfied.
Operating Parameter Limits (OPLs)
Once the CPT has been completed, Bostik will summarize the applicable OPLs that were established to verify ongoing compliance with the emission standards. The expected OPLs are identified in **Table 2-2** of this CPT Plan.
Automatic Waste Feed Cutoff (AWFCO) Limits
Once the CPT has been completed, Bostik will summarize the applicable AWFCO limits that will be operational to immediately and automatically cut off the hazardous waste feed when OPLs or emission standards are exceeded. The expected regulatory AWFCOs are identified in **Table 4-4** of this CPT Plan.
Residence Time
A discussion of the hazardous waste residence time is presented in **Section 4.3.5** of this CPT Plan.

Table 1-2 (continued)

Fugitive Emissions
Bostik complies with the provisions of 63.1206(c)(5)(A) relative to control of combustion system leaks by keeping the combustion chamber sealed and by performing daily inspections of the equipment.
Other MACT Operating Requirements
On or before the MACT compliance date of October 14, 2008, Bostik will prepare and operate under an **Operation and Maintenance Plan** as required by 40 CFR 63.6(e), 63.8(c) and 63.1206(c)(7).
On or before the MACT compliance date of October 14, 2008, Bostik will prepare and operate under a **Startup, Shutdown and Malfunction Plan** as required by 40 CFR 63.6(e)(3) and 63.1206(c)(2)(ii)(B)
On or before the MACT compliance date of October 14, 2008, Bostik will prepare and operate under a **CMS Performance Evaluation Plan** as required by 40 CFR 63.8(e).
On or before the MACT compliance date of October 14, 2008, Bostik will prepare and operate under a **Feed Stream Analysis Plan** as required by 40 CFR 63.1209(c)(2).
On or before the MACT compliance date of October 14, 2008, Bostik will develop and implement an **Operator Training and Certification Program** as required by 40 CFR 63.1206(c)(6).
Certification
Bostik, Inc. hereby certifies that: (i) All required CEMS and CMS are installed, calibrated and continuously operating in compliance with the requirements of Subpart EEE; (ii) Based on the results of the initial CPT conducted in **[Insert Date Here]**, the boiler is operating in compliance with the emission standards and operating requirements of 40 CFR Part 63 Subpart EEE; and (iii) The OPLs required by 40 CFR 63.1209 and specified in this NOC ensure compliance with the emission standards.
Signature:
Name: Mark Hufziger
Title: Transportation Division General Manager
Date:

1.4.3 Other MACT Requirements

1.4.3.1 CPT Plan

The requirements for a CPT Plan under MACT are outlined under 63.1207(f)(1). These requirements are summarized below with reference to where the particular item can be found within the body of this document.

- An analysis of each feed stream sent to the combustor [63.1207(f)(1)(i)] – See Section 3.0
- Identification of hazardous air pollutants (HAPs) expected to be present in the feed stream(s) [63.1207(f)(1)(ii)] – See Section 3.0
- Detailed engineering description of the HWC [63.1207(f)(1)(iii)] – See Section 4.0
- Detailed description of sampling and monitoring procedures [63.1207(f)(1)(iv)] – See Section 6.0 and Appendix A (QAPP)
- Quantities of waste to be burned and test schedule [63.1207(f)(1)(v)] – See Sections 5.3 and 5.4
- Detailed test protocol including range of waste feed rates [63.1207(f)(1)(vi)] – See Section 5.0
- Description of and planned operating conditions for any emission control equipment [63.1207(f)(1)(vii)] – Not applicable (no pollution control equipment)
- Procedures for rapidly stopping hazardous waste feed and controlling emissions in the event of equipment malfunction [63.1207(f)(1)(viii)] – See Section 4.6.4
- A determination of hazardous waste residence time [63.1207(f)(1)(ix)] – See Section 4.3.5
- Information pertaining to extrapolation of metal feed rate limits from test levels [63.1207(f)(1)(x)] – Not applicable (not seeking to extrapolate metal feed rates)
- Documentation of expected levels of regulated constituents in natural gas and process air feed streams if they are not continuously monitored [63.1207(f)(1)(xi)] – See Sections 3.1 and 3.3
- Documentation of duration of system conditioning time required to achieve steady-state operation [63.1207(f)(1)(xii)] – See Section 5.3.2
- Information pertaining to cement kilns with in-line raw mills [63.1207(f)(1)(xiii)] – Not applicable
- Information pertaining to cement kilns with dual stacks [63.1207(f)(1)(xiv)] – Not applicable
- Additional Information for facilities requesting to use Method 23 for PCDDs/PCDFs [63.1207(f)(1)(xv)] – Not applicable – previous request has been denied
- Documentation of compliance with Section 63.1207(m) if not required to conduct testing based on maximum theoretical emission concentration (MTEC) calculations [63.1207(f)(1)(xvi)] – See Section 1.4.1 and Table 1-1
- Documentation that a surrogate proposed for measuring gas flowrate adequately correlates [63.1207(f)(1)(xvii)] – Not applicable – previous request has been denied
- Request(s) for alternative monitoring under 63.1209(g)(1) [63.1207(f)(1)(xviii)] – Not applicable
- Documentation of the temperature measurement location in the combustion chamber [63.1207(f)(1)(xix)] – See Section 4.6.1 and Figure 4-5
- Documentation for sources equipped with activated carbon injection [63.1207(f)(1)(xx)] – Not applicable

- Documentation for sources equipped with a carbon bed system [63.1207(f)(1)(xxi)] – Not applicable
- Documentation for sources that feed a dioxin/furan inhibitor [63.1207(f)(1)(xxii)] – Not applicable
- Documentation for sources equipped with a wet scrubber [63.1207(f)(1)(xxiii)] – Not applicable
- Documentation for sources equipped with other types of particulate matter control devices [63.1207(f)(1)(xxiv)] – Not applicable
- Documentation for sources equipped with a dry scrubber [63.1207(f)(1)(xxv)] – Not applicable
- Procedure for handling non-detects when calculating feed rate limits [63.1207(f)(1)(xxvi)] – See Section 4.6.2

1.4.3.2 Startup, Shutdown Malfunction Plan

By October 14, 2008, Bostik will need to develop a Startup, Shutdown and Malfunction Plan (SSMP) in accordance with 63.6(e)(3) and 63.1206(c)((2)(ii)(B). The SSMP must describe, in detail, procedures for operating and maintaining the source during periods of startup, shutdown, and malfunction; and a program of corrective action for malfunctioning process and monitoring equipment used to comply with the relevant standard. This plan will be developed and placed in the operating record by the compliance deadline.

1.4.3.3 Operation and Maintenance Plan

Also by October 14, 2008, Bostik will need to develop an Operation and Maintenance Plan (O&M Plan) in accordance with 63.1206(c)((7). The O&M Plan must describe in detail procedures for operation, inspection, maintenance, and corrective measures for all components of the combustion system that could affect emissions of regulated hazardous air pollutants. The plan must prescribe how Bostik will operate and maintain the combustor in a manner consistent with good air pollution control practices for minimizing emissions at least to the levels achieved during the comprehensive performance test. This plan ensures compliance with the operation and maintenance requirements of 63.6(e) and minimizes emissions of pollutants, automatic waste feed cutoffs, and malfunctions. This plan will also be developed and placed in the operating record by the compliance deadline.

1.4.3.4 CMS QC Program Plan

On or before the MACT compliance date of October 14, 2008, Bostik will prepare and operate under a CMS Performance Evaluation Plan as required by 40 CFR 63.8(e). This document will be placed in the operating record and will provide detailed audit and calibration procedures for the HWC's continuous monitoring instrumentation.

1.4.3.5 Feed Stream Analysis Plan

As part of the Part B application, Bostik has updated and revised the RCRA waste analysis plan (WAP) such that it now incorporates all required elements of a MACT Feed Stream Analysis Plan (FSAP). This document was submitted to EPA as part of Revision 1 to the permit application, dated August 7, 2007.

1.4.3.6 Operator Training and Certification

On or before the MACT compliance date of October 14, 2008, Bostik will develop and implement an Operator Training and Certification Program as required by 40 CFR 63.1206(c)(6).

1.5 Test Program Overview

This document describes how Bostik intends to conduct performance testing for the regulated HWC unit at the Middleton, MA facility. Testing will be conducted to demonstrate that the regulated unit complies with all applicable performance and emission standards and to establish operating limits that will be used in an operating permit. In addition, this plan describes testing that will be conducted to generate information for use in a screening level human health risk assessment (SLRA), in accordance with recent U.S. EPA policy and guidance.

Bostik plans to conduct the performance test during the 2nd quarter of 2008 depending on the actual schedule for plan approval and test preparation.

Bostik is proposing to conduct the test program under a single test condition representative of normal operating conditions or conditions that would result in higher than normal emissions as specified at 63.1207(g). Four (4) sampling runs will be completed for all test parameters at this single test condition. **Table 1-3** provides an overview of the test parameters required for demonstrating compliance with the aforementioned performance standards.

Table 1–3 Overview of Performance Testing Requirements (Stack Gas Emissions)

Test Parameter	Sampling Method	Analytical Method(s)	Testing Required for:
Particulate Matter	EPA Method 26A	EPA Method 5	RCRA & MACT
PCDDs/PCDFs	EPA Method 0023A	EPA Method 8290	MACT
POHC DRE	EPA Method 0030	EPA Methods 5041A and 8260B	RCRA & MACT
THC	EPA Method 25A	EPA Method 25A	MACT
O_2 and CO_2	EPA Method 3A	EPA Method 3A	RCRA & MACT
CO	Facility CEM	Facility CEM	RCRA & MACT
Flow and Moisture	EPA Methods 2 & 4	EPA Methods 2 & 4	RCRA & MACT

The test program has been designed to demonstrate compliance with all of the performance standards outlined previously in Section 1.3. The test program will also serve to establish the OPLs specified by MACT to ensure compliance with these emission standards. A full summary and description of the OPLs required to be established and the expected limits is provided in **Section 2.0**.

As stated previously, this test plan has been developed to achieve multiple objectives including demonstration of compliance with applicable MACT and RCRA emission standards; establishment of operating limits that will be used in an operating permit; and gathering of critical emission data for a screening level risk assessment. This document addresses all of these areas. The primary objectives of the test program are listed below and explained in further detail in other sections of this document.

1. Define, evaluate, and propose the RCRA permit limits that Bostik will operate the Polyester Burner Unit under, while ensuring compliance with other regulations concurrently applicable to the unit (i.e., HWC MACT).
2. Define operating condition(s) to generate the data necessary to support the permit limits identified in item 1 above. In addition, incorporate data collection needs for a screening level human health risk

assessment in order to show that regulatory permit limits applicable to the Polyester Burner Unit are protective of human health and the environment.

3. Ensure that data collected as part of the test burn program is technically and legally defensible via development of appropriate data usability criteria established for the specific intended uses above (i.e., RCRA permit, risk assessment, HWC MACT NOC, and subsequent Title 5 permit).
4. Document all data and information from the burn program so that the RCRA permit may be issued in a timely manner and all supporting/complementary data and information can be used for the HWC MACT NOC requirements, and subsequent Title V permit.

1.6 Risk Assessment Data Requirements

As outlined in EPA's May 31, 2006 Part B call-in letter, *..."an assessment of the potential risk and/or health impacts from the emissions of the unit will be conducted. The assessment will include direct and indirect exposures pathways. The initial assessment will be based on the unit's trial burn results that include the results of the dioxin/furan test. This assessment will aid in the determination on whether a more detailed site-specific risk assessment will be required."...*

Testing for key risk parameters will be performed as summarized here and as further described in Section 5.1.2 of this Plan. This emission information will then be used to conduct a human health SLRA that conforms to current guidance. The screening level risk assessment protocol has been submitted under separate cover as Volume III of III of this permit application. **Table 1-4** provides an overview of the testing to be performed to gather the necessary information for input to the SLRA.

As noted previously in Section 1.4.1, Bostik intends to pursue the waiver of testing for mercury, SVM, LVM and hydrogen chloride/chlorine as allowed under 63.1207(m)(2). Nevertheless, the test program will incorporate emission testing for HCl / Cl_2 and metals (see Table 1-4 for full list) so that actual data can be used in the SLRA. It is expected that stack gas detection limits (or measured values) will be significantly lower than the equivalent feed rates computed with waste feed detection limits owing to the differences in analytical detection limits due to matrix effects.

Table 1–4 Overview of SLRA Testing Requirements (Stack Gas Emissions)

Test Parameter	Sampling Method	Analytical Method(s)	Comments
Particulate Matter	EPA Method 26A	EPA Method 5	
HCl and Cl_2	EPA Method 26A	EPA Method 26A	
Mercury	EPA Method 29	EPA Method 7470A	
Other Metals **(a)**	EPA Method 29	EPA Methods 29 & 6020	
PCDDs/PCDFs	EPA Method 0023A	EPA Method 8290	
PAHs	EPA Method 0010	CARB Method 429	
SVOCs	EPA Method 0010	EPA Method 8270C	Full Method Scan + Top 10 TICs
VOCs	EPA Method 0030	EPA Methods 5041A and 8260B	Full Method Scan + Top 10 TICs
THC	EPA Method 25A	EPA Method 25A	
CO	Facility CEM	Facility CEM	
O_2 and CO_2	EPA Method 3A	EPA Method 3A	

(a) Other metals include: antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver and thallium.

1.7 Document Organization

This Plan is organized to provide the information required in 40 CFR Part 270 and 40 CFR 63.1207(f)(1) and follows the example outline for a combined RCRA and MACT Test Burn Plan provided by the state of Louisiana. This section has presented an overview of the facility in terms of regulatory background, compliance history, applicable performance standards, MACT rule integration issues and overview of the planned test program. Section 2.0 provides a detailed discussion of the operating parameter limits (OPLs) that the HWC will operate under to ensure continuous compliance with applicable performance standards. Section 3.0 describes the chemical and physical characteristics for the hazardous and non-hazardous streams fed to the regulated unit. Section 4.0 provides a technical engineering description of the combustion unit and the auxiliary systems, including process monitoring instrumentation. Section 5.0 describes the test protocols, planned operating conditions and test schedule. Section 6.0 provides an overview of the waste liquid and stack gas sampling and analysis program and Section 7.0 provides a discussion of the report format for the program. Document appendices include the Quality Assurance Project Plan (Appendix A) and the Continuous Monitoring System Performance Evaluation Test Plan (Appendix B).

2.0 System Operating Parameter Limits

Based on the results of the CPT, Bostik will propose operating limits for the boiler in the NOC submitted in accordance with 40 CFR 63.1207(j)(1) that ensures continuous compliance with the applicable performance standards. Most of the parameters will be proposed from the operating and monitoring data demonstrated during the CPT. However, several limits will be based on regulatory guidance, manufacturer's recommendations and/or good operating practice. If the required performance objectives of the testing are achieved, the combustion system should be allowed to operate under the OPLs proposed in this section.

The expected permit limits / OPLs discussed below are based on the provisions of the HWC MACT regulations in 40 CFR 63 Subpart EEE. **Table 2-1** provides an overview of the specific OPLs required, the applicable regulatory citation and the MACT performance standard with which the OPL ensures compliance. **Table 2-2** provides a summary of the limits expected to result from the CPT along with the measurement basis and the manner in which the OPL limit will be determined from the test results.

Table 2–1 MACT Operating Parameter Matrix

Process Parameter	Regulatory Citation	Ensures Compliance with these MACT Performance Standards
Maximum Total (and Pumpable) Hazardous Waste Feed Rate	63.1209(j)(3) and 63.1209(k)(4)	DRE and PCDDs/PCDFs
Minimum Combustion Chamber Temperature	63.1209(j)(1) and 63.1209(k)(2)	DRE and PCDDs/PCDFs
Maximum Flue Gas Flowrate	63.1209(j)(2); 63.1209(k)(3); 63.1209(m)(2); 63.1209(n)(5) and 63.1209(o)(2)	DRE, PCDDs/PCDFs, PM, SVM, LVM and HCl/Cl_2
Minimum Flue Gas Flowrate	63.1207(m)(i)	SVM, LVM, Hg and HCl/Cl_2
Maximum Total Mercury Feed Rate	63.1209(l)(1)	Hg
Maximum Total Ash Feed Rate	63.1209(m)(3)	PM
Maximum Total SVM (Cd & Pb) Feed Rate	63.1209(n)(v)(A)	SVM
Maximum Total LVM (Cr only) Feed Rate	63.1209(n)(v)(B)	LVM
Maximum Total Chlorine Feed Rate	63.1209(n)(4) and 63.1209(o)(ii)(B)	SVM, LVM and HCl/Cl_2

Table 2–2 MACT Operating Parameter Limits

Process Parameter	Units	Avg. Period	How Limit Established	Expected Limit
Maximum Total (and Pumpable) Hazardous Waste Feed Rate	lb/hr	1-hr (HRA)	Avg. of max. HRAs for each run	652
Maximum Total (and Pumpable) Hazardous Waste Feed Rate	gal/hr	1-hr (HRA)	Avg. of max. HRAs for each run	85.0
Minimum Combustion Chamber Temperature	°F	1-hr (HRA)	Avg. of the test run averages	1,130
Maximum Flue Gas Flowrate	wet scfm	1-hr (HRA)	Avg. of max. HRAs for each run	2,400
Minimum Flue Gas Flowrate	wet scfm	1-hr (HRA)	MTEC Calculation	1,639
Maximum Total Mercury Feed Rate	g/hr	12-hr (RA)	MTEC Calculation	0.041
Maximum Total Ash Feed Rate	g/hr	12-hr (RA)	Avg. of the test run averages	300
Maximum Total SVM (Cd & Pb) Feed Rate	g/hr	12-hr (RA)	MTEC Calculation	0.33
Maximum Total LVM (Cr only) Feed Rate	g/hr	12-hr (RA)	MTEC Calculation	0.81
Maximum Total Chlorine Feed Rate	g/hr	12-hr (RA)	MTEC Calculation	103

2.1 Control Parameters

Bostik plans to operate under a number of operational control parameters, which will be maintained as an indication that the combustion system will continue to operate in compliance with all regulatory standards. To facilitate review, the control parameters are grouped into the following categories:

- **CPT demonstrated** parameter limits are established from test operating data, and are used to ensure that boiler operating conditions are not significantly less rigorous than those demonstrated during the test. Most of these parameters are continuously monitored and recorded and are interlocked with the automatic waste feed cutoff (AWFCO) system. During the CPT, continuously monitored and interlocked parameters will be operational, but will be set at values which will allow the desired operating limits to be demonstrated.
- **Regulatory** parameter limits are regulatory specified limits, and are not based on the test operating conditions, e.g., the maximum stack CO concentration. These parameters are continuously monitored and recorded, and are interlocked with the AWFCO system. Interlocks for continuously monitored parameters will be operational during the test periods, without modification to the interlock set points.
- **Recommended** parameter limits are based on manufacturer's recommendations, operational safety, and good operating practice considerations rather than on the test operating conditions. These parameter limits may also be regulatory specified limits. Some of these parameters may be continuously monitored and recorded, and may be interlocked with the AWFCO system. Interlocks for continuously monitored parameters will be operational during the test periods, but will be set at values which will allow the desired operating limits to be demonstrated.

The specifics of the proposed limits are presented in proposed in following sections.

2.2 Establishment of Operating Parameter Limits

The permit limits for each of the control parameters will be established as specified in the HWC MACT regulations given in 40 CFR 63.1209. The following sections describe how each control parameter limit will be established.

2.2.1 Parameters Demonstrated During the CPT

2.2.1.1 Minimum Combustion Chamber Temperature [40 CFR 63.1209(j(1), (k)(2)]

The minimum combustion chamber operating limit is established for maintaining compliance with the DRE and dioxin/furan emission standards. The CPT will serve to establish the minimum combustion chamber temperature during the single test condition proposed. Combustion gas temperature will be monitored on a continuous basis. Based on successful demonstration of DRE and acceptable dioxin/furan emissions during the test, the minimum combustion temperature limit for the combustor will be established as an hourly rolling average (HRA) equal to the average of the test run average values.

2.2.1.2 Maximum Flue Gas Flowrate [40 CFR 63.1209(j(2), (k)(3), (m)(2), (n)(5), (o)(2)]

The maximum flue gas flowrate operating limit is established for maintaining compliance with the DRE, dioxin/furan, SVM, LVM, PM, and HCl/Cl_2 emission standards. Flue gas flowrate will be maximized during the single test condition. Maximum flue gas flow rate is to be established as an appropriate surrogate for gas residence time in the combustion chamber. Based on successful demonstration of DRE and acceptable emissions of other parameters, the maximum flue gas flowrate will be established from the average of the maximum HRAs observed during the CPT.

2.2.1.3 Maximum Total Hazardous Waste Feed Rate [40 CFR 63.1209(j(3), (k)(4)]

The maximum total hazardous waste feed rate operating limit is established for maintaining compliance with the DRE and dioxin/furan emission standards. Since Bostik feeds only a single hazardous waste liquid stream to the combustor, total hazardous waste feed rate and total pumpable hazardous waste feed rate are the same. Based on successful demonstration of DRE and acceptable emissions of dioxins/furans, the limit will be established as an HRA limit from the average of the maximum HRAs demonstrated during the CPT.

2.2.1.4 Maximum Ash Feed Rate [40 CFR 63.1209(m)(3)]

The maximum ash feed rate operating limit is established for maintaining compliance with the PM emission standard. Bostik will feed waste normally treated in the combustor during the CPT. This waste stream typically contains non-detectable levels of native ash. The total ash feed rate limit will be expressed as a 12-hour rolling average (RA), equal to the average of the test run averages observed during the CPT.

2.2.1.5 Maximum Total Chlorine and Chloride Feed Rate [40 CFR 63.1209(n)(4), (o)(ii)(B)]

The maximum total chlorine/chloride feed rate operating limit is established to maintain compliance with the SVM, LVM, and HCl/Cl_2 emission standards. Because the waste normally treated in the combustor typically contains non-detectable levels of native chlorine/chloride, Bostik plans to comply with the MTEC provisions outlined under 63.1207(m). Under this scenario, a feed rate limit is back-calculated from the emission standard and Bostik sets a limit on the minimum stack gas flowrate and maximum total chlorine/chloride feed rate that ensures compliance. (The calculations were presented previously in Table 1-1.) The calculated total chloride feed rate limit will be expressed as a 12-hour RA.

2.2.1.6 Maximum Mercury Feed Rate [40 CFR 63.1209(l)(1)]

The maximum mercury feed rate operating limit is established to maintain compliance with the mercury emission standard. Because the waste normally treated in the combustor typically contains non-detectable levels of native mercury, Bostik plans to comply with the MTEC provisions outlined under 63.1207(m). Under this scenario, a feed rate limit is back-calculated from the emission standard and Bostik sets a limit on the minimum stack gas flowrate and maximum total mercury feed rate that ensures compliance. (The calculations were presented previously in Table 1-1.) The calculated total mercury feed rate limit will be expressed as a 12-hour RA.

2.2.1.7 Maximum Total SVM Feed Rate [40 CFR 63.1209(n)(2)(v)(A)]

The maximum semivolatile metal (SVM) (lead + cadmium) feed rate operating limit is established for maintaining compliance with the SVM emission standard. Because the waste normally treated in the combustor typically contains non-detectable levels of native cadmium and lead, Bostik plans to comply with the MTEC provisions outlined under 63.1207(m). Under this scenario, a feed rate limit is back-calculated from the emission standard and Bostik sets a limit on the minimum stack gas flowrate and maximum total mercury feed rate that ensures compliance. (The calculations were presented previously in Table 1-1.) The calculated total SVM feed rate limit will be expressed as a 12-hour RA.

2.2.1.8 Maximum Total LVM Feed Rate [40 CFR 63.1209(n)(2)(v)(B)]

The maximum low volatile metal (LVM) (chromium only) feed rate operating limit is established for maintaining compliance with the LVM emission standard. Because the waste normally treated in the combustor typically contains non-detectable levels of native chromium, Bostik plans to comply with the MTEC provisions outlined under 63.1207(m). Under this scenario, a feed rate limit is back-calculated from the emission standard and Bostik sets a limit on the minimum stack gas flowrate and maximum total mercury feed rate that ensures compliance. (The calculations were presented previously in Table 1-1.) The calculated total LVM feed rate limit will be expressed as a 12-hour RA.

2.2.1.9 Maximum Pumpable LVM Feed Rate [40 CFR 63.1209(n)(2)(vi)]

Since all hazardous waste fed to the combustor is pumpable, a separate limitation on maximum pumpable LVM feed rate is not necessary.

2.2.2 Parameters Established by Regulatory Requirements

2.2.2.1 Maximum Stack Gas CO Concentration [40 CFR 63.1203(b)(5)(i)]

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

2.2.3 Parameters Established by Manufacturer's Recommendations, Operational Safety and/or Good Operating Practice

2.2.3.1 Fugitive Emissions Control [40 CFR 63.1206(c)(5)(i)(A), 63.1209(p)]

Bostik's combustion system is a sealed system operating at positive pressure. Daily inspections are performed to ensure that fugitive emissions do not occur. Corrective actions taken in such an event will be fully described in the SSMP to be developed prior to October 14, 2008.

2.2.3.2 Operation of Waste Firing System [40 CFR 63.1209(j)(4)]

This regulation stipulates that facilities should specify operating limits to ensure that good operation of the firing system is maintained. There are presently no manufacturer-recommended values for either a maximum viscosity limit for the as-fired waste or a minimum required atomization pressure for the waste distillate. In the absence of such specific values, the HWC operators manually adjust the air-to-fuel ratio to respond to minor fluctuations in the waste characteristics that are encountered in day-to-day operations.

3.0 Description of Feed Streams

This section describes the three streams fed to the polyester burner unit at the Bostik facility. The feed streams include a natural gas primary fuel, a supplemental liquid hazardous waste fuel, and process vent gases. The HOCs listed in 40 CFR Part 261 Appendix VIII, the hazardous air pollutants (HAPs) listed in Section 112(b) and non-hazardous constituents expected in the hazardous waste feed stream are identified later in this section. Storage and delivery of the feed streams to the HWC unit are described in Section 4.0.

3.1 Primary Fossil Fuel

Natural gas is the primary fossil fuel used to fire the HWC unit. The natural gas is obtained from Keyspan (transport and distribution) and Sprague (bulk supplier) and has a nominal heating value of 1,100 Btu/scf. Natural gas can be used as the sole fuel stream (for startup for example) or it can be co-fired with the single hazardous waste feed stream.

No sampling or analysis of the natural gas has been conducted. Given the source and normal composition of natural gas, Bostik believes that the natural gas supplied to the process heater does not contain detectable levels of metals, chlorine, or ash. Efforts to obtain more definitive information from the natural gas supplier were unsuccessful.

3.2 Supplemental Hazardous Waste Fuel

The polyester burner unit burns a single pumpable liquid hazardous waste stream generated from the Polyester and Direct Solvation resin manufacturing units at the facility. The condensed waste byproduct from the two resin plants is sent to the distillate storage tanks designated as T-1 and T-2.

The waste is categorized as a characteristic hazardous waste by ignitability because it exhibits a flash point of less than 140°F (D001). An overall summary of analytical data for physical parameters and metal constituents from the January 2003 compliance recertification and recent (November 2006) waste analyses is provided in **Table 3-1**. The recovered hydrocarbon material has a heat value of approximately 6,500 Btu/lb and generally low or non-detectable levels of chlorine and toxic metals as determined by analytical procedures specified in SW-846.

The hazardous waste to be fed during the test program will be the waste that is normally generated and treated in the polyester burner unit. Historically, the polyester distillate has consisted of various organics as summarized in **Table 3-2**.

On the basis of process knowledge and waste analysis, the only HOCs listed in 40 CFR Part 261, Appendix VIII that can be expected to be present in the hazardous waste are MEK and toluene. The remaining nonmetal compounds listed in 40 CFR Part 261, Appendix VIII could not reasonably be expected to be present in the hazardous waste, since they are not identified in the raw material specifications and material safety data sheets (MSDS); they are not identified on the product quality specifications; they are not expected to be formed during the production process; and they have not been detected in prior waste analyses.

Also on the basis of process knowledge and waste analysis, the only HAPs listed in Section 112(b) of the Clean Air Act Amendments of 1990 that can be expected to be present in the hazardous waste are methanol, xylene, ethylene glycol and toluene.

Table 3–1 Representative Analysis for Distillate Waste Stream

Analytical Parameters	Units	Expected Range
METALS --		
Antimony	mg/kg	2 - 10
Arsenic	mg/kg	0 - 0.1
Barium	mg/kg	0 - 0.2
Beryllium	mg/kg	0 - 0.06
Cadmium	mg/kg	0 - 0.03
Chromium	mg/kg	0 - 1.5
Lead	mg/kg	0 - 0.1
Mercury	mg/kg	0 - 0.04
Silver	mg/kg	0 - 0.03
Thallium	mg/kg	0 - 0.4
PHYSICAL PARAMETERS --		
Chlorine	mg/kg	200 - 400
Ash Content	% (wt)	0.1 - 0.6
Heat Content	Btu/lb	5,300 - 7,500
Density	g/cc	0.90 - 1.00

C:\PROJECTS\BOSTIK\CY 2006\Part B Renewal\[Feed Stream Data.xls]Part B Smry

Note: Data from January 2003 compliance recertification and November 2006 waste analyses.

Table 3–2 Major Constituents Typically Expected in the Distillate Waste Stream

Component	**Polyester Distillate (%)**	**RCRA Part 261 App VIII?**	**NESHAPs Sect. 112(b) HAP?**
Methanol	10 - 40	No	**Yes**
Water	30 - 50	No	No
Xylene	0 - 5	No	**Yes**
Butanediol	4 - 10	No	No
Diethylene Glycol	1 - 3	No	No
Tetrahydrofuran	5 - 15	No	No
Ethylene Glycol	0 - 20	No	**Yes**
Dimethyl terephthalate	0 - 2	No	No
Hexanediol	1 - 3	No	No
Ethyl Acetate	0 - 5	No	No
Methyl Ethyl Ketone	0 - 5	**Yes**	No
Toluene	0 - 5	**Yes**	**Yes**

3.3 Vent Gases

In 2006, the polyester burner unit was retrofitted to enable the burning of process vapors from the various manufacturing batch reactors and process equipment. During the preliminary engineering design phase of this project (conducted by ENSR), a test program was conducted to quantify organic emissions for target compounds in the vent gas header. The target compounds for the vent header test included both RCRA constituents and MACT-regulated HAPs expected to be present based on process knowledge and also capable of being detected using online, continuous gas chromatography / thermal conductivity detector (GC/TCD). The majority of the vapor-phase material discharged to the vent gas stream fed to the HWC unit is nitrogen, as all process vessels are nitrogen-blanketed. **Table 3-3** presents a summary of the constituent concentrations observed during this preliminary engineering test.

Table 3–3 Expected Composition of Vent Gas Stream

Target Analyte	**Overall Test Results, ppm(v/v)**		
	Average	Minimum	Maximum
Methyl Ethyl Ketone	321	110	1,432
Tetrahydrofuran	9,712	5,599	38,160
Cyclohexane	8.3	< 5.0	50
Methyl Cyclohexane	< 5.0	< 5.0	< 5.0
Toluene	11	< 5.0	23
m- & p-Xylene	66	47	93
Methanol	10,970	7,141	22,131
o-Xylene	16	7.0	22
Ethyl Acetate	< 5.0	< 5.0	< 5.0
Hexane	< 5.0	< 5.0	< 5.0
Unknowns (as Hexane)	7.5	5.4	14

C:\PROJECTS\BOSTIK\CY 2006\Part B Renewal\[Feed Stream Data.xls]Vent Gas

Note: Data from testing conducted on April 11, 2006

4.0 Engineering Description of the HWC Unit

This section provides a technical description of the regulated unit and the associated feed tanks, delivery systems, auxiliary equipment, exhaust stack, automatic waste feed cut-off (AWFCO) system, and process instrumentation associated with the combustion of hazardous waste. A detailed process flow diagram of the overall waste distillate storage and transfer system (including both liquids and vapors) is provided in **Figure 4-1**.

4.1 Waste Storage System

There are four (4) aboveground tanks that are used to store and transfer the waste distillate material. These tanks are designated DT-1, T-9, T-1 and T-2. As described below, only T-1 and T-2 are used to feed waste directly to the HWC.

The waste distillate generated by the polyester department is collected in the "Day Tank" (DT-1), a 950-gallon tank located under the vacuum pump room in Building 39. When the Day Tank's level reaches a predetermined point (75% capacity), the pump and bottom outlet valve are automatically turned on and the tank's contents are pumped to either T-1 or T-2. Should the level in the tank continue to rise above the pump out point, an alarm signal is sent to the data acquisition system and an operator must then take action. DT-1 is included in the Part B permit application for purposes of RCRA permitting.

Tank T-9 is a 10,000 gallon stainless steel tank located outside Building 9 which contains waste distillate generated by the direct solvation department. The tank is constructed in accordance with ASME pressure vessel code, Section VIII, Division I. The tank is a horizontal tank with dimensions of 18-ft long x 10-ft O.D. and has dished heads rated for atmospheric service. The tank is fabricated from 304 stainless steel with the walls and heads both $^3/_{16}$ inch thick. The level of waste in T-9 is tracked on the computer, but operators must take manual action to pump waste from Tank T-9 to either T-1 or T-2. Tank T-9 is also included in the Part B permit application for purposes of RCRA permitting.

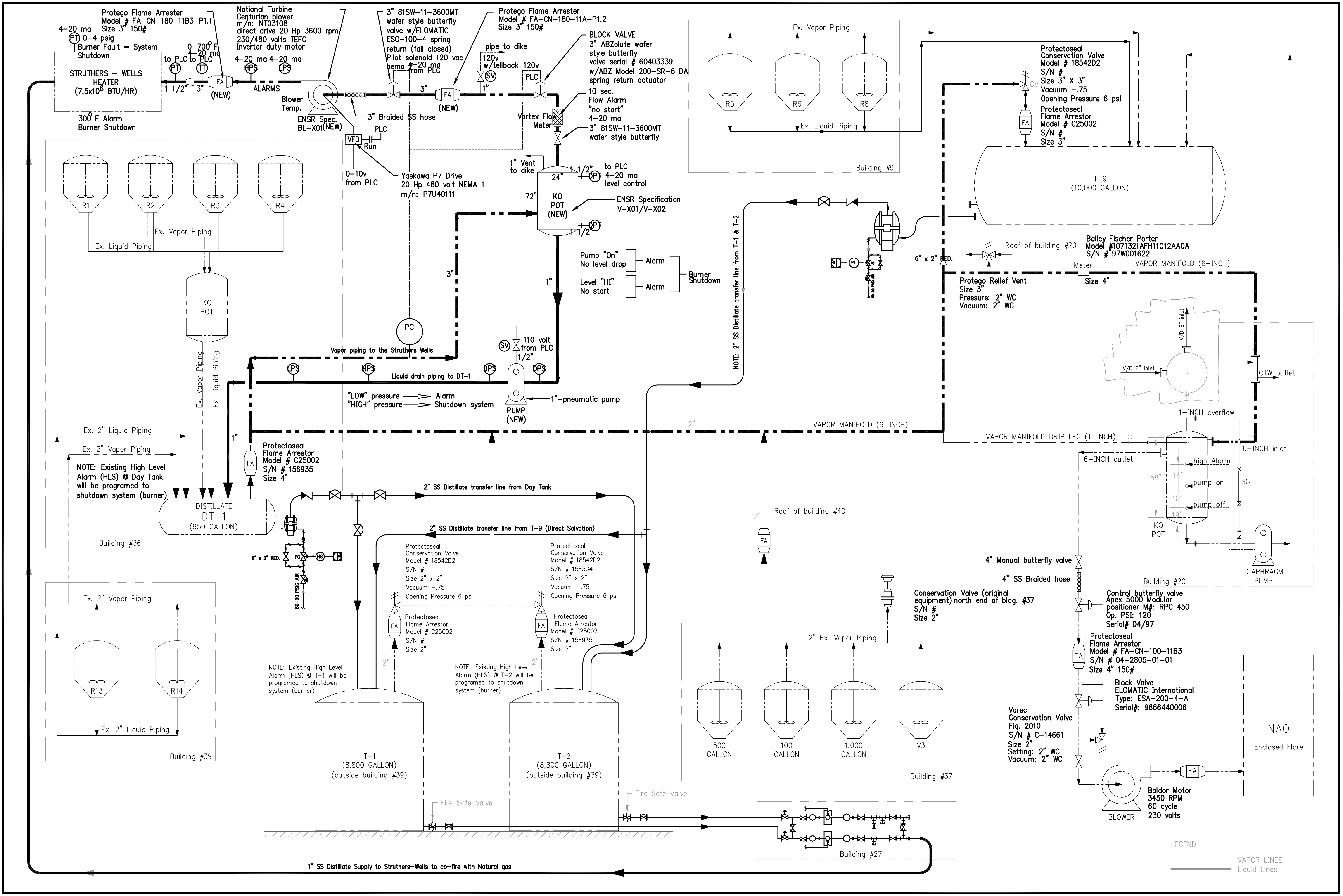
Tanks T-1 and T-2 are identical, vertical 8,800-gallon mild carbon steel tanks located adjacent to Building 27, which contain all waste distillate for feeding directly to the HWC unit. Both are designed in accordance with U.L. 142 Standard except that they have flat roofs. The tanks are rated for 1 psi. Both are fabricated from carbon steel with top, bottom, and walls at $^5/_{16}$ inch thick. Each tank has dimensions of 15-ft high x 10-ft O.D. and wall thickness of $^5/_{16}$ inches. Both tanks are included in the Part B permit application for purposes of RCRA permitting.

Detailed process and instrumentation diagrams (P&IDs) for all of these tank systems are provided in **Figures 4-2, 4-3 and 4-4**.

4.2 Feed Stream Delivery Systems

4.2.1 Waste Distillate Feed System Description

A single waste byproduct stream known as polyester distillate generated by the resin manufacturing process is continuously fed to several storage tanks as shown in Figure 4-1 and discussed above in Section 4.1. Waste distillate from the polyester resin department is piped to the 950-gallon day tank designated as DT-1. From there, the material is sent to the 8,800-gallon storage tanks designated as T-1 and T-2. Waste distillate from the direct solvation resin department is piped to the 10,000-gallon day tank designated as T-9. From there, the material is also sent to the 8,800-gallon storage tanks designated as T-1 and T-2.



FILENAME: 00963-033-01B.DWG

NO.:	DESCRIPTION:	DATE:	BY:
#1	Revised 07/13/06....corrected vacuum design # from 75 to -.75		Bostik
			JCC
#2	Revised 09/26/06....updated text & equipment @ both existing TO & SW		Bostik
			JCC
#3	Revised 08/10/06....added dashed 2" SS distillate transfer line		Bostik
			JCC
#4	Added liquid lines in black...made vapor lines phantom line type, color blue	08/15/07 01/21/2008	Bostik JCC

DESIGNED BY: X
DRAWN BY: K.P.B.
CHECKED BY: D.R.
APPROVED BY: X

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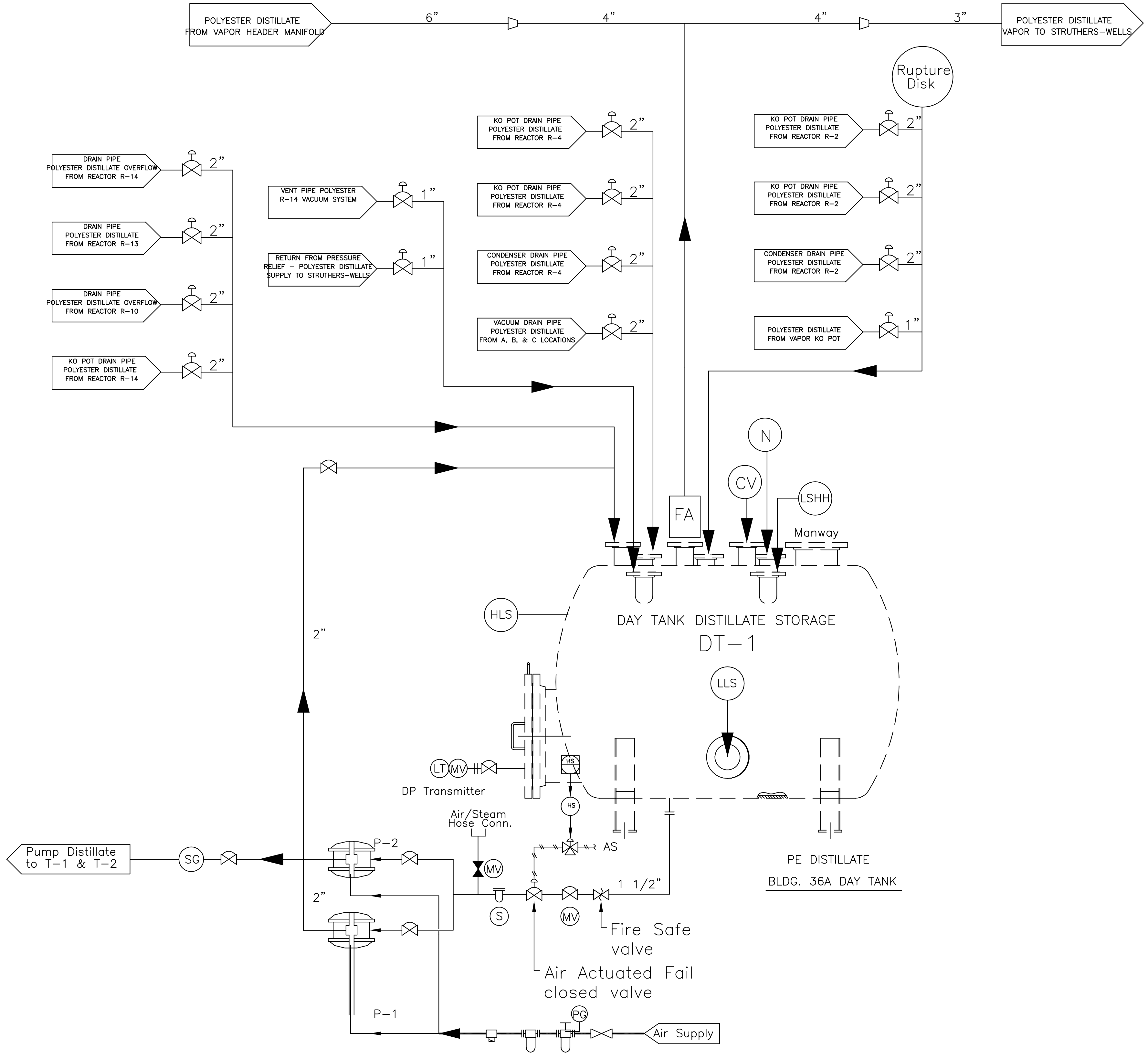
PROCESS FLOW DIAGRAM
BOSTIK, INC.
211 BOSTIK STREET
MIDDLETON, MA 01949

PROJECT NUMBER: 00963-033-0001
DATE: 5/06
SCALE: NONE

DRAWING NUMBER: 4-1

SHEET NUMBER: 1



P-1 & 2 TRANSFER PUMP

20 GPM, 50 FT. TDH
AIR OPERATED DOUBLE DIAPHRAM

REFERENCE:
BOSTIK, INC.
DISTILLATE STORAGE & TRANSFER, DWG. NO. F-27-045
DATED: 06/14/95

REVISIONS			
NO.:	DESCRIPTION:	DATE:	BY:
Rev. 1	Added designation DT-1 at title & Day Tank	1/21/2008	JCC
	Added Liquid Lines to Day Tank	1/21/2008	JCC

DESIGNED BY: X
DRAWN BY: K.P.B.
CHECKED BY: D.R.
APPROVED BY: X

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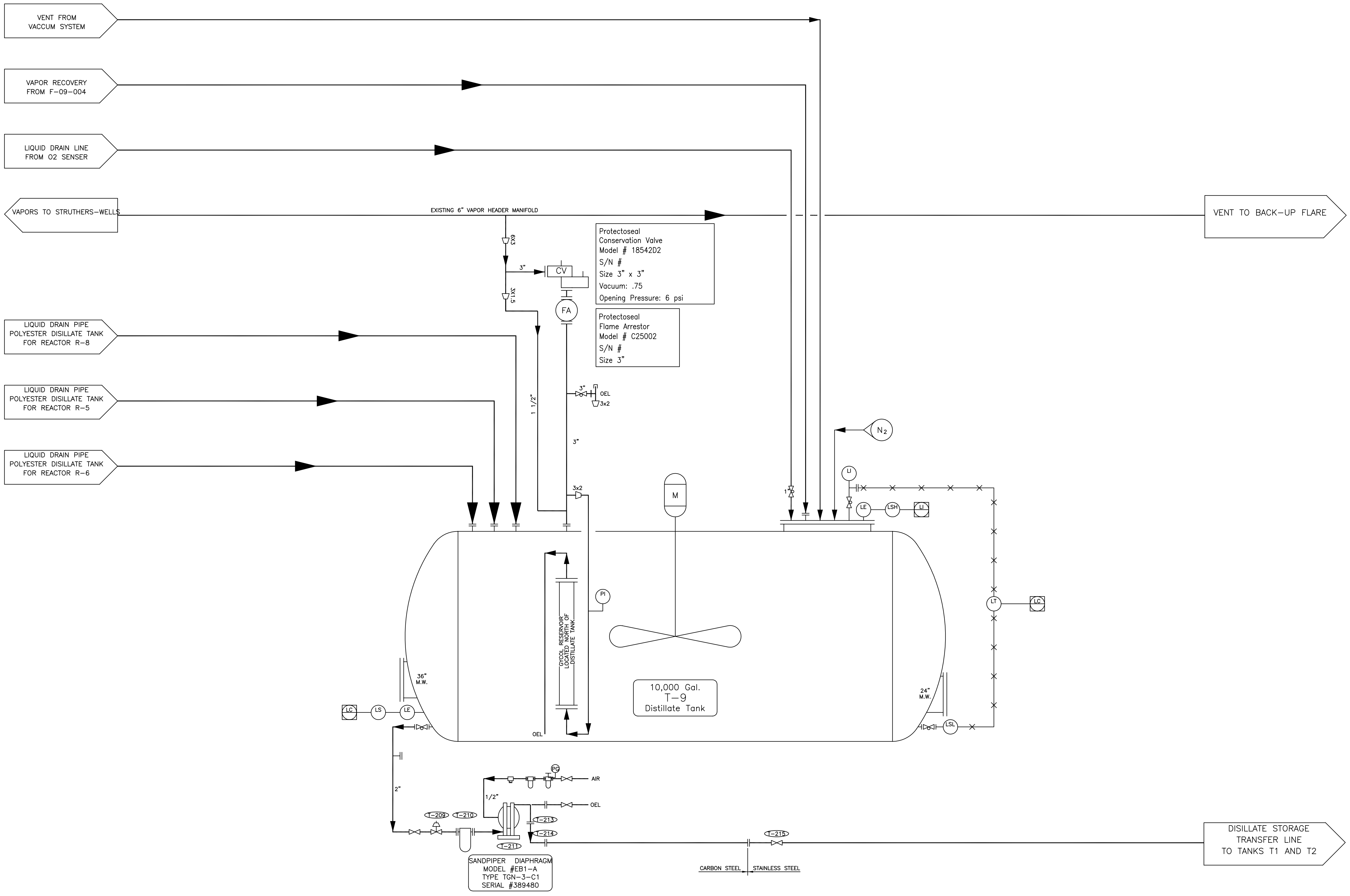
DAY TANK (DT-1) DISTILLATE STORAGE & TRANSFER
PROCESS FLOW DIAGRAM
BOSTIK, INC.
211 BOSTON STREET
MIDDLETON, MASSACHUSETTS

PROJECT NUMBER: 00963-035
DATE: 11/06
SCALE: NONE

FIGURE NUMBER:
4-2

SHEET NUMBER:
X



REFERENCE:
BOSTIK, INC.
F-09-001 DISTILLATE TANK.DWG
DATED: 04/2005

REVISIONS

NO.:	DESCRIPTION:	DATE:	BY:
REV. 1	Added T-9 designation for 10,000 gal Distillate Tank	1/21/2008	JCC

DESIGNED BY: X
DRAWN BY: K.P.B.
CHECKED BY: D.R.
APPROVED BY: X

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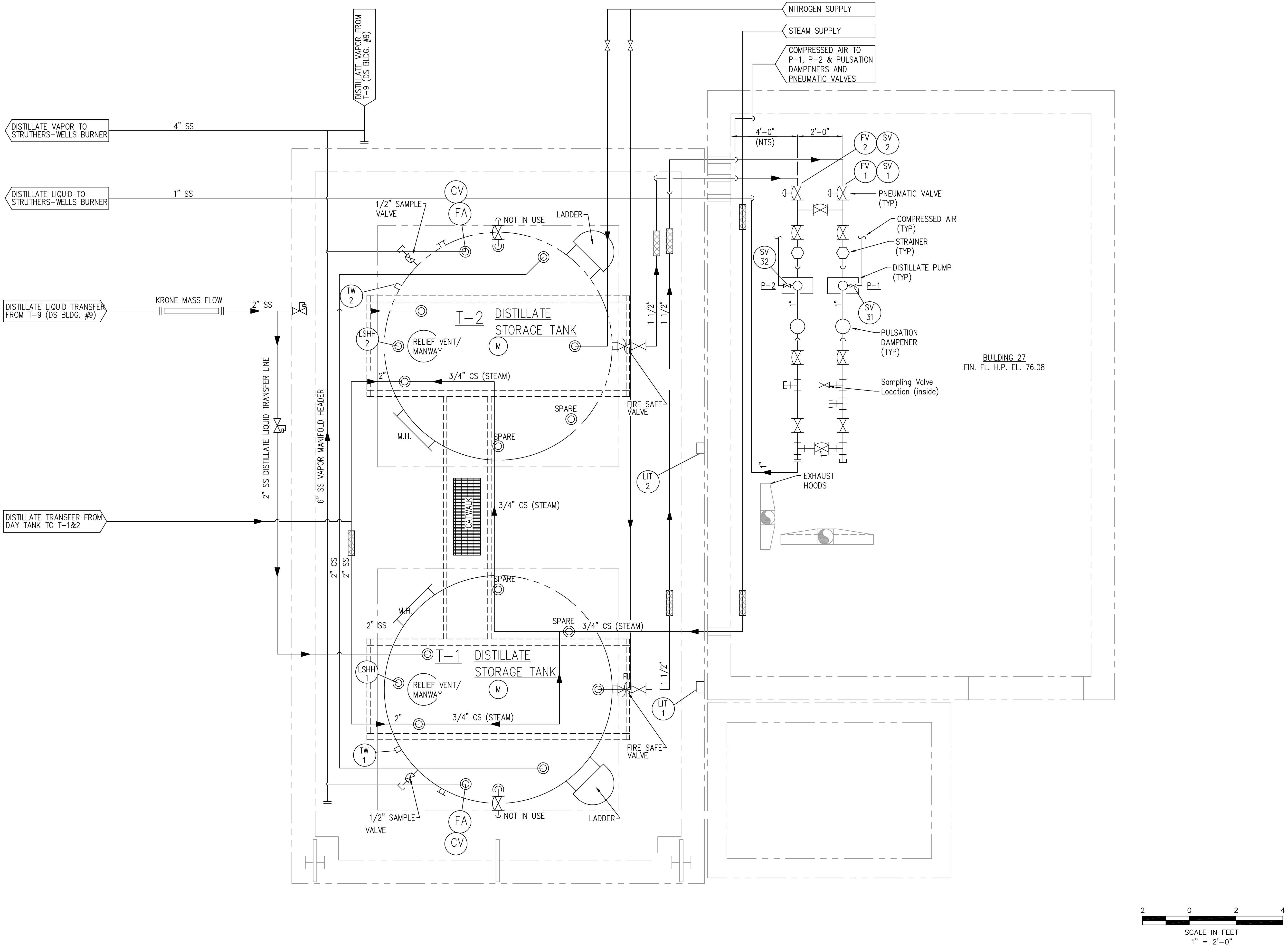
T-9 DISTILLATE TANK P&ID
BOSTIK, INC.
211 BOSTON STREET
MIDDLETON, MASSACHUSETTS

PROJECT NUMBER: 00963-035
DATE: 08/07
SCALE: NTS

FIGURE NUMBER:
4-3

SHEET NUMBER:
X



FILENAME: 00963-035-02D.DWG

NO.:	DESCRIPTION:	DATE:	BY:
1	REVISED AS PER FIELD CONDITIONS	8/14/07	JCC

REVISIONS

DESIGNED BY: X
DRAWN BY: K.P.B.
CHECKED BY: D.R.
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T-1, T-2 STORAGE TANKS
AND BLDG. 27 P&ID
BOSTIK, INC.
211 BOSTON STREET
MIDDLETON, MASSACHUSETTS

PROJECT NUMBER: 00963-035
DATE: 11/06
SCALE: 1"=2'

FIGURE NUMBER:
4-4

SHEET NUMBER:
1

4.2.2 Process Vapors Feed System Description

Process vapors from the various tanks and batch process reactors are collected in a 6-inch vapor header and routed to the HWC. The process vapor stream is fed into the combustion chamber directly above the liquid waste burner. These process vapors are also capable of being sent to an enclosed flare in case the HWC unit goes down for an extended period of time. Figure 4-1 (provided earlier) depicts the major pieces of equipment pertaining to the overall system including the process vapor collection system. Major equipment associated with the process vapor system includes:

- a pneumatic pump,
- a liquid / vapor separator tank (knock-out pot),
- a Vortex flow meter,
- two flame arrestors,
- a variable frequency drive air blower, and
- several valves and connecting lines.

During normal routine operations, the process vapors are routed continuously to the HWC through a series of valves as depicted on Figure 4-1. Should the polyester burner go into alarm or be shut down for any reason, then a series of steps are automatically initiated to start up the enclosed flare and route the vapors in the reverse direction. If the enclosed flare is unable to be started and the HWC remains shut down, then steps are initiated to shut down the batch reactors generating the process vapors.

4.2.3 Natural Gas Feed System Description

The primary fuel used in the polyester burner is natural gas with a nominal heating value of 1,100 Btu/scf. The natural gas is fed under pressure to the burner assembly for the combustion unit. Natural gas is metered to the heater by an independent, calibrated Vortex flow meter and is vertically upward-fired into the combustion zone.

4.3 Hazardous Waste Combustor

4.3.1 Description of the Combustion Unit

Bostik operates a single HWC unit at the Middleton, MA facility that heats a transfer fluid, which is then used to supply energy to the production processes. General design specifications for the polyester burner unit are provided in **Table 4-1**.

The polyester burner unit meets the EPA definition of a boiler in 40 CFR 260.10 in that:

- The combustion chamber and primary energy recovery section are of integral design:
- Thermal recovery efficiency is at least 60%; and
- At least 75% of the recovered energy is "exported" and utilized at the facility. No credit is taken for recovered heat used internally in the same unit.

In addition, the unit meets the definition of a hazardous waste combustor (specifically a hazardous waste liquid fuel boiler) identified under 63.1201.

Table 4–1 Design Specifications for the Polyester Burner Unit

Parameter	Value
Heater Manufacturer	Struthers-Wells
Heater Model Number	5C15-4
Shape	Cylindrical
Shell Diameter	5-ft
Shell Height	16-ft 6-in
Combustion Chamber Volume	324 cubic feet
Refractory Thickness	6 inches
Design Firing Rate	8.8 x 10^6 Btu/hr input
Design Heat Absorption	7.5 x 10^6 Btu/hr output
Burner Manufacturer	North American Mfg. Co.
Sootblowing	None
Air Pollution Control	None
Combustion Air Blower Manufacturer	North American Mfg. Co.
Combustion Air Blower Model No.	2420F-T15D
Combustion Air Blower Capacity	118,000 cfh
Stack Elevation Above Grade	64.5 feet
Stack Internal Diameter	24 inches

The combustor was manufactured by Struthers-Wells (SW) of Warren, PA in 1969 and is designated as Model No. 5C15-4. The unit is 5 ft in diameter by 64.5 ft high including the stack. The combustion chamber is 16 ft 6 inches long and has a volume of 324 ft^3. The original economizer was added in 1980 and was also built by SW. A second economizer (also made by SW) was added in July 1996.

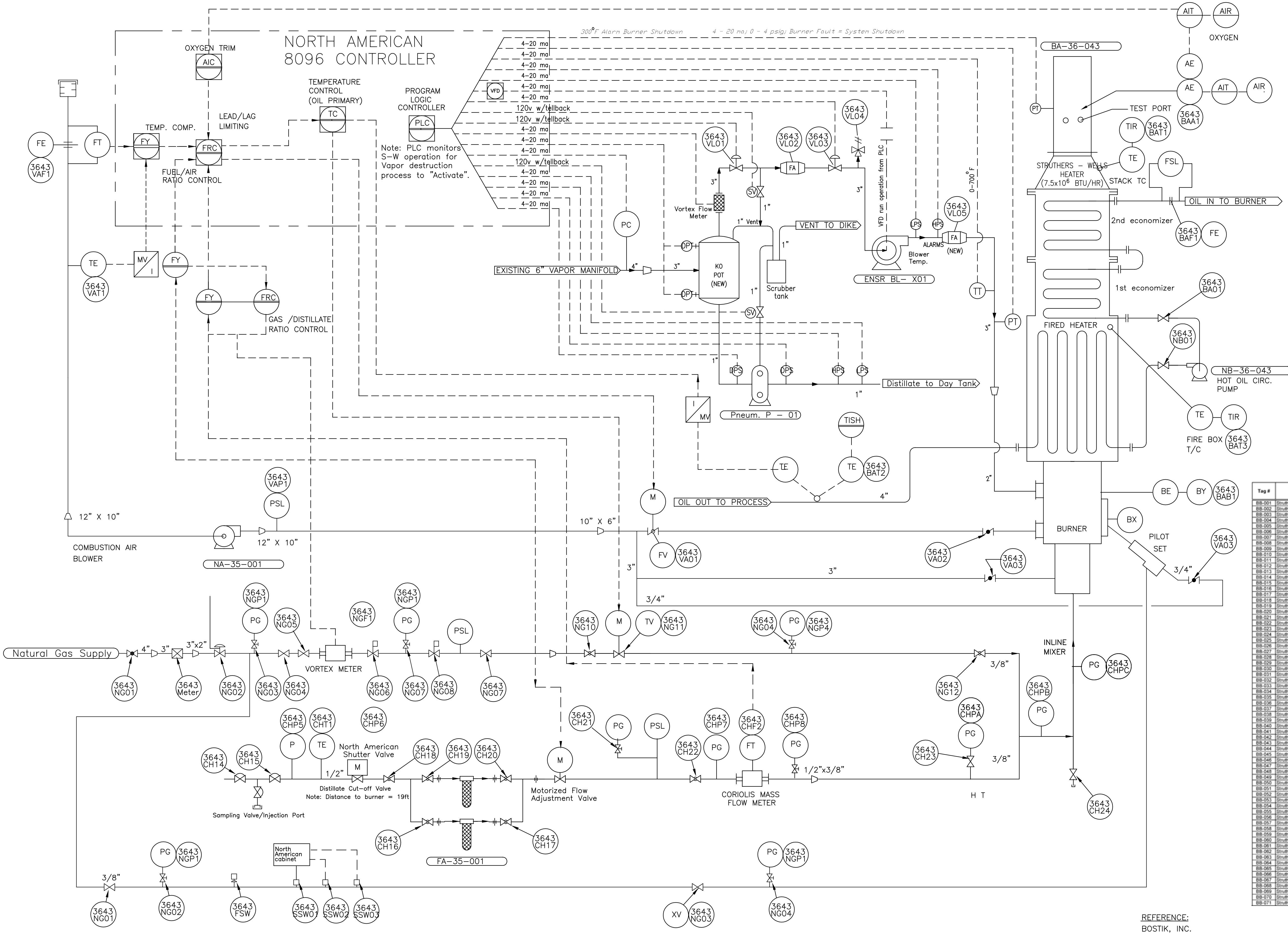
The unit has a maximum heat output of 7.5 x 10^6 Btu/hr and a maximum heat input of approximately 8.8 x 10^6 Btu/hr based on a thermal efficiency of 85%. Natural gas is used to supplement the heat supplied by the waste feed to maintain reactor temperature and for burner startup. Historically, the normal operating range for the unit has been 50-100 gph (300-970 lb/hr) waste distillate and 1,000-5,000 scfh natural gas. If operated on waste distillate alone, the unit could feed ~1,350 lb/hr (175 gph) waste distillate and if operated on natural gas alone, the unit could feed ~ 8,000 scfh. A detailed P&ID for the HWC system is provided in **Figure 4-5**.

The heat generated in the boiler is transferred by radiation and convection to the heat transfer fluid which flows through the economizers and then enters the combustor at the bottom, passes through a series of tubes and exits to the manufacturing process.

The burner unit is constructed of commercial steel according to American Society of Mechanical Engineers (ASME) Code Section I. The shell is lined with 6" of refractory. The fan, stack, flue gas piping, and combustion air piping are carbon steel. The waste fuel piping to the heater is also carbon steel.

4.3.2 Nozzle Burner Design

The polyester burner fires either natural gas or a combination of natural gas and hazardous waste and is equipped with a burner by North American Manufacturing Company. The burner is located at the base of the heater and fires vertically upward into the combustion chamber. The burner is a dual fuel burner capable of firing both liquid waste and natural gas simultaneously.



Tag #	Location	Equipment Description	Size
BB-001	Struthers Wells Boiler	Tee	1/2"
BB-002	Struthers Wells Boiler	Elbow	1/2"
BB-003	Struthers Wells Boiler	Pressure Gage (30 psi)	3/8"
BB-004	Struthers Wells Boiler	Static Mixing Tube	1/2"
BB-005	Struthers Wells Boiler	Tee	3/4"
BB-006	Struthers Wells Boiler	Ball Valve	1/2"
BB-007	Struthers Wells Boiler	Elbow	3/4"
BB-008	Struthers Wells Boiler	Union	3/4"
BB-009	Struthers Wells Boiler	Check Valve	3/4"
BB-010	Struthers Wells Boiler	Ball Valve	3/4"
BB-011	Struthers Wells Boiler	Tee	3/4"
BB-012	Struthers Wells Boiler	Ball Valve	3/4"
BB-013	Struthers Wells Boiler	Elbow	3/4"
BB-014	Struthers Wells Boiler	Elbow	3/4"
BB-015	Struthers Wells Boiler	Elbow	3/4"
BB-016	Struthers Wells Boiler	Flange on Flow Meter	1 1/4"
BB-017	Struthers Wells Boiler	Flange on Flow Meter	1 1/4"
BB-018	Struthers Wells Boiler	Elbow	3/4"
BB-019	Struthers Wells Boiler	Union	3/4"
BB-020	Struthers Wells Boiler	Ball Valve	3/4"
BB-021	Struthers Wells Boiler	Reducer	2" to 3/4"
BB-022	Struthers Wells Boiler	Ball Valve	3/4"
BB-023	Struthers Wells Boiler	Tee	3/4"
BB-024	Struthers Wells Boiler	Ball Valve	3/4"
BB-025	Struthers Wells Boiler	Elbow	3/4"
BB-026	Struthers Wells Boiler	Ball Valve	3/8"
BB-027	Struthers Wells Boiler	Elbow	3/8"
BB-028	Struthers Wells Boiler	Pressure Gage (60 psi)	3/8"
BB-029	Struthers Wells Boiler	Reducer	3/4" to 1/2"
BB-030	Struthers Wells Boiler	Motorized Flow Adjustment Valve	1/2"
BB-031	Struthers Wells Boiler	Tee	1/2"
BB-032	Struthers Wells Boiler	Ball Valve	3/8"
BB-033	Struthers Wells Boiler	Elbow	3/8"
BB-034	Struthers Wells Boiler	Pressure Gage (100 psi)	3/8"
BB-035	Struthers Wells Boiler	Tee	1/2"
BB-036	Struthers Wells Boiler	End Cap	1/2"
BB-037	Struthers Wells Boiler	Check Valve	1/2"
BB-038	Struthers Wells Boiler	Union	1/2"
BB-039	Struthers Wells Boiler	Tee	1/2"
BB-040	Struthers Wells Boiler	Ball Valve	1/2"
BB-041	Struthers Wells Boiler	Quick Disconnect Air Chuck	3/8"
BB-042	Struthers Wells Boiler	Tee	1/2"
BB-043	Struthers Wells Boiler	Elbow	3/8"
BB-044	Struthers Wells Boiler	Pressure Gage	3/8"
BB-045	Struthers Wells Boiler	Ball Valve	1/2"
BB-046	Struthers Wells Boiler	Motorized Valve	1/2"
BB-047	Struthers Wells Boiler	Tee	1/2"
BB-048	Struthers Wells Boiler	Tee	3/8"
BB-049	Struthers Wells Boiler	Pressure Switch	3/8"
BB-050	Struthers Wells Boiler	Elbow	1/2"
BB-051	Struthers Wells Boiler	Elbow	3/8"
BB-052	Struthers Wells Boiler	Globe Vavlve	3/8"
BB-053	Struthers Wells Boiler	Pressure Gage	3/8"
BB-054	Struthers Wells Boiler	Elbow	1/2"
BB-055	Struthers Wells Boiler	Union	1/2"
BB-056	Struthers Wells Boiler	Ball Valve	1/2"
BB-057	Struthers Wells Boiler	Elbow	1/2"
BB-058	Struthers Wells Boiler	Tee	1/2"
BB-059	Struthers Wells Boiler	Elbow	1/2"
BB-060	Struthers Wells Boiler	Ball Valve	1/2"
BB-061	Struthers Wells Boiler	Cam Lock End Cape	1"
BB-062	Struthers Wells Boiler	Elbow	1/2"
BB-063	Struthers Wells Boiler	Reducer	1" to 3/4"
BB-064	Struthers Wells Boiler	Union	1"
BB-065	Struthers Wells Boiler	Tee	1"
BB-066	Struthers Wells Boiler	Temperture Probe	3/4"
BB-067	Struthers Wells Boiler	Tee	1"
BB-068	Struthers Wells Boiler	Pressure Gage	3/8"
BB-069	Struthers Wells Boiler	Globe Valve (Sealed Stem)	1"
BB-070	Struthers Wells Boiler	Globe Valve (Sealed Stem)	1"
BB-071	Struthers Wells Boiler	Globe Valve (Sealed Stem)	1"

REFERENCE:
BOSTIK, INC.
DATED: 10/04/06 REV. 1 BURNER SYSTEM, DWG. NO. F-36-043

REVISIONS

NO.:	DESCRIPTION:	DATE:	BY:
REV. 1	Distillate line control update & leak detection schedule	1/21/2007	JCC

DESIGNED BY: X
DRAWN BY: K.P.B.
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BOSTIK, INC.
211 BOSTON STREET
MIDDLETON, MASSACHUSETTS

PROJECT NUMBER: 00963-035
DATE: 11/06
SCALE: NONE

FIGURE NUMBER: 4-5

SHEET NUMBER: X

FILENAME: 00963-035-12D.DWG

Waste distillate is fed to the combustor using a throttling flow control system. The flow of distillate is measured by a Coriolis-type Micro-Motion mass flow meter. Natural gas is fed to the system through a separate piping arrangement and is measured by a Vortex meter.

4.3.3 Combustion Air Flow

A forced draft, centrifugal fan is used to provide the motive force to push combustion gases through the combustion system. The installed fan was manufactured by North American Manufacturing Company. The fan has a design capacity of 118,000 cubic feet per hour (cfh) and is powered by a 15 hp motor. To ensure safe and efficient boiler operation, the combustion air is typically kept above 25% of the maximum capacity. In addition, the air-to-fuel ratio is manually adjusted (based on experience) to respond to slight variations in waste distillate water content, methanol content and heat value.

4.3.4 Ash Handling System

The polyester burner does not use soot blowers or any type of ash handling system. The ash content in the distillate waste feed stream is so low that no post-combustion residues accumulate in the combustor. Therefore, any ash in the feed is emitted as particulate matter.

4.3.5 Hazardous Waste Residence Time

The Subpart EEE MACT regulations define hazardous waste residence time to mean *"the time elapsed from cutoff of the flow of hazardous waste into the combustor (including, for example, the time required for liquids to flow from the cutoff valve into the combustor) until solid, liquid, and gaseous materials from the hazardous waste (excluding residues that may adhere to combustion chamber surfaces and excluding waste-derived recycled materials such as cement kiln dust and internally recycled metals) exit the combustion chamber".* The hazardous waste residence time must be calculated and the calculation must be included in the operating record.

The polyester distillate waste stream is a liquid material that is vaporized upon injection into the combustion chamber and thus the calculation within the combustion chamber is based on gas-phase residence time. The distance from the cutoff valve to the burner is 19 ft. as shown on Figure 4-5. The time required for the liquid waste material to move from the cutoff valve to the burner is approximately 12.7 seconds at a waste flowrate of 85.0 gph.

The combustion chamber volume for the polyester burner is 324 ft^3. Data from the most recent compliance certification test (December 2002) indicates an average combustion chamber temperature of about 1,130°F and a stack gas flowrate of 4,651 actual cubic feet per minute (acfm) at 613°F. These numbers yield a gas-phase residence time within the combustion chamber of approximately 2.8 seconds. Thus, the total hazardous waste residence time is 15.5 seconds.

4.3.6 HWC Startup Procedures

During start-up, the combustion unit is initially run on natural gas until the minimum allowable temperature is reached. Then the waste feed is initiated and slowly increased to the desired feed rate, while reducing the natural gas feed rate until the desired ratio is reached. Continuous monitoring of the exhaust gas CO concentration ensures proper combustion during start-up.

4.3.7 Fugitive Emissions and Control

Fugitive emissions from the polyester burner are controlled by maintaining a sealed combustion chamber. The shell of the unit is inspected daily to ensure no leaks are present. Fugitive emissions may escape through valves, pumps, and sampling connections in the hazardous waste feed system piping. In the heater system, seals around the forced draft fan that provide combustion air to the combustion zone and ductwork downstream of the heater may also contribute to fugitive emissions. This equipment is subject to the plant's leak detection and repair (LDAR) program, which was implemented in accordance with 40 CFR Part 264, Subpart BB requirements. Applicable pumps, valves, compressors, pressure relief valves, sampling connections, and open-ended lines are monitored routinely and visually inspected for leaks.

4.4 Air Pollution Control System

The HWC is not equipped with an air pollution control system.

4.5 Exhaust Stack

Combustion gases exiting the burner are exhausted to the atmosphere through a steel stack. The exhaust stack has a 24-inch inside diameter and a gas discharge point at an elevation of 138 ft or approximately 64.5 feet above ground surface. The stack is equipped with 4 sampling ports located 19-ft upstream of the discharge point and 8.5-ft downstream of the upper economizer / expansion section. More detailed information on the exhaust stack dimensions, elevations and typical conditions (flow, temperature, etc.) are included in the QAPP (Appendix A). Please refer to Figure 4-1 in the QAPP.

4.6 Process Monitoring and Instrumentation

4.6.1 Continuous Monitoring System

Current regulations require continuous monitoring of several process parameters to verify that the unit is within interim status operating limits. In addition, other operating parameters are monitored to ensure proper and safe combustion conditions or to maintain desired production rates. The combustion system is controlled by a process control computer system that includes monitoring of the following parameters:

- Waste distillate feed rate;
- Natural gas feed rate;
- Combustion chamber temperature;
- Stack temperature; and
- Stack CO and O_2 concentrations.

Figures 4-1 through 4-5 (provided earlier) depict the location of all critical process instrumentation. In addition, **Table 4-2** provides key information concerning Tag IDs and the measurement basis for these parameters.

Micro-Motion mass flow meters measure the hazardous waste feed rate, and Vortex meters measure the natural gas and process vapor feed rates. The waste feed flow meter is located in the waste transfer line between the storage tanks and the boiler, upstream of the flow control valve and AWFCO valves. The natural gas flow meter is located in the natural gas feed line, upstream of the flow control valve and shutoff valve. The process vapor butterfly control valve is located upstream of the air blower and braided hose connection and downstream of the flame arrestor and Vortex flow meter.

Combustion chamber temperature is monitored by a thermocouple located at the top of the combustion chamber and below the first economizer. The location of the thermocouple is at elevation 94.3 ft and is depicted on Figure 4-5. The bulk gas temperature in the combustion chamber usually ranges from 1,100 to 1,400°F. An additional thermocouple located in the expansion section above the second economizer measures stack gas temperature.

Table 4–2 Process Instrumentation Overview

Parameter	Tag Name	Units	Measurement Basis (a)		
			Instant.	OMA	HRA
Waste Distillate Feed Rate (Input)	WFR	gal / hr		X	
Waste Distillate Feed Rate (Calc.)	WFRra	lb/hr			X
Natural Gas Feed Rate (Input)	NGFR	CCFHR (10^2 ft^3/hr)		X	
Natural Gas Feed Rate (Input)	NGR ra	CCFHR (10^2 ft^3/hr)			X
Heat Input Rate (Calc.)	HIR	10^6 Btu/hr		X	
Combustion Chamber Temperature	C TMP	°F		X	
Oxygen Concentration	O2	%		X	
Raw CO from Analyzer Low Range	CO Lo	ppm		X	
Raw CO from Analyzer High Range	CO Hi	ppm		X	
Raw CO Used to Generate Corrected Data	CO	ppm		X	
CO corrected to 7% O_2	COc7%	ppm		X	
CO corrected to 7% O_2	CO ra	ppm			X

(a) Instant. = Instantaneous; OMA = one-minute average; HRA = hourly rolling average

Stack CO and O_2 concentrations are monitored by TECO and Servomex analyzers, respectively. Probes for the analyzers are located in the exhaust stack. Further information on these instruments is provided in Section 4.6.3.

Under Subpart EEE, Bostik is required to submit a CMS performance evaluation test (PET) plan pursuant to 63.8(e)(4) and 63.1207(b)(1). The CMS PET Plan is included as **Appendix B** of this document.

4.6.2 Constituent Feed Rate Determination

Historically, Bostik has effectively “monitored” the feed rate of applicable parameters by knowing the concentrations of these parameters in the waste stream and limiting the actual waste feed rate such that the feed rate limits were not exceeded.

Under the MACT regulation as applied to Bostik, the feed rate limits for metals and total chlorine will be back-calculated from the applicable standard. This concept was discussed and presented in Section 1.0.

Even though many of the regulated constituents are typically measured as non-detect (ND) values, Bostik plans to use the full detection limit (reporting limit) in making any compliance determinations. The appropriate value would be entered into the computer system following each update of the measured concentrations.

4.6.3 Continuous Emissions Monitoring System

CO and O_2 monitors are currently installed in the exhaust stack of the polyester burner unit. **Table 4-3** provides specifications for the existing analyzers.

Table 4–3 Design Specifications for the Continuous Emissions Monitoring System

Parameter	CO Analyzer	O_Z Analyzer
Manufacturer	TECO Model No. 48H	Servomex Model No. 1400
Serial Number	55952-305	01421 / B-156
Range	0 - 200 ppmv (low range) 0 - 3,000 ppmv (high range)	0-25%
Accuracy	The greater of 10% of the performance test method or 10 ppmv	Incorporated into the CO relative accuracy calculation
Calibration Drift	< 6 ppmv (low range) < 90 ppmv (high range)	< 0.5%
Calibration Error	< 10 ppmv (low range) < 150 ppmv (high range)	< 0.5%
Response Time	< 2 minutes	< 2 minutes
Calibration Frequency	Daily	Daily

Stack gas is continuously drawn through a filter and heated sample transport line. The gas is conditioned to remove water and any condensate. The resulting dry gas flows into the CO and O_2 gas analyzers. Raw data from each analyzer is polled by the process control/data acquisition system every second. CO and O_2 readings correspond to dry conditions due to the gas conditioning system prior to the gas analyzer. The O_2 reading is used to correct the CO reading to 7% O_2, using the following equation:

$$CO\,Conc\,(ppmv@7\%O_2, dry) = CO\,Conc.(ppmv@\,stack\,O_2\;dry)\;x\,\frac{(21-7\%O_2)}{(21-stack\,O_2)}$$

From the O_2 corrected readings, a one-minute average CO concentration is calculated every minute. At each successive minute, the 60 most recent one-minute average CO concentrations are used to calculate an HRA CO concentration. The one-minute and HRA CO (O_2 corrected) and O_2 concentrations are automatically recorded by the process control/data acquisition system. If the HRA CO concentration exceeds 100 ppmv corrected to 7% O_2, then an AWFCO is initiated. The CEMS output and alarms are displayed remotely on the process computer system. As per the requirements of 63.1209(a)(3), one-minute average CO values that exceed the upper span limit for the analyzer (3,000 ppm) will be recorded as 10,000 ppm and used in the calculation of the HRA.

Zero and span calibrations are performed daily by an automatic calibration system. The calibration cycle takes approximately 20 minutes to complete including the O_2 analyzer and separate CO low and high ranges. Each analyzer is dosed with zero and span calibration gas standards. Calibration drift is calculated and compared to the analyzer's specifications. Verification of the calibration results are printed on a daily basis and stored in the CEM Control Room.

4.6.4 Automatic Waste Feed Cutoff System

- The HWC continuously operates with an AWFCO system to ensure compliance with all applicable operating and feed rate limits. For the Bostik unit, the AWFCO system triggers a waste feed cutoff whenever any of the following conditions exist:
- when an OPL is exceeded;
- when an emission standard monitored by a CEMS (i.e., CO) is exceeded;
- when the span value of any CMS detector (except a CEMS) is met or exceeded;
- upon malfunction of a CMS; and
- when any component of the AWFCO system fails.

Table 4-4 lists the AWFCO limits and set points that are expected to be operational once a successful test has been completed. The waste feed will automatically be shut off whenever one of the set points is exceeded. Each of these operating parameters will continue to be monitored during a cutoff event. The waste feed can be restarted only after each of the above AWFCO conditions is satisfied. The AWFCO system and any associated alarms are tested on a weekly basis to verify functionality.

Table 4–4 AWFCO Parameters and Expected Limits

Process Parameter	Units	Automatic Waste Feed Cutoff Limit	
		Regulatory	Non-Regulatory
Maximum Total (and Pumpable) Hazardous Waste Feed Rate	lb/hr	652	None
Minimum Combustion Chamber Temperature	°F	1,130	None
Maximum Flue Gas Flowrate	wet scfm	2,400	None
Minimum Flue Gas Flowrate	wet scfm	1,639	None
Low Combustion Air Pressure	in. w.c.	None	10 - 45
Low Heat Transfer Oil Pressure	psi	None	15
Low Pilot Gas Pressure	psi	None	8
High Pilot Gas Pressure	psi	None	27
Low Heat Transfer Oil Flow	gpm	None	200
Low Heat Transfer Oil Level	inches	None	2.0
High Heat Transfer Oil Temperature	°F	None	600
CO Concentration @ 7% O_2	ppm	100	None

Emergency shutdown of the burner can also be initiated manually or through an additional set of sensors and alarms. There is a manual emergency shutdown button in the North American 8096 controller panel which is wired to the PLC. Emergency buttons in several locations are also received by the PLC. These four buttons, located at the CEM Building, the Day Tank, T1 & T2, and Building 27, shutdown the entire system, including the polyester burner. Additionally, a number of other alarms and sensors are in place to monitor heater operations. Table 4-4 also indicates the set points for each of the alarm, shutdown, warning, and waste feed cutoff designations. These shutdown alarms are part of the overall burner management system and are designed to ensure proper and safe heater operation.

During the test burn, all current AWFCOs will remain operational to ensure that the testing will not present a hazard to human health or the environment. The only cut-off limit that is being re-established is the hazardous waste feed rate and that parameter will be set at a lower limit (~652 lb/hr) than the current value of 970 lb/hr.

4.6.5 Alternative Monitoring Request

Under 63.1209(g), facilities are allowed to submit an application to the Administrator for approval of alternative operating parameter monitoring requirements to document compliance with the emission standards. At this time, Bostik is not making any such request.

5.0 Test Program Operations

This section provides an overview of test program design, including required performance testing, planned testing for input to the SLRA, planned test conditions and operating scenarios, planned waste feed requirements, overall sampling strategy and anticipated test schedule.

5.1 Test Program Requirements

The test program has been designed to meet the applicable performance standards of both RCRA and MACT as previously described in **Section 1.3** of this plan. Additionally, the test program has been designed to gather critical emission measurement data to be used as input to the SLRA. A single boiler operating condition is being proposed to accommodate all required testing. This single waste feed operating condition will be representative of normal boiler operations at the facility and will be conducted using normally generated waste polyester distillate. Target operating conditions for the polyester burner are summarized below in **Table 5-1**.

Table 5–1 Target Operating Conditions and MACT OPLs

Process Parameter	Units	Normal Operating Range	Target Value for CPT
HWC Operating Parameters --			
Total (and Pumpable) Hazardous Waste Feed Rate	lb/hr	300 - 970	652
	gal/hr	50 - 100	85.0
Hazardous Waste as-fired Heat Content	Btu/lb	5,300 – 7,500	6,500
Natural Gas Feed Rate	scfh	1,000 – 5,000	2,500
Vent Gas Stream	cfm	50 - 500	350
System Heat Input Rate	10^6 Btu/hr	0 – 8.8	7.0
Boiler Operating Load	%	50 - 85	79
Combustion Chamber Temperature	°F	1,100–1,350	1,130
Flue Gas Flowrate	wet scfm	1,700 – 2,700	2,400
HWC Feed Constituents --			
Total Ash Feed Rate	g/hr	0 – 750	300
Total Chlorine/Chloride Feed Rate	g/hr	0 - 150	103
Total Mercury Feed Rate	g/hr	0 – 0.044	0.041
Total LVM (Chromium) Feed Rate	g/hr	0 – 0.85	0.81
Total SVM (Cadmium & Lead) Feed Rate	g/hr	0 – 0.50	0.33
Toluene (POHC) Feed Rate	lb/hr	0.4 – 7.0	4.0

Note: Values listed are targets and may vary by ± 20% during the actual test.

Detailed information on the sampling and analytical methods to be followed for the program along with other information related to the field test program procedures and analytical protocols is provided in **Section 6.0** (Sampling and Analytical Program) and **Appendix A** (Quality Assurance Project Plan).

5.1.1 Testing to Demonstrate Compliance with Performance Standards and Required OPLs

The test program will feature emission measurements for the following parameters to demonstrate compliance with applicable performance standards as previously listed in Section 1.3:

- particulate matter;
- carbon monoxide;
- PCDDs/PCDFs (one-time test requirement);
- destruction and removal efficiency (DRE) for a principal organic hazardous constituent (POHC); and
- total hydrocarbon (THC) emissions in conjunction with the DRE test noted above.

Testing for the above parameters will also serve to establish the following operating limits for the polyester burner unit:

- Maximum total hazardous waste feed rate;
- Maximum total pumpable hazardous waste feed (same as total hazardous waste feed);
- Maximum total ash feed rate;
- Minimum combustion chamber temperature; and
- Maximum flue gas flowrate.

Feed rate limits are also being established (by the MTEC mechanism) for mercury, SVM, LVM and total chlorides as discussed previously in Section 1.4.1 and below in Section 5.1.3. It is noted that the hazardous waste distillate feed stream is the only feed stream to the burner that is expected to contain any of the regulated metal or chloride constituents. Therefore, the total feed rate of these constituents in the hazardous waste fuel feed stream is equal to the total feed rate of these constituents in all feed streams.

5.1.2 Risk Assessment Testing

Recent EPA guidance (Risk Burn Guidance for Hazardous Waste Combustion Facilities, EPA530-R-01-001, July 2001) suggests that coordinated emission testing be conducted during trial burns and comprehensive performance tests for purposes of obtaining data to use in risk assessments. Gathering such additional emission measurement data for use in the SLRA is therefore an additional objective of the test program. A work plan for a screening-level human health risk assessment has previously been provided in Volume III of III of Bostik's updated Part B permit application.

EPA guidance also states that sampling to obtain results for use in a risk assessment can be conducted at worst-case operating conditions or at normal operating conditions. EPA considers worst-case conditions to be those that would tend to maximize emissions of a constituent, while normal conditions reflect those that are typically encountered during day-to-day operation. If testing is performed at normal conditions, then additional permit limits may be required to ensure that the combustion unit operates within the definition of those normal conditions over a long period of time.

Since the polyester burner typically operates only within a small window of hazardous waste feed rate and since the waste has low or non-detectable levels of metals and total chlorides, Bostik plans to conduct the risk-related measurements under the single operating condition which is representative of normal operations. For purposes of the SLRA, the following emission parameters will be evaluated during the single test condition:

- Particulate matter (there is too little particulate matter generated to be able to do any meaningful particle size analysis and therefore, total PM emissions will be allocated to various size categories as described in EPA guidance and the risk protocol);
- Carbon monoxide and total hydrocarbons;
- Target volatile organics (full method scan plus top 10 TICs);
- Target semivolatile organics (full method scan plus top 10 TICs);
- Polycyclic aromatic hydrocarbons (PAHs);
- Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDDs/PCDFs);
- Metals (arsenic, antimony, barium, beryllium, cadmium, total chromium, lead, mercury, nickel, silver and thallium); and
- Hydrogen chloride and chlorine.

5.1.3 Special Test Considerations

There are several components of the planned test program that can be considered as unique or special features for Bostik's initial CPT. These are discussed below:

- Since the waste distillate stream fed to the HWC has low or non-detectable levels of regulated metals and total chlorine/chlorides, Bostik intends to comply with the MTEC provisions outlined under 63.1207(m)(2) and as such testing for mercury, LVM, SVM and HCl/Cl_2 would not be required. However, testing for these parameters will be performed so as to provide actual emission data for the SLRA. While testing for these parameters will not be required for future MACT CPTs, the fact that testing is being performed for the initial CPT will allow demonstration of compliance with the MACT emission standards for these parameters.
- As noted previously in Section 1.4.1, Bostik is electing to comply with the CO standard (100 ppm corrected to 7% oxygen) in lieu of the hydrocarbon standard (10 ppm corrected to 7% oxygen) as specified under 63.1217(a)(5). Testing for hydrocarbons is being performed, however, as such a test must be performed in conjunction with the DRE test requirement.
- As a liquid fuel fired HWC boiler operator, Bostik is not subject to a numerical dioxin standard under 63.1217. However, 63.1207(b)(3) specifies that a one-time test be performed. Testing for PCDDs/PCDFs is being performed to satisfy the one-time test requirement and also to collect data for this parameter which is critical to the risk assessment.

5.1.4 Sampling Strategy

The overall testing strategy has been developed to satisfy test program objectives. The test matrix will provide the data needed to demonstrate compliance with applicable performance standards, to establish permitted operating conditions and to gather emission data vital to the risk assessment process.

The polyester burner unit is equipped with a stack sampling arrangement consisting of two ports at one elevation and two other ports at slightly different elevations. The two "isokinetic" ports are oriented at 90-degrees to each other. This somewhat limited sample port configuration prevents simultaneous operation of all sampling trains during the test program and requires that one full day will be needed for completion of each sampling run.

Since only a single test condition is being performed, four (4) separate sampling runs will be conducted for each emission parameter. Fuel feed rates and unit operating conditions will be maintained at steady state levels across all replicate-sampling runs, to the maximum extent possible. Since only 3 runs are required for compliance purposes, decisions regarding the use of the 4th run will need to made quickly and with the full support of EPA. The following criteria will be considered in determining how the 4th run may or may not be used:

- At the conclusion of each day of testing, a review of Bostik's process operating data will be conducted to verify that all parameters have been appropriately tracked and recorded. If the process data for any run are considered suspect or incomplete, then the 4th run will be used.
- If any of the sample bottles from either the waste distillate or the sampling trains are broken or otherwise rendered unusable during any of the individual runs, then the 4th run's samples will be used.
- If any of the samples are lost or broken during transit to the analytical laboratory, then the 4th run's samples will be used.
- Once all samples have been received by the lab, an assessment will be made as to the completeness of the test program. If all samples collected from the first 3 runs are intact and all process data has previously been determined to be valid, then the 4th run will not be used.
- Decisions regarding the need to analyze the 4th run will also need to be made quickly so as to not exceed any sample holding times.
- If the samples from the 4th run are analyzed, then data from all runs will be reported.

The length of each sampling run will be determined by both the need to collect sufficient sample volume to obtain adequate detection limits and the need to obtain sufficient operating data for the development of permit limits. Expected individual sample train run times are described more completely in Section 6.0 (Sampling and Analysis Program) and Appendix A (QAPP) of this document.

5.1.5 Dealing with Potential Process Interruptions

The system will be lined out on waste for one hour prior to test initiation. This will serve to establish all hourly rolling averages (HRAs) at or near their desired set points when sampling begins. If there is a waste feed interruption (i.e., AWFCO) during a sampling run, the following guidelines will be followed:

- Sampling will be stopped as quickly as possible after the interruption.
- If the interruption is less than 5 minutes, there will be no line out period, and testing will recommence as soon as possible.
- If the interruption is between 5 and 30 minutes, there will be a 15-minute line out period, and then sampling will recommence.
- If the interruption is between 30 and 60 minutes, there will be a 30-minute line out period and then sampling will recommence.

- If the interruption exceeds 60 minutes, there will be a one-hour line out period before testing is resumed.
- If the interruption lasts well in excess of 60 minutes and there is little hope of completing the day's run, then the run will be aborted and begun anew the following day.

5.2 Waste Feed Requirements

5.2.1 POHC for DRE Determination

5.2.1.1 Evaluation of Candidate POHCs

Chemical compounds that can be considered as possible POHCs for the Bostik test burn include:

- MEK (raw material present in the waste at 0-5% by weight),
- Toluene (raw material present in the waste at 0-5% by weight), and
- Monochlorobenzene (non-native compound).

An ideal POHC for a DRE test program would be a compound that is native to the waste stream, easily sampled and analyzed and considered difficult to incinerate. POHC incinerability is based on thermal stability, according to a method developed at the University of Dayton Research Institute (UDRI). (The current list is taken from the "Technical Implementation Document for EPA's Boiler and Industrial Furnace Regulations", EPA-530-R-92-011, March 1992.) This guidance is based on gas-phase thermal stability under oxygen-starved conditions. Compounds are ranked on the basis of the temperature required for 99 percent destruction at a residence time of two seconds. A total of 329 RCRA HOCs have been ranked by UDRI using this methodology. The breakdown of the various classes of compounds is as follows:

- Class 1 – total of 37 compounds (Ranking 1 – 37)
- Class 2 – total of 33 compounds (Ranking 38 – 70)
- Class 3 – total of 57 compounds (Ranking 71 – 127)
- Class 4 – total of 78 compounds (Ranking 128 – 205)
- Class 5 – total of 60 compounds (Ranking 206 – 265)
- Class 6 – total of 20 compounds (Ranking 266 – 285)
- Class 7 – total of 44 compounds (Ranking 286 – 329)

Compounds ranked high on the list (Class 1, 1-37) are considered the most difficult to incinerate. Current EPA interpretation of the use of this list assumes that the ability to successfully destroy a given compound directly implies the ability to destroy all other compounds in the same class, as well as those ranked lower.

Monochlorobenzene (MCB) is a Class 1 compound and thus, considered the most difficult to incinerate. However, MCB is not a native waste constituent and would, therefore, need to be artificially spiked (injected) into the waste stream. MCB is readily sampled by the volatile organic sampling train (VOST) methodology (EPA Method 0030). A drawback to using this compound, however, would be that it would introduce chlorine to the system where none is currently present.

Toluene is the highest ranked compound in Class 2 (Rank = 38). Toluene is also native to Bostik's waste stream, albeit at varying concentrations (0-5% by weight) depending on the specific adhesive products being produced. Toluene is also readily sampled by the VOST methodology (EPA Method 0030).

MEK ranks near the bottom of Class 3 (Rank = 118). MEK is also native to Bostik's waste stream, and at varying concentrations (0-5% by weight) also depending on the specific adhesive products being produced. MEK is also readily sampled by the VOST methodology (EPA Method 0030).

On the basis of the above information, toluene represents the best choice for a POHC for this test program as it native to the waste, will likely be present at a sufficient concentration, can readily be sampled and analyzed by VOST and would not artificially introduce chlorine to the system.

5.2.1.2 POHC Feed Strategy

Table 5-2 provides general calculations on the appropriate feed quantity, based on assumptions regarding achievable DRE, stack gas flowrate and VOST sample volume. This information will be used to ensure that toluene is fed at the appropriate rate. In order to demonstrate adequate DRE on this difficult to destroy compound, the feed rate must be sufficient to allow calculation of at least 99.99% DRE yet not so high as to exceed the upper calibration range of the GC/MS instrumentation used for VOST analysis. The target feed rate for toluene should be in the range of 0.40 – 7.0 lb/hr and at this time is anticipated to be **4.0 lb/hr**.

Table 5–2 POHC DRE Supporting Calculations

POHC or Analytical Parameter	Feed Rate (lb/hr)	Assumed DRE	Stack Emissions (g/hr)	Total Collected	Method Dynamic Range
Volatile POHC - min.	0.40	99.99%	0.018	107 ng	10 - 2,000 ng
Volatile POHC - max.	7.00	99.99%	0.318	1,869 ng	10 - 2,000 ng
Volatile POHC - min.	3.50	99.999%	0.016	93 ng	10 - 2,000 ng
Volatile POHC - max.	70.0	99.999%	0.318	1,869 ng	10 - 2,000 ng

Stack Gas Assumptions:

	Value	**Units**
VOST (M 0030)	20	dsL
Sample Volume	0.71	dscf
Stack Gas Flowrate	2,000	dscfm

Additional Information Relative to Toluene Concentration in Waste Distillate:

At the planned waste distillate feed rate of 85.0 gph (652 lb/hr), the range in possible toluene feed rate would be as shown below:

Waste Distillate Toluene Concentration (% wt)	Waste Distillate Toluene Concentration (mg/kg)	Toluene Feed Rate (lb/hr)
0.01%	100	0.065
0.05%	500	0.326
0.075%	750	0.489
0.10%	1,000	0.652
0.15%	1,500	0.978
0.25%	2,500	1.630
0.50%	5,000	3.261
1.00%	10,000	6.522

As shown in Table 5-2, the minimum toluene feed rate required for demonstration of at least 99.99% destruction is about 0.4 lb/hr. As shown above, this equates to a concentration of 0.05-0.075% by weight or 500-750 mg/kg. In order to ensure that sufficient toluene is present in the waste distillate, Bostik has embarked on a weekly waste analysis program to track the presence of key constituents with the production processes. This program will continue until just prior to the CPT field test program. This information will be used to determine whether additional toluene may need to be added to the waste feed material at the time of testing. If additional toluene is needed, Bostik will add the material directly to the feed tank that will be used to feed the boiler during the test (either T-1 or T-2). Records will be kept regarding any toluene added to the tank for this purpose. Based on the minimum concentration noted above, the need to add additional toluene to the tank is considered very unlikely.

5.2.2 Supplemental Feeding of Regulated Constituents

Bostik does not plan to spike (inject) any regulated constituents into the waste distillate stream for purposes of establishing feed rate limits.

5.3 Test Materials and Quantities

5.3.1 Quantity of Hazardous Waste to be Burned

The quantity of hazardous waste to be burned during this program is based on the target feed rate specified in Table 5-1. Assuming about 12-hr per day of waste burning per day for 4 days, and the planned schedule outlined later in this section, it is estimated that about 7,800 pounds of hazardous waste would be burned per day and the total waste burned would therefore be approximately 31,300 pounds over 4 test days excluding the overnight periods when the burner would return to normal operation.

5.3.2 Time to Achieve Steady-State Operation

Because there is only a single liquid hazardous waste stream fed to the unit and since there is no air pollution control system, the time required to reach steady-state operation is governed primarily by the time to establish acceptable rolling averages for the process parameters. The liquid waste feed material will instantly vaporize and be swept through the combustion chamber. HRAs for all applicable parameters will be established at or near their desired values prior to test initiation. (It is expected that approximately one-hour of steady state operation will be required to establish desired HRAs prior to test initiation.) If emission sampling has to be interrupted during the middle of a run, the one-minute averages during the interruptions will not be used for the calculations of HRAs following the interruption. The last HRA considered will be concurrent with the end of the test run sampling period.

5.4 Test Schedule

This section summarizes the anticipated schedule for test program implementation. Two schedules are provided. The first describes the overall project schedule for planning, executing, and reporting on the test bum; the second provides a detailed schedule for the field sampling program. These schedules must still be considered tentative because changes or modifications may arise during the ongoing review and approval process for this Plan and associated Part B application.

Table 5-3 provides an overall project schedule for test planning, implementation and permitting activities. The schedule assumes that EPA will approve this Plan (Revision 2) by March 31, 2008 and that the testing will be performed in the 2nd or 3rd quarter of 2008.

Table 5-4 provides a more detailed schedule associated with the day-to-day activities of the test burn field schedule. This schedule includes days for arrival, safety orientation, and testing. This schedule assumes that testing will be conducted during '"extended" (10-14-hr) day shifts and that no around-the-clock testing is planned.

Table 5–3 Overall Project Schedule

Task	Schedule	Status
Revision 0 TBP Submittal	December 8, 2006	Complete
General Comments on TBP Received	April 10, 2007	Complete
Revision 1 TBP Submittal	June 12, 2007	Complete
EPA Review and Comment	November 28, 2007	Complete
Revision 2 TBP Submittal	January 31, 2008	Complete
EPA Review and Comment	March 31, 2008	Planned
EPA Review and Approval of TBP	April 15, 2008	Planned
EPA Prepares Public Notice	April 15, 2008	Planned
Public Comment Period	May 2008	Planned
Test Burn Planning Efforts	May 2008	Planned
Test Burn Field Program	June 2008	Requires 1 week to complete scheduled testing
Submit Test Burn Report / MACT Notification of Compliance	October 2008	Prior to October 14, 2008
Conduct Screening Level Risk Assessment and Submit Report	Oct – Dec 2008	Assume 3 months from test burn report submittal
Draft Permit Development	To be determined	To be determined

Table 5–4 Detailed Test Burn Field Schedule

General Overview of Planned Schedule

Activity	Schedule
Arrival onsite, site safety training and equipment set-up	Day 1
Preliminary stack measurements followed by Run 1 for all parameters	Day 2
Run 2 for all parameters	Day 3
Run 3 for all parameters	Day 4
Run 4 for all parameters	Day 5
Ship samples. Pack equipment and depart site.	Day 6

Example of Detailed Daily Schedule – Day 3 Above

Test Activity	Time
Unit lined out on test burn waste; CEMS daily calibrations conducted	06:00-07:00
All operating conditions and associated hourly rolling averages at or near their target values	07:00-08:00
Begin all continuous emissions monitoring. (Facility CEMS for CO and O_2 and ENSR CEMS for O_2, CO_2 and THC) Continuous monitoring performed throughout the test.	08:00
Begin Run 2 for Method 0023A (PCDDs/PCDFs/PAHs) and Method 0010 (SVOCs)	08:00
Complete Run 2 for above parameters	11:15
Begin Run 2 for Method 0030 (target volatile organics and POHC DRE), Method 29 (metals) and Method 26A (PM / HCl / Cl_2)	12:15
Complete Run 2 for above parameters	14:30
Complete sample train recoveries and sample train setup / preparations for Run 3 (next day)	14:30 – 17:00

6.0 Sampling and Analysis Program

This section presents a summary of the sampling and analysis program for this project. Further details on the overall sampling and analysis program are found in the QAPP for this project, located in Appendix A. As noted in previous sections of this document, the test program will consist of one test condition and four (4) sampling runs will be conducted at this single test operating condition.

6.1 Liquid Waste Sampling and Analysis

The HWC burns a single liquid waste feed stream that is fed from either Tank T-1 or T-2 to the burner. This liquid waste feed material will be sampled prior to being fed to the burner in accordance with acceptable protocols. Waste feed sampling will occur upstream of any POHC spiking location (if supplemental POHC is required to be injected). A sampling tap in the feed line is used to collect the waste distillate material.

The waste feed stream will be sampled at the beginning, middle and end of each run and analyzed for the parameters listed below in **Table 6-1**.

Table 6–1 Sampling and Analytical Summary for Liquid Waste Distillate

Sampling Method	Sampling Frequency	Analytical Parameter	Analytical Method
Grab / Composite	**(a)**	Heat Content	ASTM D 240
Grab / Composite	**(a)**	Ash Content	ASTM D 482
Grab / Composite	**(a)**	Density	ASTM D 1963
Grab / Composite	**(a)**	Viscosity	ASTM D 445
Grab / Composite	**(a)**	Total Chlorine / Chlorides	ASTM E 442 and EPA M 5050 / 9056
Grab / Composite	**(a)**	Water Content	ASTM E 203
Grab / Composite	**(a)**	Mercury	EPA M 7471A
Grab / Composite	**(a)**	Other metals **(b)**	EPA M 6010B
Grab / Composite	**(a)**	Target VOCs **(c)**	EPA M 8015B and 8260B

(a) Beginning, middle and end of each run

(b) Antimony, arsenic, barium, beryllium, cadmium, chromium, lead, nickel, silver and thallium

(c) Toluene, methyl ethyl ketone, xylene, tetrahydrofuran, ethyl acetate, methanol, ethylene glycol and diethylene glycol

Facility personnel will collect these samples under ENSR's direction using pre-cleaned sample bottles suitable for the type of sample being collected and the intended analysis. ENSR will provide all sample containers and assume custody of the samples at the end of each day. Prior to initiating field testing activities, ENSR will hold a training session with facility staff responsible for sample collection to review grab sampling techniques, size of sample aliquots, compositing procedures and sample bottles to be used. Agency personnel who will be providing testing oversight are invited to attend this training session. **Table 6-2** summarizes the regimen to be followed for collection of the waste feed samples. It is noted that the samples collected for analysis of volatile organics (3, 40-mL VOA vials per run) will be composited in the laboratory under controlled conditions to yield one sample per run.

Table 6–2 Grab Sampling Details for Liquid Waste Distillate

Analytical Parameter	Bottle Size	Grab Amount	Bottles Collected per run
Metals (antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver & thallium)	500-mL	150-mL	1
Physical Parameters (heat content, ash content, water content, density, viscosity & total chlorine/chlorides)	500-mL	150-mL	1
Volatile Organics (toluene, methyl ethyl ketone, xylene, tetrahydrofuran, ethyl acetate, methanol, ethylene glycol and diethylene glycol	40-mL VOA	40-mL	3

6.2 Process Vent Gas Sampling and Analysis

The process vent gas stream fed to the polyester burner originates from the 9 process batch reactors in the Direct Solvation and Polyester resin departments and the 4 storage tanks used to hold the waste distillate stream. The vent gas is continuously fed to the HWC at a modest rate of 50 – 500 cfm. The vent gas stream would not be expected to contain any metals or chlorine and would contain the same volatile organics as expected in the waste distillate stream. As such, no sampling of the vent gas stream will be performed during the test program.

6.3 Solid Waste Sampling and Analysis

No solids of any kind are fed to the HWC and thus, no solids sampling will be performed.

6.4 Supplemental Feed Material Sampling and Analysis

The only material that may be added to the waste distillate stream is the POHC, toluene, as discussed previously in Section 5.2.1.2. Toluene is purchased as a raw material by Bostik and typically has a certified purity level of at least 99% by weight. If toluene is added to the waste feed tank prior to the test, then Bostik will provide the certified analysis of this material as provided by the manufacturer and no sampling of the toluene will be performed.

6.5 Stack Gas Sampling and Analysis

- Gases discharged from the exhaust stack will be sampled for the parameters listed below and as delineated further in **Table 6-3**:
- Flue gas velocity, flow rate, temperature, moisture content and fixed gas (O_2 and CO_2) composition;
- Particulate matter;
- Hydrogen chloride and chlorine;
- Metals (antimony, arsenic, barium, beryllium, cadmium, total chromium, lead, mercury, nickel, silver and thallium)
- Target Volatile Organics (full method scan plus top 10 TICs);
- PCDDs/PCDFs and PAHs;
- Target Semivolatile Organics (full method scan plus top 10 TICs); and
- Total Hydrocarbons (THC) and Carbon Monoxide (CO).

Table 6–3 Sampling and Analytical Summary for Exhaust Gas Stream

Sampling Method	Run Duration	Analytical Parameter	Analytical Method
EPA Method 26A	2-hr	Particulate Matter	EPA Method 5
		and HCl and Cl_2	EPA Method 26A
EPA Method 29	2-hr	Mercury	EPA Method 7470A
		Other metals **(a)**	EPA Method 6020
EPA Method 0010	3-hr	Target SVOCs	EPA Method 8270C
EPA Method 0023A	3-hr	PCDDs/PCDFs	EPA Method 8290
EPA Method 0010	3-hr	PAHs	CARB Method 429
EPA Method 0030	2-hr	Target VOCs	EPA Methods 5041A and 8260B
EPA Method 25A	**(b)**	Total hydrocarbons	EPA Method 25A
EPA Methods 2 and 4	**(b)**	Gas velocity, temperature and moisture content	EPA Methods 2 and 4
EPA Method 3A	**(b)**	CO_2 and O_2	EPA Method 3A
Bostik CEMS	**(b)**	CO	Bostik CEMS QA Plan

(a) Antimony, arsenic, barium, beryllium, cadmium, chromium, lead, nickel, silver and thallium

(b) Continuous throughout all runs.

Stack gas emission samples will be collected from test ports that meet the minimum criteria specified in EPA Method 1. Only one test port level (with 2 isokinetic sampling ports and 2 additional ports) is available to accommodate testing of all emissions test parameters. Therefore, concurrent sampling for all parameters will not be possible during the test program. Further details on the stack configuration, field data sheets, isokinetic sampling train setup and recovery and program QA/Qc are provided in the QAPP (Appendix A).

Gas stream flowrate and moisture will be determined during each test run in conjunction with the isokinetic sampling trains. Gas stream velocity will be determined using a pitot tube and water manometer in accordance with EPA Method 2. Gas stream temperature will also be determined at each of the Method 2 traverse points using a Type “K” thermocouple and pyrometer. Gas stream moisture will be will be determined as specified in EPA Method 4 concurrent with the isokinetic sampling methods. In this procedure the impinger contents are measured or weighed before and after each test run and used in conjunction with the metered gas volume to determine the gas stream moisture content.

7.0 Final Data Reporting

The final report for this project will be a comprehensive data compilation that properly and logically documents and certifies all required test results. The report will include all of the required elements of a MACT NOC as outlined earlier in Section 1.4.2. ENSR plans to follow the guidance provided by EPA that defers to the suggested format as offered by the Louisiana Department of Environmental Quality (LDEQ) for a combined NOC and CPT report. As such, the report would be structured in a similar manner with sections delineated as follows:

- Summary of Test Results
- Introduction and Process Description
- Process Operating Conditions
- Feed Stream Sampling and Analysis
- Performance Test Results
- Risk Assessment Test Results
- Quality Assurance / Quality Control Documentation
- Proposed Permit Limits and OPLs
- Continuing Compliance Methods

Report appendices will also provide detailed supporting documentation as delineated in the above referenced LDEQ guidance. Appendices for the project report would include:

- Stack Sampling Report
- Feed Stream Sampling Report
- QA/QC Data Report
- Process Operating Data
- Sample Emission Calculations
- Field Logs
- Analytical Data Packages
- CMS / CEMS performance Evaluation Test Reports

Further details on data reporting are provided in Section 13.0 of the QAPP (Appendix A).

Appendix A

Quality Assurance Project Plan (QAPP)

Quality Assurance Project Plan for Bostik's Polyester Burner Unit (Middleton, MA) for the RCRA Trial Burn / MACT CPT Program

This document presents the Quality Assurance and Quality Control goals, objectives, and procedures for the Bostik – Middleton, MA facility Test Burn program. The quality assurance/quality control procedures and criteria for this program will comply with the requirements of this document and any updates. The analytical work conducted will incorporate the QA/QC requirements of the approved methods. This document has been prepared using available guidance provided in the following documents:

- Louisiana DEQ – Regulatory Burn Plan Recommendations: "Example Outline for Combined RCRA and MACT Test Burn Plan", April 2007
- "Component 2 - How to Review a Quality Assurance Project Plan (including Attachment A - Generic Trial Burn QAPP", Hazardous Waste Combustion Unit Permitting Manual, U.S. EPA Region 6, January 1998.
- "Handbook – Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration" (EPA/625/6-89/023 January 1990).

Facility ID Number: MAD 001 039 767

Prepared for: Bostik, Inc., 211 Boston Street, Middleton, MA 01949

Prepared by: ENSR Corporation, Westford, MA 01886

Revision No.: 2

Date: January 31, 2008

Expected Trial Burn Date: 2nd Quarter 2008

Quality Assurance Project Plan

Bostik, Inc.
RCRA Trial Burn Plan / MACT CPT Plan

Project Approvals

Mr. Dan Welch, Bostik Project Manager — Date

Mr. Douglas Roeck, ENSR QA Officer — Date

Mr. Patrick Ford, ENSR Project Manager — Date

Mr. Fred Sanguedolce, ENSR Field Team Leader — Date

Ms. Martha Maier, Vista Analytical Laboratory Coordinator — Date

Mr. William Anderson, Test America Laboratory Coordinator — Date

Ms. Bryanna Langley, Air Toxics, Ltd. Laboratory Coordinator — Date

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1.0 Project Description

This project will consist of a comprehensive sampling and analysis program designed to demonstrate compliance with applicable RCRA (Subpart H) and MACT (Subpart EEE) performance standards. Testing will be performed under a single process operating condition that will entail four (4) sampling runs. Operating limits for a number of process parameters will be set based on the results of the program. In addition, emission measurements are being made to collect relevant data for input to a screening level risk assessment (SLRA). The reader is referred to other sections of the overall test burn plan for further details on program scope, test objectives and target parameters for emission measurements and process monitoring. The remainder of this QAPP outlines the detailed measures that will be followed to ensure collection of valid data.

2.0 Project Organization

ENSR will be responsible for all emission measurements on this test burn program. The ENSR Project Manager, Mr. Patrick Ford, will provide overall direction of the program and will report to the Bostik Project Manager, Mr. Dan Welch. As project manager, Mr. Ford will be responsible for project design and implementation, communicating with the client and scheduling all activities. Mr. Ford is a Senior Program Manager in ENSR's Air Measurements Department and has over 30 years' experience in air measurements, compliance testing and sampling and analysis.

2.1 Facility Owner / Operator: Bostik, Inc.

Mr. Dan Welch is the Health, Safety, Environment and Quality Manager for the Transportation Division at Bostik. He is the Bostik project manager for the test burn program. Mr. Welch will be responsible for coordinating the Bostik effort during the program (including collection of waste feed samples) and will be the principal point of contact during implementation of the field test program.

2.2 ENSR Field Team Leader

Mr. Fred Sanguedolce will be responsible for directing and coordinating all field activities, communicating with the client, reviewing all project field data and preparing all reports. He will be assisted in the oversight of Quality Assurance activities by the program Quality Assurance Officer (QAO) and each Analytical Laboratory Services Coordinator (LSC). Each contract laboratory will have one individual designated as the person responsible for project activities. Mr. Sanguedolce is a Senior Field Technician in ENSR's Air Measurements Department and has over 20 years' experience in performing and directing complex field measurement programs.

2.3 ENSR QA Officer

Mr. Douglas Roeck will serve as the project Quality Assurance Officer (QAO) and will be responsible for review and approval of this QAPP, as well as any subsequent revisions. He will monitor implementation of field and laboratory activities and schedule performance and/or system audits as discussed later in Section 9.0. The QAO will report on any conditions noted which may adversely affect data quality. Mr. Roeck will be onsite throughout the duration of the field test program.

Mr. Roeck will provide independent oversight of Air Measurements Department activities for field sampling, data verification and data quality assessment activities. He will prepare a section for the Final Report summarizing QA/QC activities and provide an overall evaluation of data quality.

2.4 Regulatory Oversight

EPA Region 1 will be the lead Agency involved in review and approval of this QAPP. Mr. Stephen Yee is the primary point of contact for EPA's Boston, MA office. Additional review support is being provided by Ms. Cynthia Kaleri from EPA Region 6 in Dallas, TX. EPA will review and approve all documents associated with this project, will provide oversight of the field program and will be responsible for providing any audit samples at the time of the field measurement program.

2.5 Laboratory Services Coordinators

Each analytical laboratory will designate a coordinator, who will be the principal point of contact for the ENSR Field Team Leader. The LSC will review QA requirements with all laboratory staff to ensure that all required measures are taken to meet data quality objectives. They will monitor the shipment and receipt of samples, track analytical progress and review data as reported from the laboratories for completeness. Ms. Martha Maier will serve as the LSC for Vista Analytical Laboratories. Mr. William Anderson will serve as the LSC for Test America, Inc. Ms. Bryanna Langley will serve as the LSC for Air Toxics, Ltd. Each LSC will be responsible for validation of all data generated by the laboratory for this program and will provide all necessary documentation for inclusion in the final report.

3.0 Data Quality Objectives

This section provides a general overview of the data quality objectives (DQOs) for this test program. Specific DQOs for each individual sampling train and/or each analytical methodology performed by the subcontractor laboratories are provided later in Section 7.0 of this QAPP.

3.1 Precision, Accuracy and Completeness

The collection of data to fully characterize the burner waste feed material and stack gas emissions requires that sampling and analysis procedures be conducted with properly operated and calibrated equipment by trained personnel. The overall program has been designed with consideration of sampling parameters and analytical limits to ensure that the achieved method-specific detection limits for measured emissions will be more than adequate for demonstrating compliance and supporting the SLRA. **Table 3-1** provides a summary of the overall precision, accuracy and completeness objectives for the program.

Precision is defined as a measurement of mutual agreement among individual measurements made under prescribed similar conditions. Precision is expressed in terms of relative percent difference (RPD) between duplicate determinations and in terms of relative standard deviation (RSD) when 3 or more determinations are made. Overall precision for analysis of the waste feed streams will be assessed through the analysis of one set of duplicate samples for each designated parameter.

Accuracy is the degree of agreement of a measurement with an accepted reference or true value. Analytical accuracy will be measured through the recoveries of surrogate spikes, matrix spikes, analysis of standard reference materials or audit sample analysis. Surrogates are compounds added to samples submitted for organic analyses prior to extraction and analysis; their recoveries are measured to assess sample-specific analytical efficiency and accuracy. Matrix spike (MS) samples for the waste feed will be prepared by spiking known amounts of target analytes into a portion of the sample. Matrix spike samples for the stack organic analyses will be prepared by spiking known amounts of target analytes into the sampling media and then carrying the spiked sample through the entire preparation and analysis sequence. Recoveries are monitored to assess laboratory and method accuracy. Laboratory control samples (LCS) will also be used to distinguish between method performance and matrix effects on accuracy. LCS and MS solutions will be independent from calibration standards.

Completeness is a measure of the amount of valid data obtained compared to the amount that was expected under normal conditions. The overall program objective is to obtain valid data for three (3) runs for each test condition. For all data considered critical to the investigation, a completeness objective of 100% has been established. As a result, critical priority data from each set of three (3) runs should achieve the precision and accuracy goals established herein. This completeness criterion applies to all permit parameters in emissions samples as well as any feed/process stream samples. Individual samples for which the critical data points do not achieve accuracy and/or precision data quality objectives may require reanalysis. Results for samples where matrix interferences preclude meeting objectives for the recoveries of surrogates or spikes will be evaluated for potential bias to calculated emission results. In summary, the completeness goals are stated at 100%, since a minimum of three valid runs are necessary to assess operation at the one test condition.

Table 3–1 Precision, Accuracy and Completeness Objectives

Stream Sampled / Sampling Method	Parameter	Sampling Precision (RPD)	Analytical Precision (RPD)	Analytical Accuracy (%)	Completeness (%)
HW Liquid Feed					
Grab / Composite	Ash Content	< 50	< 35	75 -125	85
Grab / Composite	Water Content	< 50	< 35	75 -125	85
Grab / Composite	Density	< 50	< 35	75 -125	85
Grab / Composite	Viscosity	< 50	< 35	75 -125	85
Grab / Composite	Heat Content	< 50	< 35	75 -125	85
Grab / Composite	Total Chlorine	< 50	< 10	75 -125	85
Grab / Composite	Metals **(a)**	< 50	< 35	75 -125	85
Grab / Composite	VOCs **(b)**	< 50	< 35	75 -125	85
Stack Flue Gas					
EPA Method 26A	Particulate Matter	**(e)**	± 0.5 mg	± 0.1 mg	--
EPA Method 26A	HCl and Cl_2	**(e)**	± 30%	70 - 130	75
EPA Method 29	Metals **(a)**	**(e)**	± 25%	70 - 130	75
M29 (Hg) Audit	**EPA Audit Sample**			**50 -150**	**100**
EPA Method 0023A	PCDDs/PCDFs	**(e)**	--	--	75
EPA Method 0010	PAHs	**(e)**	< 40 %	50 - 150	75
EPA Method 0010	SVOCs **(c)**	**(e)**	< 40%	50 - 150	75
EPA Method 0030	VOCs **(d)**	**(e)**	± 25%	75 - 125	75
VOST Audit	**EPA Audit Cylinder**			**50 - 150**	**100**
EPA Method 25A	THC	**(e)**	± 5% calib.	± 5% calib.	100
Facility CEM	CO	**(e)**	± 3% span	± 3% span	100
EPA Method 3A	O_2 & CO_2	**(e)**	0.5%	0.5%	100

(a) Target metals (waste feed and stack) include: antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver and thallium.

(b) Target volatile organics (VOCs) in waste feed include: toluene, methyl ethyl ketone, xylene, tetrahydrofuran, ethyl acetate, methanol, ethylene glycol and diethylene glycol.

(c) Target semivolatile organics (SVOCs) in stack gas include: full method scan plus top 10 TICs

(d) Target VOCs in stack gas include: full method scan plus top 10 TICs

(e) Precision not determinable for stack gas sampling since co-located sampling trains will not be used.

Note: This table represents an overall summary of the QA objectives for this project. Please refer to the method-specific QA summary tables in Section 7.0 of this QAPP.

3.2 Representativeness and Comparability

It is recognized that the usefulness of the data is also contingent upon meeting the criteria for representativeness and comparability. Wherever possible, reference methods and standard sampling procedures will be used. The QA objective is that all measurements be representative of the matrix and operation being evaluated. The detailed requirements for sampling given in the various EPA Reference Methods will be followed to ensure representative sampling of flue gases. The grab/composite sampling regimen for the boiler feed stream during each test run will also provide representative samples of this matrix.

The corresponding QA objective is that all data resulting from sampling and analysis be comparable with other representative measurements made by the field sampling team, on this or a similar process operating under similar conditions. The use of published sampling and analytical methods and standard reporting units will aid in ensuring the comparability of the data.

3.3 Data Usability and Detection Limit Considerations

ENSR and each of the subcontract laboratories on this program are aware of the requirement that all data generated for a program of this nature are of high quality and that detection limits reported are usable for both compliance and risk assessment purposes. We have reviewed the EPA Region 6 guidance on this issue and believe that the data to be generated for this program will meet or exceed EPA's goals based on our past experience with each specific laboratory on past similar programs. All of the laboratories to be used on this program follow 40 CFR Part 136, Appendix A for the determination of method-specific method detection limits (MDLs) for the various analytes to be measured in this program. However, for the purposes of data reporting for this program, method specific reporting limits (RLs) will be used wherever a sample is determined to be below detection. Three categories of such RLs are envisioned for this project:

- **Waste Feed Samples** – RLs for metals and total chlorides in the waste distillate samples will be specific to the actual waste matrix. The actual anticipated RLs were noted previously in Table 1-1 of the CPT Plan. The full value of the RL will be used in performing any required calculations pertaining to compliance with feed rate limits. The RLs to be reported for these parameters are equivalent to sample quantitation limits (SQLs) as defined by EPA, since they take into account any required sample dilutions.
- **Isotope Dilution Methods** – For this program, isotope dilution methods include EPA Method 8290 (PCDDs/PCDFs) and CARB Method 429 (PAHs). Reporting limits for these two methods incorporate specific criteria for development of estimated detection limits (EDLs) and estimated maximum possible concentrations (EMPCs). Emission calculations that rely on either the EDL or the EMPC are not expected to present any problems on this project. It is noted that for establishing compliance with the MACT PCDD/PCDF standard, detection limits can be treated as zero. For purposes of the SLRA, any EDLs or EMPCs for PCDDs/PCDFs and PAHs will use the full value reported.
- **Non-Isotope Dilution Methods** – For this program, such methods include EPA Methods 6020 (metals), 5041A and 8260B (volatile organics), 26A (hydrogen chloride and chlorine) and 8270C (semivolatile organics). Reporting limits anticipated for these methods are not expected to present any problems on this project. The full value of any RL will be used in making any emission determinations if the analyte is reported below detection.

Projected emission rates calculated using the expected RLs for this project for key organic and inorganic parameters considered most important to the SLRA are provided in **Attachment A**.

4.0 Sampling and Monitoring Procedures

This section describes the procedures that will be followed during the field sampling program. Throughout the overall program, all sampling will be performed using sampling protocols described herein and approved by EPA. Regulatory agency approval will be obtained for any deviations from or changes to the approved QAPP which may be warranted prior to program implementation as a result of changes in personnel or facility circumstances. If situations occur during any preliminary testing that may be done prior to the test burn which necessitates deviations from the plan, the agency will be notified and onsite approval requested. Any such deviations from the specified protocols will be fully documented in the final report for the project.

A discussion of the compliance strategy, test conditions and sampling and analysis program was provided previously in Sections 1.0, 5.0 and 6.0 of the test burn plan. In general, however, the test program is configured to collect samples during four runs conducted under a single process operating condition.

Sample team meetings will be held to designate responsibilities to each team member. Assignments will be based on individual experience and relative importance of the assigned task. Other activities performed in the office prior to the field test program include generation of sample checklists, printing of computer-generated sample labels, and proper packing of all equipment. Equipment will then be transported by truck to the sampling location. Site setup will involve moving the equipment to the vicinity of the sample collection area. A separate office trailer or other suitable onsite facility will be used to serve as a sample train setup and recovery area and sample custody area.

4.1 Waste Distillate Sample Collection

4.1.1 Sampling Location

Liquid waste feed material will be sampled from a sample tap in the feed line located approximately 22 ft upstream of the burner. The sampling valve in the feed line is shown in Figure 4-5 of the test burn plan and is located just upstream of the waste distillate automatic waste cutoff valve.

4.1.2 Sampling Procedure

The waste feed stream will be sampled at the beginning, middle and end of each run and analyzed for the parameters listed in **Table 4-1**. Facility personnel will collect these samples under ENSR's direction using pre-cleaned sample bottles suitable for the type of sample being collected and the intended analysis. ENSR will provide all sample containers and assume custody of the samples at the end of each day. Prior to initiating trial burn testing activities, ENSR will hold a training session with facility staff responsible for sample collection to review grab sampling techniques, size of sample aliquots, compositing procedures and sample bottles to be used. Agency personnel who will be providing test program oversight are invited to attend this training session. Table 4-2 summarizes the grab sampling details for the waste distillate stream.

The feed stream will be characterized for the expected volatile organic constituents (toluene, MEK, xylene, tetrahydrofuran, ethyl acetate, methanol, ethylene glycol and diethylene glycol), metals, ash, density, viscosity, water content, heat content and total chlorine.

Table 4–1 Sampling and Analysis Summary for Waste Distillate

Stream Sampled / Sampling Frequency	Test Parameter	Sampling Method	Analytical Method(s)
HW Liquid Feed beginning, middle and end of each run (4 runs)	Ash Content	Grab / Composite (1 bottle / run)	ASTM D 482
	Water Content	Grab / Composite (1 bottle / run)	ASTM E 203
	Density	Grab / Composite (1 bottle / run)	ASTM D 1963
	Viscosity	Grab / Composite (1 bottle / run)	ASTM D 445
	Heat Content	Grab / Composite (1 bottle / run)	ASTM D 240
	Total Chlorine	Grab / Composite (1 bottle / run)	TA SOP WC 0016
	Metals **(a)**	Grab / Composite (1 bottle / run)	EPA Methods 6010B and 7471A
	VOCs **(b)**	Grab (3 VOA vials per run) Composited at Laboratory	EPA Methods 8015B and 8260B

(a) Target metals include: antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver and thallium.

(b) Target volatile organics (VOCs) in waste feed include: toluene, methyl ethyl ketone, xylene, tetrahydrofuran, ethyl acetate, methanol, ethylene glycol and diethylene glycol

Table 4–2 Liquid Waste Stream Grab Sampling Details

Analytical Parameter	Bottle Size	Grab Amount	Bottles Collected per run
Metals (antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver & thallium)	500-mL	150-mL	1
Physical Parameters (heat content, ash content, water content, density, viscosity & total chlorine/chlorides)	500-mL	150-mL	1
Volatile Organics (toluene, methyl ethyl ketone, xylene, tetrahydrofuran, ethyl acetate, methanol, ethylene glycol and diethylene glycol	40-mL VOA	40-mL	3

4.2 Stack Emission Measurements

Gases discharged from the exhaust stack will be sampled for the following parameters:

- Flue gas velocity, flow rate, temperature, moisture content and fixed gas (O_2 and CO_2) composition;
- Particulate matter;
- Target Volatile Organics;
- PCDDs/PCDFs and PAHs;
- Target Semivolatile Organics; and
- Total Hydrocarbons (THC) and Carbon Monoxide (CO) corrected to 7% O_2.

Table 4-3 provides a summary of the stack sampling protocols and procedures for the program. The following sections provide additional information on the sampling location and summaries of the sampling methodologies. In addition, example field data sheets to be used during the program are provided in **Attachment B**. Summaries of relevant information pertaining to setup and recovery of each isokinetic sampling train are provided in **Attachment C**.

Table 4–3 Overview of Stack Emission Measurement Program

Stack Sampling Frequency	Test Parameter	Sampling Method	Analytical Method(s)
2-hr run / 4 runs	Particulate Matter	EPA Method 26A	EPA Method 5
2-hr run / 4 runs	HCl and Cl_2	EPA Method 26A	EPA Method 26A
2-hr run / 4 runs	Metals **(a)**	EPA Method 29	EPA Methods 29, 6020 and 7470A
3-hr run / 4 runs	PCDDs/PCDFs	EPA Method 0023A	EPA Method 8290
3-hr run / 4 runs	PAHs	EPA Method 0010	CARB Method 429
3-hr run / 4 runs	SVOCs **(b)**	EPA Method 0010	EPA Method 8270C
2-hr run / 4 runs total / 4 VOST tube sets per run	VOCs **(d)**	EPA Method 0030	EPA Methods 5041A and 8260B
CEM / 4 runs	THC	EPA Method 25A	EPA Method 25A
CEM / 4 runs	CO	Facility CEM	Facility CEM
CEM / 4 runs	O_2 and CO_2	EPA Method 3A	EPA Method 3A
(a) Target metals include: antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver and thallium.			
(b) Target semivolatile organics (SVOCs) include: full method scan plus top 10 TICs			
(c) Target VOCs include: full method scan plus top 10 TICs			

4.2.1 Sampling Location

Gaseous emissions samples will be collected from test ports that meet the minimum criteria specified in EPA Method 1. Only one test port level (with 2 isokinetic sampling ports and 2 additional ports) is available to accommodate testing of all emissions test parameters. Therefore, concurrent sampling for all parameters will not be possible. **Figure 4-1** provides a schematic drawing of the burner stack showing the location of the sampling ports and the upstream/downstream distances from flow disturbances. This schematic drawing also provides information related to the traverse point locations applicable to the isokinetic sampling trains as well as key stack parameters needed to select the appropriate size sampling nozzle.

4.2.2 Gas Stream Velocity and Moisture

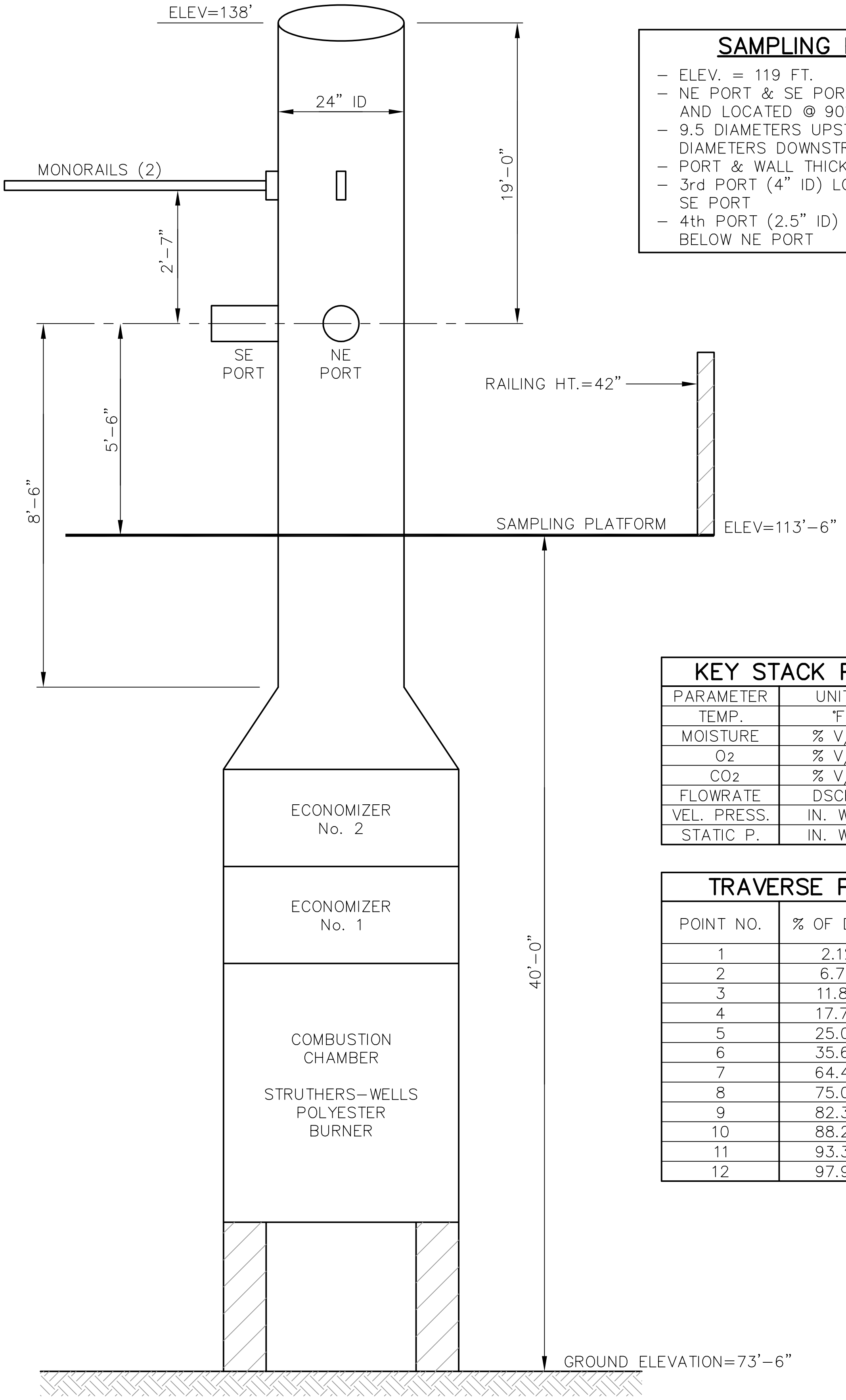
Gas stream flowrate and moisture will be determined during both test conditions concurrent with each of the isokinetic sampling trains. Gas stream velocity will be determined using a pitot tube and water manometer in accordance with EPA Method 2. Gas stream temperature will also be determined at each of the Method 2 traverse points using a Type “K” thermocouple and pyrometer. Gas stream moisture will be will be determined as specified in EPA Method 4 concurrent with the isokinetic sampling methods. In this procedure the impinger contents are measured or weighed before and after each test run and used in conjunction with the metered gas volume to determine the gas stream moisture content.

4.2.3 Particulate Matter (PM), Hydrogen Chloride (HCl) and Chlorine (Cl_2)

Sampling for PM, HCl and Cl_2 will be performed in accordance with EPA Method 26A, as written. Specific sampling details for the Method 26A sampling train are as follows:

- Target sampling rate - 0.75 cfm
- Sample train run time - 2-hr
- Estimated sample volume - 2.4 dscm (85.0 dscf)
- No. of sampling points per stack traverse - 12
- Total No. of sampling points - 24
- No. of field reagent blank sets collected – 1

The sampling method incorporates the option for collection of particulate matter as delineated in Section 2.0 of Method 26A. The sampling train consists of 6 glass impingers connected in series with leak-free ground glass and Teflon o-ring connections. Each of the first two impingers are filled with 100-mL of 0.1N sulfuric acid; the third impinger is empty and the fourth and fifth impingers are each filled with 100-mL of 0.1N sodium hydroxide. The sixth impinger is loaded with ~ 400 g of silica gel. The sampling train is operated as specified in the method. Because of the high stack gas temperature at this source, a tared quartz filter rather than a Teflon filter will be used as specified in the method. **Attachment C** provides the field setup and recovery schematics for this sampling train.



SAMPLING PORTS (4)

- ELEV. = 119 FT.
- NE PORT & SE PORT ARE 4" ID AND LOCATED @ 90° TO EACH OTHER
- 9.5 DIAMETERS UPSTREAM AND 4.25 DIAMETERS DOWNSTREAM OF DISTURBANCES
- PORT & WALL THICKNESS = 6.625"
- 3rd PORT (4" ID) LOCATED 8" BELOW SE PORT
- 4th PORT (2.5" ID) LOCATED SLIGHTLY BELOW NE PORT

KEY STACK PARAMETERS

PARAMETER	UNITS	VALUE
TEMP.	°F	623
MOISTURE	% V/V	21.6
O_2	% V/V	4.0
CO_2	% V/V	12.7
FLOWRATE	DSCFM	1,890
VEL. PRESS.	IN. W.C.	0.11
STATIC P.	IN. W.C.	−0.20

TRAVERSE POINT DATA

POINT NO.	% OF DIAM.	DIST. INCL. PORT (IN.)
1	2.1%	7.1
2	6.7%	8.2
3	11.8%	9.5
4	17.7%	10.9
5	25.0%	12.6
6	35.6%	15.2
7	64.4%	22.1
8	75.0%	24.6
9	82.3%	26.4
10	88.2%	27.8
11	93.3%	29.0
12	97.9%	30.1

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STACK SAMPLING LOCATIONS – POLYESTER BURNER UNIT
BOSTIK, INC.
211 BOSTON STREET
MIDDLETON, MASSACHUSETTS

FIGURE NUMBER: 4-1

DRAWN BY:	DATE:	PROJECT NUMBER:	SHEET NUMBER:
K.P.B.	2/08	00963-044	X

FILENAME: 00963-044-01A.DWG

4.2.4 Metals

Sampling for target metals (arsenic, antimony, barium, beryllium, cadmium, total chromium, lead, mercury, nickel, silver and thallium) will be performed in accordance with EPA Method 29, as written. Specific sampling details for the Method 29 sampling train are as follows:

- Target sampling rate - 0.75 cfm
- Sample train run time - 2-hr
- Estimated sample volume - 2.4 dscm (85.0 dscf)
- No. of sampling points per stack traverse - 12
- Total No. of sampling points - 24
- No. of field reagent blank sets collected – 1

The sampling train consists of 6 glass impingers connected in series with leak-free ground glass and Teflon o-ring connections. Each of the first two impingers are filled with 100-mL of a 5% nitric acid / 10% hydrogen peroxide solution; the third impinger is empty and the fourth and fifth impingers are each filled with 100-mL of a 10% sulfuric acid / 4% potassium permanganate solution. The sixth impinger is loaded with ~ 400 g of silica gel. The sampling train incorporates an untared quartz filter and is operated as specified in the method. Details pertaining to the setup and recovery of the sampling train are presented in **Attachment C**.

4.2.5 Target Volatile Organics

EPA Method 0030 will be followed as written to determine stack gas concentrations of the volatile POHC as well as a full list of target volatile compounds (see Attachment E). Data collected from VOST samples will be used to calculate the DRE for toluene. The VOST method utilizes Tenax and Tenax/Charcoal cartridges to adsorb target volatile organic compounds; each cartridge is preceded by a condensing module. Specific sampling details for the Method 0030 train are as follows:

- Sampling rate - 1.0 Lpm
- VOST pair run time - 20-minutes
- VOST sample volume per tube pair – 20 L
- VOST tube pairs collected per run - 4 (a, b, c and d)
- VOST tube pairs designated for analysis - 3 (a, b and d)
- Minimum probe temperature - 135 °C
- No. of field blank pairs collected – 1 per day of testing
- No. of trip blank pairs collected - 1

The recovery activities for the VOST method will include:

- Sealing the sorbent cartridges with threaded caps and Swagelok fittings and placing them in their original glass culture tubes with glass wool to absorb shock.

- Transferring the collected condensate into a 40 mL VOA vial, noting the volume collected by marking the VOA vial with a black indelible marker and diluting to volume with HPLC water to decrease headspace and the possibility of revolatilization of the compounds.
- Further reducing reactivity by storing all samples at 4°C.

4.2.6 PCDDs/PCDFs and PAHs

A combined EPA Method 0023A / 0010 sampling train will be used to sample for all target parameters. PCDDs/PCDFs will be collected following the procedures outlined in EPA Method 0023A. Target PAHs will be collected following the procedures outlined in Method 0010. Specific sampling details for the Method 0023A / 0010 sampling train are as follows:

- Target sampling rate - 0.75 cfm
- Sample run time - 3-hr
- Minimum sample volume required - 3.0 dscm (105.6 dscf)
- Sample train rinse solvents: acetone, methylene chloride and toluene
- No. of sampling points per stack traverse - 12
- Total No. of sampling points - 24
- No. of field blanks collected – 1

The sampling train consists of 5 glass impingers connected in series with leak-free ground glass and Teflon o-ring connections. The first impinger is left empty and the second and third impingers are filled with 100-mL of HPLC water; the fourth impinger is empty and the fifth impinger is loaded with ~ 400 g of silica gel. The sampling train uses an untared glass fiber filter, an XAD resin trap and condensing module and is operated as specified in the method. Details pertaining to the setup and recovery of the sampling train are presented in **Attachment C**.

4.2.7 Target Semivolatile Organics

A Method 0010 sampling train will be used to sample for target semivolatile organics (see Attachment E). The method will be followed as written and specific sampling details for the Method 0010 sampling train are outlined below.

- Target sampling rate - 0.75 cfm
- Sample run time - 3-hr
- Minimum sample volume required - 3.0 dscm (105.6 dscf)
- Sample train rinse solvents: methanol / methylene chloride (1:1 v/v)
- No. of sampling points per stack traverse – 12
- Total No. of sampling points – 24
- No. of field reagent blank sets collected – 1

The sampling train consists of 5 glass impingers connected in series with leak-free ground glass and Teflon o-ring connections. The first impinger is left empty and the second and third impingers are filled with 100-mL of HPLC water; the fourth impinger is empty and the fifth impinger is loaded with ~ 400 g of silica gel. The sampling train uses an untared glass fiber filter, an XAD resin trap and condensing module and is operated as specified in the method. Details pertaining to the setup and recovery of the sampling train are presented in **Attachment C**.

4.2.8 Continuous Emission Monitoring – Bostik

Plant-owned CEMS, installed in the burner's exhaust stack, will be used during all test runs to monitor the concentrations of O_2 and CO in the stack gas. The Bostik CEMS includes a TECO Model 48H CO analyzer and a Servomex Model 1400 O_2 analyzer; specifications were provided earlier in Section 4.6.3 and Table 4-3 of the CPT Plan.

Stack gas is continuously drawn through a filter and heated sample transport line. The gas is conditioned to remove water, and any condensate is removed. The resulting dry gas flows into each of the gas analyzers. The O_2 results are used to correct the CO reading to 7% O_2 using the following equation:

$$CO_{Corr} = CO_{meas}\ x\ \frac{14}{21-Y}$$

where:

CO_{Corr} = CO concentration corrected to 7% oxygen

CO_{meas} = CO concentration as measured directly in the stack gas stream

Y = the oxygen content measured in the stack gas stream

From the O_2 corrected readings, a one-minute average CO concentration is calculated every minute. At each successive minute, the 60 most recent one-minute average CO concentrations are used to calculate an HRA CO concentration. The one-minute and HRA CO (O_2 corrected) and O_2 concentrations are automatically recorded by the process control / data acquisition system. If the HRA CO concentration exceeds 100 ppmv corrected to 7% O_2, then an AWFCO is initiated. As per the requirements of 63.1209(a)(3), one-minute average CO values that exceed the upper span limit for the analyzer (3,000 ppm) will be recorded as 10,000 ppm and used in the calculation of the HRA.

The Bostik CEMS will be certified prior to conducting the test following the performance specification (PS) test procedures provided in PS 3 (“Specifications and Test Procedures for O_2 and CO_2 Continuous Emission Monitoring Systems in Stationary Sources”) and 4B (“Specifications and Test Procedures for Carbon Monoxide and Oxygen Continuous Monitoring Systems in Stationary Sources”) found in 40 CFR Part 60, Appendix B. In addition, the certification will follow the general guidelines outlined in the Appendix to Subpart EEE (“Quality Assurance Procedures for Continuous Emissions Monitors Used for Hazardous Waste Combustors”). The CEMS certification will take place in accordance with the normal schedule followed by the facility on an annual basis. This normal schedule also includes daily calibrations and quarterly audits in accordance with the regulations.

4.2.9 Continuous Emission Monitoring – ENSR

During all test runs of the test burn, ENSR will also perform continuous emission monitoring for O_2, CO_2 and total hydrocarbons (THC). Measurement of O_2 and CO_2 will be conducted in accordance with EPA Method 3A and measurement of THC will be in accordance with EPA Method 25A. The O_2 and CO_2 measurements will be performed to allow computation of the stack gas molecular weight. The THC measurements are required to be performed in conjunction with the DRE test for the purpose of demonstrating compliance with the MACT rule.

5.0 Sample Custody

A variety of activities are performed prior to and during the field sampling program to ensure proper sample collection, documentation and sample transport. These activities include equipment calibration, sample media preparation, cleaning of sample train glassware, preparation of computer-generated sample labels, and other miscellaneous tasks. Each of these activities are described or referenced in the following subsections. Other pre-sampling activities include such details as team meetings, equipment packing and shipment, equipment setup, and finalization of all details leading up to the coordinated initiation of the sampling program.

5.1 Field Sampling Operations

5.1.1 Glassware Preparation

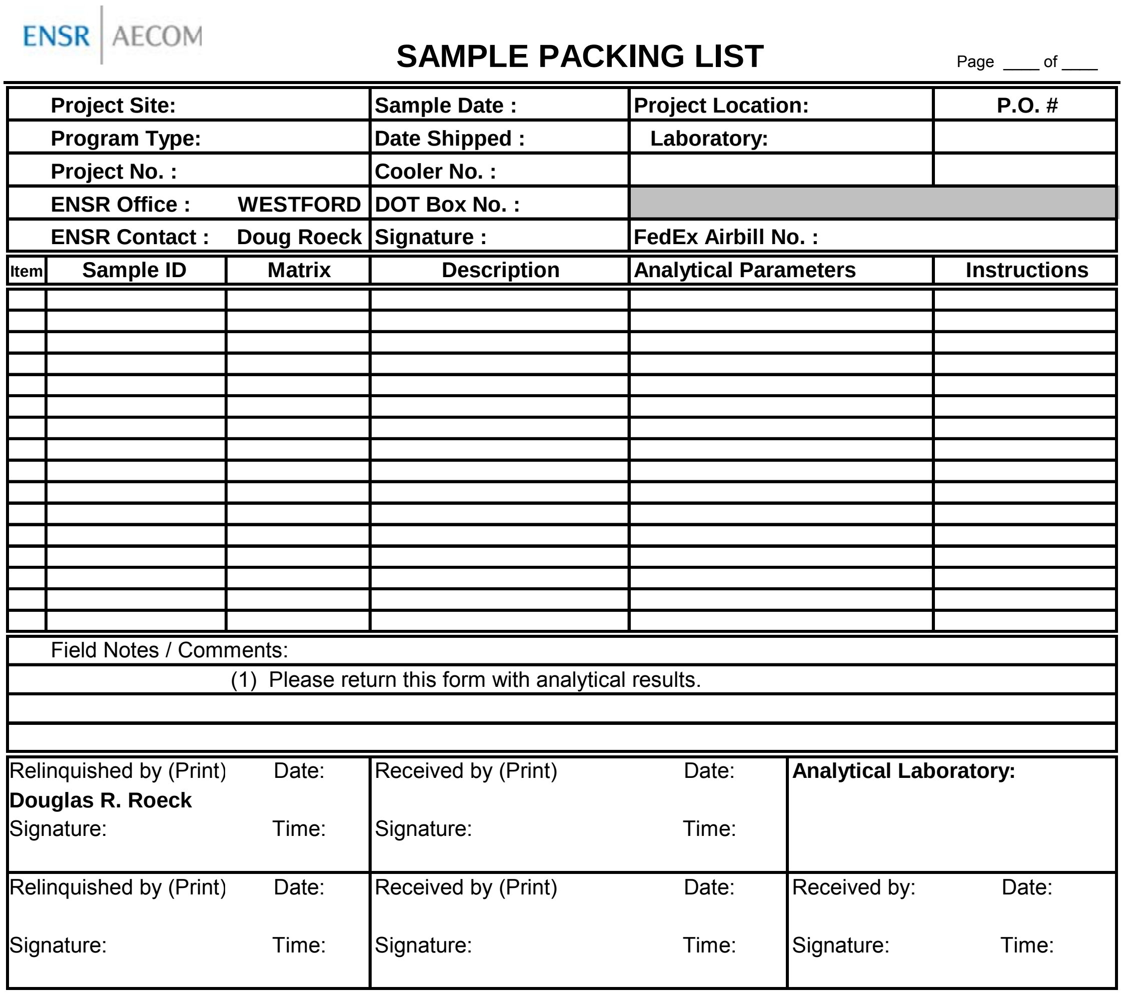
Sample train glassware and sample containers require specialized pre-cleaning to avoid contamination of the sample from the collection container or devices. Cleaning/storage procedures for sample train glassware are summarized below. Note that all bottle caps are fitted with Teflon liners which are cleaned in the same manner as the bottles themselves. Sample containers used for all waste feed and stack gas samples are purchased pre-cleaned and sealed to specified EPA protocols (PC class).

- **EPA Method 0023A / 0010 glassware and containers (PCDDs/PCDFs, PAHs and other semivolatile organics)** - wash with soap and water, rinse three times with deionized (DI) water, bake at 400°C for 2-hours, rinse three times with pesticide grade methylene chloride, rinse three times with pesticide grade toluene and air dry. Open ends will be sealed prior to shipment to the field with clean aluminum foil.
- **EPA Method 29 glassware and containers (metals)** – wash with soap and water, rinse with hot tap water, rinse three times with reagent water. The glassware is next soaked in a 10% nitric acid solution for a minimum of 4-hours, rinsed three times with reagent water, rinsed a final time with acetone and air dried. All glassware openings where contamination can occur will be covered until the sampling train is assembled prior to sampling.
- **EPA Method 26A glassware and components (particulate matter and HCl/Cl_2)** - wash with soap and water, rinse three times with DI water and air dry. Open ends will be sealed prior to shipment to the field with paraffin.
- **EPA Method 0030 glassware and containers (volatile organics)** - wash with detergent (Alconox) and hot water, rinse three times with HPLC grade water and oven dry at 110°C for 2 hours. Open ends will be sealed prior to shipment to the field with clean aluminum foil.

5.1.2 Sample Labels and Sampling Checklists

Preprinted sample identification labels are used by ENSR to ensure that all required information is fully documented. When sample batches are shipped to the specified laboratory, a sample packing list (chain-of-custody form) such as that shown in **Figure 5-1** accompanies the shipment. This form is based on established laboratory format and will be used to document sample transfer in the field and from sampling personnel to the laboratory. ENSR uses an in-house proprietary program for generating sample labels and the accompanying sample packing lists. These lists are also used by the Field Team Leader to ensure that all samples are collected as planned and recovered and packed accordingly.

Figure 5-1 Sample Packing List



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SAMPLE PACKING LIST Page ____ of ____

Project Site:	**Sample Date :**	**Project Location:**	**P.O. #**
Program Type:	**Date Shipped :**	**Laboratory:**	
Project No. :	**Cooler No. :**		
ENSR Office : WESTFORD	**DOT Box No. :**		
ENSR Contact : Doug Roeck	**Signature :**	**FedEx Airbill No. :**	

Item	Sample ID	Matrix	Description	Analytical Parameters	Instructions

Field Notes / Comments:
(1) Please return this form with analytical results.

Relinquished by (Print) Date: **Douglas R. Roeck** Signature: Time:	Received by (Print) Date: Signature: Time:	**Analytical Laboratory:**
Relinquished by (Print) Date: Signature: Time:	Received by (Print) Date: Signature: Time:	Received by: Date: Signature: Time:

5.1.3 Preliminary Measurements

Normally, preliminary tests are conducted at the stack location to verify the presence or absence of cyclonic flow conditions and to determine flue gas moisture, temperature and velocity. These measurements facilitate determination of nozzle size selection and sample train operation rates for the isokinetic sampling trains. However, extensive past testing at the Bostik facility eliminates the need for such preliminary measurements on the polyester burner stack. Cyclonic flow conditions do not exist and expected stack parameters were noted previously on Figure 6-1.

5.1.4 Field Documentation

The QAO will maintain a field log of all daily activities including facility preparations, sample run times, problems encountered, any corrective actions taken and other important events related to POHC spiking or equipment operation. The field log will be included in an appendix of the final report.

All materials such as field and laboratory notebooks and logbooks, field and laboratory data records, correspondence, reports, sample tags, traceability records and instrument printouts will be clearly labeled with the project number and become a permanent part of the project file. Project samples will be disposed of in an appropriate manner 60 days after acceptance and approval of a final report. All project-related documentation at both ENSR and the subcontractor laboratories will be kept on file for 2 years following submittal of the final report.

5.2 Field Laboratory Operations

5.2.1 Sample Media Preparation

All reagents will be checked in accordance with ENSR's existing QC Program to minimize the probability of using contaminated solvents. This includes the use of the proper grade reagents/solvents as specified in the test method, selection of reagents from the same lot and the collection and analysis of the appropriate blanks. Sampling media will be procured and prepared in accordance with the appropriate test methods as described below:

- **Tenax and Tenax/charcoal sorbent traps** are purchased new and conditioned in accordance with protocols outlined in Methods 0030 and/or 5041A.
- **XAD resin** is purchased new and packed in specially designed sorbent traps. All glass cleaning and sorbent packing procedures will follow the protocols specified in EPA Methods 0023A and 0010.
- **Untared Quartz filters** used in the Method 29 sampling train are purchased from Pallflex Products Co. who pre-screen filters for metals content.
- **Tared Quartz filters** used in the Method 26A sampling train (due to high stack temperature) are also purchased from Pallflex Products Co. with designated technical specifications and efficiency ratings.

5.2.2 Field Laboratory Facility

ENSR will arrange for the rental of an office trailer (~40 ft x 12 ft) to serve as a clean area for equipment staging, sample train setup and recovery, team meetings and to serve as the central area for coordinating testing activities and interacting with facility and Agency personnel. Special areas will be established in this office trailer for setting up and recovering the isokinetic sampling trains and/or for performing preliminary equipment checks. The use of special designated areas for each sampling train will help to eliminate sample train cross-contamination and ensure that the appropriate solvents and reagents are kept in their own specific area for use on the sampling train intended.

5.2.3 Sample Storage

Sample integrity will be maintained throughout all phases of the sampling and analysis program. Samples will be held within sight of the samplers or sample custodian, or will be kept in sealed or secured containers at all times. Sealed coolers and DOT shipping boxes will be used to ship samples to the designated laboratory via Priority 1 overnight FedEx service. All samples will be kept in the office trailer prior to shipment and the office trailer will be locked overnight.

5.2.4 Sample Shipment

The ENSR Field Team Leader will coordinate the packing and shipment of all samples. Worksheets specifically designed for this program will be generated prior to the field effort. These sheets will assist the Field Team Leader in assuring that all samples have been collected, accounted for and shipped under sample traceability documentation to the appropriate laboratory.

5.2.5 Sample Preservation and Holding Times

All samples will be kept on ice in method-specific coolers until they are ready for shipment to the designated laboratory. As stated earlier, these samples will be shipped in either sealed coolers or DOT shipping boxes (dangerous goods items). PM samples (filters and front-half rinse samples) from the Method 26A sampling train will be driven back to ENSR's laboratory at the end of each test day. **Table 5-1** below provides additional requirements pertaining to sample preservation and recommended holding times.

Table 5–1 Sample Preservation and Holding Time Requirements

Stack Gas Samples [(a)]

Parameter	Matrix	Preservation	Holding Time
Volatile Organics (Method 0030)	Aqueous	Cool, 4°C	14 days
	Tenax and Tenax/charcoal	Cool, <10°C	14 days
HCl / Cl_2 (Method 26A)	Aqueous	N/A	30 days
Mercury (Method 29)	Aqueous	Cool, 4°C	28 days
	Solid/Filter	Cool, 4°C	28 days
Other Metals (Method 29)	Aqueous	Cool, 4°C	6 months
	Solid / Filter	Cool, 4°C	6 months
PCDDs/PCDFs/PAHs and SVOCs (Methods 0023A and 0010)	XAD Resin	Cool, 4°C	30 days (to extraction)
			45 days (extraction to analysis)
[(a)] Holding times will be calculated from the day of sample collection.			

Waste Feed Samples

Parameter	Matrix	Preservation	Holding Time
Metals	Organic Liquid	Cool	6 months
Metals - Mercury	Organic Liquid	Cool	28 days
Total Chlorine	Organic Liquid	Cool	30 days
Volatile Organics	Organic Liquid	Cool	14 days

6.0 Calibration Procedures and Frequency

A very important aspect of pre-sampling preparations is the inspection and calibration of all equipment planned to be used for the field effort. Equipment is inspected for proper operation and durability prior to calibration. Calibration of equipment is conducted in accordance with the procedures outlined in the EPA document entitled "Quality Assurance Handbook for Air Pollution Measurement Systems; Volume III—Stationary Source Specific Methods" (EPA/600/R-94/038c, September 1994). Equipment calibration is performed in accordance with EPA guidelines and/or manufacturer's recommendations. Documentation of all calibration records will be kept in the project file during the field program and will be available for inspection by test observers. Recommended practices from the QA Handbook for field equipment to be used during this program and specific calibration procedures performed by ENSR are listed below.

- **Sampling Nozzles** [QA Handbook Section 3.4.2, pg. 19 - make three measurements of the nozzle ID (to the nearest 0.001 in.) using different diameters with a micrometer. Difference between the high and low values should not exceed 0.004 in. Post-test check - inspect for damage.] Each glass nozzle is calibrated with a micrometer prior to testing and identified with a unique ID number. Any stainless steel nozzles used during the program are calibrated onsite prior to testing.
- **Pitot Tubes** [QA Handbook Section 3.1.2, pp. 1-13 - measured for appropriate spacing and dimensions or calibrate in a wind tunnel. Rejection criteria given on the calibration sheet. Post-test check - inspect for damage.] Each S-type stainless steel pitot tube used is designed to meet geometric configurations as defined in EPA Method 2.
- **Thermocouples** [QA Handbook Section 3.4.2, pp. 15-18 - verify against a mercury-in-glass thermometer at two or more points including the anticipated measurement range. Acceptance limits - impinger ±2°F; DGM ±5.4°F; stack ±1.5 percent of stack temperature.] The Type K thermocouples in each meter control box, heated sample box, impinger umbilical connector, XAD resin trap and sample probe are calibrated against ASTM mercury-in-glass thermometers at two or more points: an ice bath, ambient temperature and a boiling water bath.
- **Dry Gas Meters** [QA Handbook Section 3.4.2, pp. 1-12 - calibrate against a wet test meter or calibrated orifice. Acceptance criteria - pretest Yi = Y ± 0.02; post test Y = ± 0.05 Yi.] Dry gas meters for all sampling trains are calibrated using critical orifices. The procedure entails four runs using four separate critical orifices running at an actual vacuum 1-2 in. greater than the theoretical critical vacuum. The minimum sample volume required per orifice is 5 ft^3. Meter boxes are calibrated annually and then verified by use of the alternative Method 5 post-test calibration procedure. This procedure is based on the principles of the optional pretest orifice meter coefficient check outlined in Section 4.4.1 of Method 5. The average Y-value obtained by this method must be within 5% of the initial Y-value.
- **Field Balance** The analytical balance used in the field to determine initial and final silica gel weights is calibrated against Class M weights provided by the Mettler Corporation.
- **Field Barometer** [QA Handbook Section 3.4.2, pp. 18-19 - compare against a mercury-in-glass barometer or use Airport Station BP and correct for elevation. Acceptance criteria - ± 0.02 in. Hg; post-test check - same.] Most ENSR field staff own watches that record barometric

pressure. In the absence of such an instrument, BP readings will be obtained from the closest airport and corrected for elevation (-0.10 in. Hg per 100-ft of elevation increase as per Section 6.1.2 of EPA Reference Method 5).

- **CEM Equipment and Instrumentation** ENSR's CEM equipment is housed in a trailer that is transported to the test site and set up adjacent to the sampling location. All equipment (analyzers, calibration gases and ancillary equipment) is thoroughly checked prior to each job and the appropriated calibration standards are procured. Daily calibrations and other instrument bias checks are performed in accordance with the specific method followed.

ENSR's field equipment is calibrated annually or more often if problems occur. Copies of all calibration data will be brought to the test site and a copy will be made available to the test observer, if requested. All calibration data are subsequently included in the final report appendices.

7.0 Analytical Methods and Procedures

This section delineates the analytical protocols that will be followed to analyze samples during this test program. The methods cited will be followed as written unless specific modifications are made in the laboratorys' standard operating procedures (SOPs). For this program, we have included several laboratory-specific SOPs for certain methods (as discussed below) in **Attachment D**. Samples of liquid waste feed and stack gas will be collected and analyzed for the parameters previously discussed using the appropriate laboratory protocols detailed in this section and as outlined previously in Section 6.0 of the CPT Plan. All referenced EPA methods will be from SW-846, 3rd edition, unless noted otherwise. **Table 7-1** provides a detailed summary of the overall sampling and analysis program including the number of field, QA/QC and audit samples anticipated for the program.

Table 7–1 Detailed Overview of Sampling and Analysis Program

Sample Matrix and Sampling Method	Analytical Parameters	Analytical Method	Lab **(a)**	Total Samples Analyzed				
				Total Runs	Field Blanks	Audit	Lab QC	Total
Waste Feed --								
(Grab/Comp)	Heat Content	ASTM D 240	TA	4	0	0	1	5
	Ash Content	ASTM D 482	TA	4	0	0	1	5
	Density	ASTM D 1963	TA	4	0	0	1	5
	Viscosity	ASTM D 445	TA	4	0	0	1	5
	Total Chlorine	TA SOP WC-0016	TA	4	0	0	1	5
	Water Content	ASTM E 203	TA	4	0	0	1	5
	VOCs **(b)**	EPA M 8015B / 8260B	TA	4	0	0	1	5
	Metals **(c)**	EPA M 6010B / 7471A	TA	4	0	0	1	5
Stack Flue Gas --								
EPA M 26A	Particulate	EPA M 5	ENSR	4	1	0	1	6
EPA M 26A	HCl and Cl2	EPA M 26A	TA	4	1	0	1	6
EPA M 29	Metals **(c)**	EPA M 6020 / 7470A	TA	4	1	1	1	7
EPA M 0023A	PCDDs/PCDFs	EPA M 0023A / M 8290	VISTA	4	1	0	1	6
EPA M 0010	PAHs	CARB M 429	VISTA	4	1	0	1	6
EPA M 0010	SVOCs **(d)**	EPA M 8270C	TA	4	1	0	1	6
EPA M 0030 (VOST)								
VOST Tube Pairs	VOCs **(e)**	EPA M 5041A / 8260B	ATL	16	4	3	2	25
VOST Tube Prep		EPA M 5041A	ATL	32				32
VOST Condensate	VOCs **(e)**	EPA M 8260B	ATL	4	1	0	1	6
EPA M 3A	O_2 and CO_2	EPA M 3A (CEM)	ENSR	4	0	0	0	4
EPA M 25A	THC	EPA M 25A (CEM)	ENSR	4	0	0	0	4
Facility CEM	O_2 & CO	Facility CEM QA Plan	Bostik	4	0	0	0	4

(a) ATL = Air Toxics Ltd., Folsom, CA
TA = Test America, Inc. at various locations
VISTA = Vista Analytical Laboratories, Inc., El Dorado Hills, CA
(b) Target VOCs in waste distillate include: toluene (POHC), methyl ethyl ketone, xylene, tetrahydrofuran, ethyl acetate, methanol, ethylene glycol, and diethylene glycol.
(c) Metals include : Sb, As, Ba, Be, Cd, Cr, Pb, Hg, Ni, Ag and Tl
(d) Target SVOCs in stack gas include full method scan plus top 10 TICs; see Attachment E for detailed list.
(e) Target VOCs in stack gas include full method scan plus top 10 TICs; see Attachment E for detailed list.
All VOST tube pairs will be desorbed individually to assess breakthrough.

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7.1 Analysis of Waste Distillate Feed Material

7.1.1 Chemical and physical properties

Analyses to determine the chemical and physical properties of the waste feed material will be performed using appropriate ASTM or EPA analytical methods as outlined in the table below.

Parameter	Analytical Procedure
Total chlorine/chlorides	Test America SOP WC-0016 (incorporates elements of ASTM E 442 and EPA Methods 5050 / 9056. Copy of the SOP included in Attachment D)
Ash content	ASTM D 482
Moisture	ASTM E 203 (Karl Fischer)
Viscosity	ASTM D 445
Density	ASTM D 1963
Heat content	ASTM D 240

Quality assurance requirements for the determinations of chemical and physical properties are summarized in **Table 7-2**.

Table 7–2 Summary of QA/QC Procedures for Chemical/Physical Properties in Waste Feed

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Initial analysis standards	Prior to sample analysis	Instrument dependant
	Continuing calibration standards	Before and after sample analysis; once every 20 samples	90%-110% of expected value
Interference check	Interference check sample	Before and after sample analysis	90%-110% of true value
Accuracy - calibration	Analysis of calibration check standard	After every calibration	90%-110% of expected value
Accuracy - spikes (chlorine)	Spike sample at twice sample level	Once every 20 samples	90% to 110% of spiked value
Precision	Duplicate preparation and analysis of at least one run's samples	Once every 20 samples	Range < 30% if sample result above lowest standard
Blank	Method blank carried through all sample preparation and analysis steps	Once every 20 samples	Below detection limit

7.1.2 Metals in Waste Feed Material

Target metals for the waste distillate include: antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver and thallium. Analyses for metals other than mercury will be performed using inductively coupled plasma - mass spectroscopy (ICP-MS) as described in EPA Method 6020. Mercury analysis will be performed using either EPA Method 245.1 or 7471A. Quality assurance requirements for the analyses of metals in waste feed samples are summarized in **Table 7-3**.

Table 7–3 Summary of QA/QC Procedures for Metals in Waste Feed Samples

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Initial analysis of standards at different concentration levels	At least once before sample analysis	Instrument-dependent. Linear correlation coefficient of standard data >=0.995
	Continuing mid-range calibration standard	Before and after sample analysis	90% to 110% of expected value.
Interference check	Interference check sample	Before and after ICP-MS analysis	80% to 120% of expected value
Accuracy – calibration	Analysis of calibration check standard	After every initial calibration	90% to 110% of expected value
Accuracy – spikes	Aliquot of one sample from a run spiked with analytes at 3 times the detection limit or twice the sample level prior to digestion	One per sample matrix	70% to 130% recovery
Precision	Duplicate preparation and analysis of one sample from each matrix	One per sample matrix	Range < 35% if sample result above lowest standard
Blank	Method blank carried through all sample preparation and analysis steps	Once per sample batch	Below detection limit
CVAA = cold vapor atomic absorption **ICAP = inductively coupled argon plasma**			

7.1.3 Volatile Organics in Waste Feed Material

The waste feed will be analyzed for expected volatile organic constituents (toluene, MEK, xylene, tetrahydrofuran, ethyl acetate, methanol, ethylene glycol and diethylene glycol) following EPA Methods 8015B and 8260B. Quality assurance requirements for the analyses of organics in waste feed are summarized in **Table 7-4.**

Table 7–4 Summary of QA/QC Procedures for Organics in Waste Feed Samples

Quality Parameter	Method Determination	Frequency	Target Criteria
Blanks – verify no lab contamination and system control	Lab blanks	Daily, before analysis of samples and in-between high-level samples	Less than lowest standard
Initial calibration of GC/MS	5 standards bracketing expected level	Prior to sample analysis	Variability of average RRF = <= 20% RSD
Calibration check standard	Mid-level standard different from initial calibration standards	After each standard preparation and initial calibration	Within continuing calibration criteria
Continuing calibration	Mid-level standard	Before and after sample analysis (every 10 samples for GC)	RRF within ±15% of initial calibration (GC) RRF within ±20% of initial calibration (GC/MS)
Consistency in chromatography	Monitor internal standard retention time and area	Every sample, standard and blank	Retention time within ±30 sec of last calibration check; area within –50% to + 100% from last daily calibration check
Accuracy - spikes	One sample from each matrix spiked prior to preparation with POHC at twice the expected level	One per sample matrix	50% to 130% recovery
Precision - POHC	Duplicate preparation and analysis of one sample from each matrix	One per sample matrix	< 35% range
Blanks	Method blank carried through all sample preparation steps	Once per sample batch	< 5% of sample levels

RSD = Relative Standard Deviation

RRF = Relative Response Factor

7.2 Analysis of Stack Gas Samples

7.2.1 Particulate Matter

Gravimetric analyses will be performed on samples collected from the Method 26A sampling train. Weights will be obtained on the front-half acetone rinse and particulate filter using a Mettler H35 analytical balance. Balance accuracy is checked by using Class "S" standard weights before and after tare weighings and sample determinations.

7.2.2 Hydrogen Chloride and Chlorine

Impinger samples from stack gas sampling will be analyzed by ion chromatography in accordance with EPA Method 26A. QA/QC procedures for stack gas analyses for chloride are presented in **Table 7-5**.

Table 7–5 QC Summary for Analysis of Hydrogen Chloride and Chlorine in Stack Gas Samples

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration (Qualitative)	Average retention time	Every calibration curve	Within retention time window of standards
Calibration (quantitative)	Initial calibration with a minimum of four standards	At least once before sample analysis	Linear correlation coefficient > 0.995
	Continuing calibration	Every 10 samples and at end of day	90% - 110% of theoretical concentration
Accuracy (calibration)	Laboratory control sample	After every initial calibration and before sample analysis	90% - 110% of true value
Accuracy (spikes)	Matrix spikes	Once per test	70% - 130% recovery
Precision	Duplicate analyses	All samples	RPD < or = 35%
Field Reagent Blanks	Collection of method-specified volumes of each reagent	Once per test	Less than 5% of sample levels
Blank	One method blank carried through sample preparation and analysis	Once per test	Less than 5% of sample levels
RPD = Relative Percent Difference			

7.2.3 Metals

Target analytes for the program include arsenic, antimony, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver and thallium. Each sampling train will be prepared and analyzed in accordance with EPA Reference Method 29. Test America SOPs for analysis of Method 29 samples are included in **Attachment D**. From each sampling train, seven individual samples are generated for analysis. The first two samples, labeled Fractions 1A and 1B consists of the digested sample from the front half of the train, consisting of the particulate filter and the front-half nitric acid probe rinse. Fraction 1A is for ICP analysis and Fraction 1B is for mercury analysis. Fractions 2A and 2B consist of digestates from the moisture knock out and HNO_3/H_2O_2 impingers 1, 2, and 3. Fraction 2A is for ICP analysis and Fraction 2B is for mercury analysis. Fractions 3A, 3B and 3C consist of the impinger contents and rinses from the empty and permanganate impingers 4, 5, and 6. These fractions will be analyzed for mercury.

Analyses for metals other than mercury will be performed using Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) as described in EPA Method 6020. Mercury analysis will be performed using EPA Methods 7470A. All quality control procedures, including the interference check standard, will be followed as described in the respective method. Instrument calibration will be performed daily in accordance with the procedures described in the methods and the manufacturer's instructions. The calibration is verified daily by analysis of an instrument check standard prepared from an EPA quality control concentrate or other independent standard. **Table 7-6** summarizes the QA/QC requirements for the stack metals analyses.

Table 7–6 QC Summary for Analysis of Metals in Stack Gas Samples

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Initial analysis of standards	Daily	Analysis of calibration check standard within 10% of true value
	Continuing mid-range calibration standard	At least once before and after sample analysis	90-110%
	Continuing calibration blank	With continuing calibration standard	Subject to interpretation
Accuracy - ICV	Analysis of calibration check standard	After every initial calibration	90% to 110% of true value
Accuracy - filters	Analysis of EPA audit filters, if provided	Once per test	70% to 130% of reference value
Accuracy	Post-digestion spikes	Once per test	70% to 130% recovery
Precision	Post-digestion spikes	Once per test	RPD < or = 35%
Blanks	Field Reagent Blanks and Method Blanks	One each per test	Evaluated on case by case basis
RPD = Relative Percent Difference			

7.2.4 Target Volatile Organics in Stack Gas Samples

Stack gas samples will be analyzed for volatile organics using EPA Method 5041A (VOST tubes) and EPA Method 8260B (condensate). Target analytes will include the full method scan plus the top 10 non-target compounds, which will be denoted as tentatively identified compounds (TICs). The target POHC for this analysis is toluene. The SOP used by Air Toxics, Ltd for the VOST analyses is provided in **Attachment D**.

Analysis — The samples collected from each VOST run will consist of a Tenax cartridge, a Tenax/charcoal backup cartridge, and a flue gas condensate. Cartridges will be desorbed and analyzed for volatile organics using the thermal desorption GC/MS procedures specified in Method 5041A of SW 846. Condensate samples will be analyzed using Method 8260B. All VOST tube pairs will be individually desorbed to assess compound breakthrough.

All QA/QC requirements of EPA Method 5041A for instrument calibration and performance will be met prior to sample analyses, including:

- System performance checks using the five system performance check compounds (SPCCs) will be conducted initially and after every 12 hours of analysis. The minimum response factors for the volatile SPCCS will be 0.100 (0.300 for chlorobenzene and 1,1,2,2-Tetrachloroethane).
- Daily calibration of the system, including evaluation of the internal standard responses and retention times in the check calibration standard. Performance criteria specified in the method will be used to determine whether the system has malfunctioned. If samples are analyzed under conditions of malfunction, an evaluation of the impact of that malfunction on data quality will be performed, with the results of the investigation presented in the final report.

Thermal desorption will be conducted using an automated desorption unit which is designed to accommodate sorbent cartridges in series. The desorbing gas is plumbed to direct flow through each pair of traps, then through a purge vessel to trap desorbed water and, finally, onto the head of a smaller sorbent column which is located in a purge and trap device. The volatile components adsorbed onto the secondary trap are then thermally desorbed onto the GC by heating the trap as detailed in Method 5041A. Prior to analysis, the volatile surrogate compounds and internal standards listed in the method will be flash vaporized onto each Tenax cartridge set.

The analytical performance check for the designated POHC will be completed prior to the program in accordance with SW-846 Method 0030, Section 7.1 by the laboratory conducting the analyses. The amount spiked for this analysis will be at or near the anticipated "critical level" for the POHC for this project (~500 ng) per component and will be recognized as having passed the check if the recovery is within 50% - 150% of the expected values.

Calibration for Method 5041A - The GC/MS will be tuned to BFB at the beginning of each 12-hour analysis sequence, applying the acceptance criteria for key ion abundance listed in the method.

Upon compliance with all system criteria, the GC/MS will be initially calibrated at a minimum of three to five calibration levels by analyzing sets of adsorbent tubes spiked with the volatile POHCs, internal standard and surrogates.

Calibration standards for the POHC must cover the range of concern for DRE demonstration. Method 0030 describes the calibration procedure for spiking all compounds of interest onto the tubes themselves, then desorbing for analysis. Response factors for each compound are determined and used for the calculation of analytical results. QA/QC requirements for VOST analyses are provided below in **Table 7-7**.

Table 7–7 QA Objectives for VOST Analyses

Quality Parameter	Method Determination	Frequency	Target Criteria
Blanks – sample integrity and field contamination	Field blanks, 1 pair of traps	One pair per sampling day	Less than lowest standard
Blanks – verify no contamination from storage / shipment	Trip blanks, 1 pair of traps	One pair per shipment	Less than lowest standard
Blanks – verify no lab contamination and system control	Lab blanks, 1 pair of traps	Daily, before analysis of samples and in-between high-level samples	Less than lowest standard
Initial calibration - GCMS	Lowest level of calibration curve equal to reporting limit for compound	Prior to sample analysis	CCCs ≤ 30% RSD
Continuing calibration	Standard at or near the midlevel	Prior to sample analysis, then every 12 hr, or after sample set	≤ 25% difference for CCCs
Consistency in chromatography	Monitor internal standard retention area	Every sample, standard and blank	Retention area within 60% to 140% from last daily calibration check
Precision and accuracy	Replicate analysis of 3 traps spiked at the expected level of 99.99% DRE	Demonstrated prior to sample analysis	50% - 150% recovery
Continuing accuracy check	Surrogate spikes	Every sample	50% to 150% recovery
Verification of VOST system accuracy	Analysis of samples from EPA audit cylinder, if provided	Once per test	Within 50% - 150% of certified concentration
VOST condensate: precision and accuracy	Surrogate spikes	All condensate samples	50% to 150% recovery
Breakthrough determination	Separate analysis of front and back traps	All VOST tube pairs	Quantity on TX/C must be < 30% of amount on TX trap - does not apply when < 75 ng on TX/C trap
RSD = Relative Standard Deviation **CCCs = Calibration Check Compounds**			

7.2.5 PCDDs/PCDFs and PAHs in Stack Gas

Stack flue gas samples collected using the combined Method 0023A / 0010 sampling train will be analyzed accordingly for the specified target parameters. Each sampling train will be prepared and split appropriately for the designated analyses. The Vista Analytical SOP for the combined train is included in **Attachment D** and also includes a schematic of the analytical regimen for the combined PCDD/PCDF/PAH sampling train. The analysis features separate front-half and back-half analysis for PCDDs/PCDFs and combined front-half and back-half analysis for PAHs.

Briefly, the XAD and filter will be spiked with internal standards for PCDDs/PCDFs and PAHs and then sequentially extracted with methylene chloride and toluene. The methylene chloride and acetone rinses will be combined and added to the methylene chloride Soxhlet extract. This combined sample will be concentrated and split. The portion allocated for PAH analyses will be combined with the acid/base neutral extracts of the impinger contents. The portion allocated for PCDD/PCDF analysis will be combined with an appropriate fraction of the toluene extract.

Method 0023A analyses (which include high resolution GC/MS as per EPA Method 8290) incorporate five isotopically labeled PCDD and PCDF field surrogates and nine labeled PCDD/PCDF internal standards. The field surrogates are spiked into the XAD resin prior to field sampling; their recoveries are monitored to assess overall method accuracy and precision. The internal standards are added to the combined XAD/filter/rinse concentrate sample at a level of 2,000 pg/sample prior to Soxhlet extraction. These internal standards are used for direct quantification of all surrogate and native PCDD/PCDF species. The addition of these standards prior to the extraction and cleanup procedures permits internal correction for any losses of target analytes that might occur during the preparation steps.

Method 8290 details instrument tune, GC column performance and instrument calibration requirements for the analysis of stack gas samples by high resolution gas chromatography/high resolution mass spectrometry. Instrument calibration will be performed for all 15 2,3,7,8- substituted PCDD and PCDF isomers; data will be reported for each of these target analytes and for the total dioxins and total furans at each level of chlorination from Cl_4 through Cl_8.

Additional analyses for target PAHs will be conducted in accordance with California Air Resources Board (CARB) Method 429. This method employs isotopically labeled internal standards and HRGC/HRMS analysis for target PAHs. The use of the method is intended to supplant similar data provided by Method 8270C data for target PAHs, because the method provides lower detection limits for use in the SLRA.

QA/QC requirements for these analyses are summarized in **Tables 7-8 and 7-9**.

Table 7–8 QA Objectives for PCDD/PCDF Analysis of Stack Gas Samples

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Five-level calibration curve; continuing calibration standard	At least once; continuing calibration check at beginning of each 12-hr shift	Initial: <=20% RSD for unlabelled standards <=30% RSD for internal standards S/N ratio >=2.5; Isotope ratios within control limits Continuing: <=20% of ICAL for 17 unlabelled stds <=30% of ICAL for internal standards S/N ratio >=2.5; Isotope ratios within control limits
Accuracy-calibration	Analysis of calibration check	After every initial calibration	80% - 120% of theoretical value
Accuracy-surrogates	Spiked into samples prior to sampling	Every sample	70% - 130% recovery
Accuracy-internal standards	Spiked into samples prior to extraction and analysis	Every sample	40%-135% recovery for tetra – octa homologs
Accuracy – audit samples	Prepared and analyzed along with program samples	Presented by the regulatory agency	Determined by regulatory agency
Blanks	Method blank for each component Field blank	One per batch of samples Once per test	ND or <5% of field concentration Evaluated on a case-by-case basis
Mass Spectrometer Performance	Section 8.2.2 of Method 8290	At beginning of each 12-hr period	Static resolving power of 10,000 (10% valley definition)
Qualitative Identification	Retention Time and GC Column Performance	Every sample	Compliance with Section 8.2.1 of Method 8290

S/N = Signal to Noise Ratio

RSD = Relative Standard Deviation

Table 7–9 QA Objectives for PAH Analysis of Stack Gas Samples

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Five-level calibration curve	Once before samples analyzed	Initial: <=30% RSD for unlabelled standards <=30% RSD for internal standards S/N ratio >=10
	Continuing calibration	Beginning and end of every 12-hour analysis shift	Continuing: <=30% RPD of ICAL for unlabelled stds <=30% RPD of ICAL for internal stds S/N ratio >=10 Internal standard retention times ± 30 sec of initial calibration mean All confirmation ions present
Accuracy-surrogates	Spiked into media before sampling	All samples	50% - 150% recovery
Accuracy-internal standards	Spiked into samples prior to extraction and analysis	Every sample	50% - 150% recovery
Accuracy-spikes	Laboratory control samples prepared by spiking XAD with independently prepared solution of analytes	Duplicate preparation and analysis for each set of 15 or less samples	S/N >10 for all analytes 50% - 150% recovery 50% RPD
Mass Spectrometer Performance	Section 7.3.3 of CARM Method 429	At beginning and end of each 12-hour period	Static resolving power of 8,000 (10% valley definition)
Blanks	Blank XAD extracted and analyzed with samples	Once for each set of 15 or fewer samples	Less than PQL or 5% of analyte concentration in field samples
RSD = relative standard deviation; RRF = relative response factor **RPD = relatiave percent difference; PQL = practical quantitation limit**			

7.2.6 Semivolatile Organics in Stack Gas Samples

Stack flue gas samples collected using the Method 0010 sampling train for semivolatile organics as specified in EPA Method 8270C. Target SVOCs will include all analytes listed in Method 8270C (except PAHs, Aroclors and organochlorine pesticides) where Methods 3540/3541 are listed as the appropriate preparation technique. Method 3542, which is the preferred preparation method, will be used for these analyses. In addition to the analysis for target compounds, data will be searched against the EPA/NIH library of mass spectral data using the instrument automated software routine for tentative identification of up to approximately 10 additional compounds. Up to 10 peaks with areas of 10% or more of the nearest internal standard will be searched. Estimated quantitation of these compounds will be based upon the area of the compound compared to that of the nearest internal standard, using a relative response factor of 1 unless the TIC is structurally similar to a closely-eluting target compound for which a response factor is already established.

All components of the Method 0010 sampling train will be submitted to the laboratory for extraction and analysis as specified in EPA Method 3542. Analysis for semivolatile organics will be performed by low resolution mass spectrometry following the analytical protocol of SW-846, Method 8270C. Surrogates will be added prior to extraction to monitor analytical accuracy. Method 8270C surrogates to be used are: 2-Fluorobiphenyl, 2-Fluorophenol, Phenol-d_5, Terphenyl-d_{14}, 2,4,6-Tribromophenol and Nitrobenzene-d_5. Method 8270C internal standards to be used are: 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12} and Perylene-d_{12}.

QA/QC requirements for SVOC analyses are summarized in **Table 7-10**.

Table 7–10 QA Objectives for SVOC Analysis of Stack Gas Samples

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Five-level calibration curve; continuing calibration standard	At least once; at the beginning of day; continuing calibration once every 12 h and at end of day	<30% RSD of avg RRF; within 30% of avg RRF from calibration
Accuracy (calibration)	Analysis of calibration check	After every initial calibration	70% - 130% of theoretical value
Accuracy (surrogates)	Isotopically-labeled compound spiked into samples prior to sampling and/or analysis	Every SVOC sample	Nitrobenzene d5 - 35-122% 2-Fluorobiphenyl – 34-115% Terphenyl d14 – 28-132% Phenol d5 – 15-124% 2-Fluorophenol – 19-100% 2,4,6-Tribromophenol – 33-130%
Accuracy (spike)	Representative SVOCs spiked onto blank XAD trap	Once per trial burn	Phenol – 26-90% 2-Chlorophenol – 25-102% N-Nitrosodipropylamine 41-126% 4-Cl-3-methylphenol 26-103% Acenaphthene 31-137% 4-Nitrophenol 11-114% 2,4-Dinitrotoluene 28-89% Pyrene 35-142%
Precision (surrogates)	Same as for accuracy - surrogates pool results for each SVOC component	Every SVOC sample	<40% RPD of surrogate recovery. If more than 3 determinations – RSD <35%
Blanks	Method blank for each SVOC	Once per batch of samples	Blank value <2 x DL. If greater, DL is changed to 1.5x blank level
	Field blank	Once per test	Evaluated on a case-by-case basis

RSD = relative standard deviation

RRF = relative response factor

RPD = relative percent difference

8.0 Quality Control Procedures

Quality control checks will be performed to ensure the collection of representative samples and the generation of valid analytical results for these samples. These checks will be performed by project participants throughout the program under the direction of the Project Manager and the QA Officer.

8.1 Field Sampling QC Procedures

QC checks for the process data collection and sampling aspects of this program will include, but not be limited to, the following:

1. Use of standardized data sheets, checklists and field notebooks to ensure completeness, traceability, and comparability of the process information and samples collected.

2. Field checking of standardized forms by the Field Team Leader and a second person to ensure accuracy and completeness.

3. Strict adherence to the sample traceability procedures.

4. Submission of field biased blanks.

5. Leak checks of sample trains before and after sample collection and during the test, when appropriate.

8.1.1 Equipment Inspection, Maintenance and Calibration

ENSR maintains a dedicated facility for storage, maintenance, repair and calibration of all field equipment. Prior to each job, project participants fully inspect and prepare all equipment that will be used.

Calibration of the field sampling equipment is performed in accordance with procedures recommended by the manufacturer and as described earlier in Section 6.0. Copies of the calibration sheets will be available onsite during the field sampling program for inspection, will be kept in the project file and will be incorporated as an appendix in the final report. Calibrations will be performed as described in the EPA publication "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods;" Section 4.2.1 presents acceptance limits.

8.1.2 Sampling Equipment QC Checks and Frequency

Leak checks of the sample trains will be conducted in accordance with the protocol called out for each method. Leak checks will be conducted prior to and at the end of sample collection and during the test run, if the sampling train is disassembled for any reason or if the port change requires extensive movement of the train.

Field blanks of reagents and collection media (deionized water, filters, impinger solutions, sorbent material, etc.) will be placed in appropriately cleaned and sized sample containers in the field and handled in the same way as actual field samples, to provide a QC check on sample handling.

For this program, sample collection QC checks and frequency for samples to be analyzed in the laboratory are listed below:

- One field blank VOST train (i.e., one set of blank traps exposed to conditions analogous to actual samples) for each sampling day and one trip blank for the overall program.
- Three pairs of VOST tubes collected from any EPA audit cylinder provided
- One blank Method 26A (PM / HCl / Cl_2) sampling train
- One blank Method 29 (metals) sampling train
- One blank Method 0023A / 0010 (PCDDs / PCDFs / PAHs) sampling train
- One blank Method 0010 (SVOCs) sampling train

8.2 Analytical QC Procedures

The Quality Control program for laboratory analysis makes use of a number of different types of QC samples to document the validity of the generated data. The following types of QC samples will be used during the program.

8.2.1 Quality Control Samples and Blanks

Method Blanks

Method blanks contain all the reagents used in the preparation and analysis of samples and are processed through the entire analytical scheme to assess spurious contamination arising from reagents, glassware, and other materials used in the analysis.

Calibration Check Samples

One of the working calibration standards which is periodically used to check that the original calibration is still valid.

Laboratory Control Samples (LCS) or Blank Spikes

These samples are generated from spikes prepared independently from the calibration concentrates. The LCS are used to establish that an instrument or procedure is in control. An LCS is normally carried through the entire sample preparation and analysis procedure also.

Surrogate Spikes

Samples requiring analysis by GC/MS are routinely surrogate-spiked with a series of deuterated analogues of the components of interest. It is anticipated that these compounds would assess the behavior of actual components in individual program samples during the entire preparative and analysis scheme.

The percent recovery for each surrogate will be calculated in accordance with method-specific procedures. Any values which fall outside the target QC limits described in the applicable analytical method will be flagged. Some of these recovery values may be outside the QC limit owing to matrix interferences. The following guidelines will be used:

1. All recovery data are evaluated to determine if the QC limits are appropriate and if a problem may exist even though the limits are being achieved (e.g., one compound that is consistently barely within the lower limit).

2. Any recovery data which are outside the established limits are investigated. This evaluation will include an independent check of the calculation.

3. Corrective action will be performed if any of the following are observed:

    - All recovery values in any one analysis are outside the established limits, where one analysis is considered to be one sample analyzed by one method,
    - Over 10 percent of the values for a given sample delivery group are outside limits, or
    - One compound is outside the limits in over 10 percent of the samples.

An analysis batch is defined as a group of ten or fewer samples carried through the entire preparation and analysis procedure in one batch.

Reagents used in the laboratory are normally of analytical reagent grade or higher purity; each lot of acid or solvent used is checked for acceptability prior to laboratory use. All reagents are labeled with the date received and date opened. The quality of the laboratory deionized water is routinely checked. All glassware used in the sampling and analysis procedures will be pre-cleaned according to the method requirements. Standard laboratory practices for laboratory cleanliness, personnel training and other general procedures are used. The results of these quality control procedures will be included in the final report.

8.2.2 Quality Control of Sorbents

Sorbents used for the organic sampling trains are provided by the laboratory after QC verification has been performed following recommended procedures in each applicable method. Additional details on sample media preparation were provided previously in Section 5.2.1.

9.0 Performance and System Audits

The External Quality Assurance Program includes both performance and system audits as independent checks on the quality of data obtained from sampling, analysis, and data gathering activities. Every effort is made to have the audit assess the measurement process in normal operation. Either type of audit may show the need for corrective action.

The sampling, analysis, and data handling segments of a project are checked in performance audits. A different operator/analyst prepares and conducts these audit operations to ensure the independence of the quantitative results.

ENSR will follow the guidance provided by EPA Region 6 in their *"Standard Operating Procedure for Use of Performance Audits During Regulatory Testing"*, dated July 2004. This guidance provides information pertaining to the procurement of a VOST audit cylinder and a Method 29 (mercury) audit sample that are planned for this program. Any such audit samples presented by the regulatory agencies will be analyzed along with program samples, by the appropriate lab and at the same time as all other samples. Results will be reviewed by the subcontractor laboratory and QC personnel.

If the regulatory agency advises facility program manager that audit results fall outside of acceptable ranges, the analytical data will be further reviewed for error in conjunction with the agency. If a simple, correctable error is found (e.g., an arithmetic error), correction will be made and results resubmitted. If no error is found, an investigation into other causes of the failure (e.g., lack of sample integrity) will be conducted and results evaluated in terms of the impact on sample data integrity.

10.0 Preventive Maintenance

This section provides pertinent information for field sampling equipment as well as a listing of all critical facility equipment necessary to maintain permitted operating conditions and to demonstrate continuing compliance. Information is provided for preventive maintenance and schedules and spare parts for key equipment and instrumentation.

10.1 Field Sampling Equipment

The field sampling team follows an orderly program of positive actions to prevent the failure of equipment or instruments during use. This preventive maintenance and careful calibration helps to ensure accurate measurements and minimal field delays.

All equipment that is scheduled for field use is calibrated as outlined previously in Section 6.0. Prior to each field use for a specific project, the equipment is cleaned and checked to ensure it is in good working order. An adequate supply of spare parts and sample train glassware is brought to each site to minimize downtime and field sampling delays. Any equipment that does experience problems is appropriately tagged in the field to ensure that it is repaired upon return to the office. In addition, the ENSR equipment facility is located withib 30 miles of the Bostik facility and thus any spare parts not readily available onsite can be obtained quickly, if necessary.

10.2 Facility Equipment and Instrumentation

The Bostik facility follows an orderly preventative maintenance program for the Polyester Distillate Burner to ensure continued compliance with the regulations. This program includes the following frequencies:

<u>Daily:</u>

- Waste feed piping, pump and tank integrity inspections
- CEMS calibration

<u>Weekly:</u>

Testing of AWFCO alarms to check operation of computer systems

<u>Quarterly:</u>

Conduct CEMS calibration error test

<u>Annually:</u>

Conduct CEMS 7-day calibration drift, calibration error and response time tests

11.0 Procedures Used to Assess Data Precision and Accuracy

The QA activities implemented in this program will provide a basis for assessing the accuracy and precision of the analytical measurements. Section 8.0 of this QAPP discusses the various QA activities that will generate the accuracy and precision data for each sample type. A generalized form of the equations that will be used to calculate accuracy, precision and completeness follows.

11.1 Accuracy

Accuracy (calculated as percent recovery) will be determined using the following equation:

$$\textbf{\% Recovery} = \frac{(X - S)}{T} \, x \, 100$$

where:

X = experimentally determined concentration of the spiked sample

T = true concentration of the spike

S = sample concentration before spiking

11.2 Precision

Precision (calculated as percent relative difference) will be determined using the following equation:

$$\mathrm{Re}\,lative\ Percent\ Difference\ (RPD) = \left[\frac{(D_1 - D_2)}{\left\{ \frac{D_1 + D_2}{2} \right\}} \right] x\,100$$

where:

D_1 and D_2 = results of duplicate measurements or standard deviation relative to the average value expressed as relative standard deviation:

Relative standard deviation will be expressed as follows:

$$\text{Relative Standard Deviation (\% RSD)} = \quad = \left\{ \frac{\sigma_{(n-1)}}{\bar{x}\,(x_1 \cdots x_n)} \right\} \; x \quad 100$$

where:

$\sigma_{(n-1)}$ = standard deviation of the sample data

n = number of replicates

$x_{(x1..xn)}$ = arithmetic mean of the sample data

11.3 Completeness

Data completeness is a measure of the extent to which the database resulting from a measurement effort fulfills objectives for the amount of data required. For this program, completeness will be defined as the percentage of valid data for the total valid tests. Completeness is assessed using the following equation:

$$Completeness\ (\%) = \left[\frac{D_r}{D_c} \right] \; x \quad 100$$

where:

D_r = number of samples for which valid results are reported

D_c = number of valid samples that are collected and reach the laboratory for analysis

The completeness objective will help to evaluate the accuracy and precision of the analytical measurements.

12.0 Corrective Actions

Each sampling and analysis method will be monitored for precision, accuracy and compliance with the QA/QC performance requirements of the method as part of routine performance monitoring and system and performance audits. Where test information, QA/QC data, sample analytical results or sample completeness do not conform to the defined program goals, potential nonconformance will be reported to the QAO. The QAO is responsible for deciding whether the stated DQOs can be achieved either by correcting the data using physical principles or other appropriate techniques to account for the nonconformance or by explicitly excluding the specific data point. Any nonconformance will be resolved with input from regulatory representatives.

For this CPT program, a minimum of three sets of test data (i.e., three valid test runs) are required. Plans have been made to also collect a fourth set of data to ensure that sufficient data are collected. The fourth set of samples will be archived until it is certain that the results from the first three runs have been successfully analyzed. If it is determined during or after the field test program that operational parameters for a single test run have not met stated objectives, the samples representing the nonconforming test run may be discarded and analyses conducted on the archived samples from the fourth test run. In the field, the Bostik project manager, the ENSR QAO and the ENSR Field Team Leader in consultation with the onsite regulatory representative(s) will make a preliminary determination of the conformance of each test run and the ultimate disposition of the samples from each run. The results of all samples analyzed will be reported in the final project report.

The basis and resolution of any QA nonconformance issues will be documented in the final report. If during routine performance monitoring, system audits or performance audits, weaknesses or problems are discovered, corrective action will be initiated immediately. Corrective action will include, but not necessarily limited to the following:

- recalibration of instruments using freshly prepared calibration standards;
- replacement of lots of solvents or other reagents that have given unacceptable blank values;
- additional training of laboratory personnel in correct implementation of sample preparation and analysis methods; and/or
- reassignment of personnel.

Whenever a long-term corrective action is necessary to eliminate the cause of the nonconformance, the following closed-loop corrective action system will be used. As appropriate, the field team leader, the project manager or the QAO will ensure that each of these steps is followed:

- The problem is defined.
- Responsibility for investigating the problem is assigned.
- The cause of the problem is investigated and determined.
- A corrective action to eliminate the problem is determined.

- Responsibility for implementing the corrective action is assigned and accepted.
- The effectiveness of the corrective action is established and the corrective action is implemented.
- The fact that the corrective action has eliminated the problem is verified.

13.0 Data Reduction, Validation and Data Reporting

Specific QC measures will be used to ensure the generation of reliable data from sampling and analysis activities. Proper collection and organization of accurate information followed by clear and concise reporting of the data is a primary goal in all such projects.

13.1 Field Data Reduction

Attachment B presents the standardized forms that will be used to record field sampling data. The Field Team Leader and the QAO will review the data collected from each train in its entirety in the field. Errors or discrepancies will be noted and dealt with accordingly. Both the Field Team Leader and the QAO have the authority to institute corrective actions in the field. Field data reduction (checking of valid isokinetic sampling rate and other sampling parameters) is done with a laptop computer using standardized Excel spreadsheets. **Attachment C** provides both setup and recovery schematics and a description of solutions and reagents to be used in each impinger train required for the overall program. All sample recovery sheets will be checked for completeness.

13.2 Laboratory Data Reduction

Analytical results will be reduced to appropriate units by the laboratory using the equations given in the applicable analytical method. Unless otherwise specified, results from the analysis of waste feed and process samples for specific target constituents will be reported in units of mg/kg or % wt. Other parameters will be reported in standard units such as g/cc, Btu/lb, etc.

The laboratory typically reports results from the analysis of stack flue gas samples as total mass detected for the sample submitted. For those sample fractions where liquid impinger condensate is analyzed, the laboratory will measure the total liquid volume submitted and multiply by the measured concentrations of target analytes in these samples. The laboratories will report data as follows:

- Volatile organics (VOST) – total **ng** or **µg** collected
- Particulate matter - total **mg** collected in each fraction (front-half rinse and filter)
- All metals except mercury – total **µg** of each metal in the combined front-half and back-half sample train fractions
- Mercury –total **µg** in each sample train fraction
- PCDDs/PCDFs - total **pg** collected in the separate front-half and back-half sample train fractions
- PAHs - total **ng** collected in the combined front-half and back-half sample train fractions
- SVOCs - total **µg** collected in the separate front-half and back-half sample train fractions

A listing of all target analytes for the trial burn program is provided in **Attachment E**.

Each LSC will be responsible for reviewing all results and calculations and verifying the completeness of the data set. The laboratory reports submitted by each laboratory will include the following deliverables:

- Transmittal letter listing all samples and analyses and a case narrative identifying any difficulties associated with the analyses and any anomalous QA/QC results
- Copies of Chain of Custody Forms
- Sample Report forms with sample field and laboratory identifier, dates of sample preparation and analysis, analytical results and detection limits
- Method Blank results
- MS and MSD results (as applicable)
- Replicate sample analyses (as applicable)
- Laboratory Control Sample results

Reports for organics in stack samples will include the following additional information:

- Surrogate recoveries
- Summary of initial calibrations
- Continuing calibration summaries
- Instrument tunes
- Data Validation

13.3 Data Validation

Data validation is the process of reviewing data and accepting, qualifying or rejecting it on the basis of method-specific criteria. The independent project QAO will use validation methods and criteria appropriate to the type of data and the purpose of the measurement. Records of all data will be maintained, even that judged to be an "outlying" or spurious value.

Field sampling data will be validated by the Field Team Leader based on a judgment of the representativeness of the sample, maintenance and cleanliness of sampling equipment and the adherence to an approved, written sample collection procedure.

Analytical data will be validated by the subcontractor laboratory QC or supervisory personnel using criteria outlined in their laboratory-specific QA Plan and/or written SOPs. Results from field and laboratory method blanks, replicate samples and internal QC samples will be used to further validate analytical results. Analytical results on field blanks and replicate field samples are valuable for validation of sample collection also. QC personnel will review all subcontractor laboratory raw analytical data to verify calculated results presented.

The following criteria will be used to evaluate the field sampling data:

- Use of approved test procedures
- Proper operation of the process being tested

- Use of properly operating and calibrated equipment
- Leak checks conducted before and after test runs
- Use of reagents that have conformed to QC specified criteria
- Use of NBS traceable CEM calibration gases
- Proper chain-of-custody maintained
- All sample trains --check to ensure proper sample gas volume collected

The criteria used to evaluate the analytical data are as previously defined in Section 3.0 (data quality objectives) and the method-specific QA summary tables listed in Section 7.0.

13.4 Data Reporting

13.4.1 Preliminary Data Reporting in the Field

At the end of each day of testing, several types of data will be made available to all project participants and test observers. Recovery of each isokinetic sampling train will include spreadsheet calculations to determine proper isokinetic sampling rate, stack gas moisture content, temperature and flowrate and sample volume. These data will be reviewed for acceptability and made available to facility personnel and Agency staff. Additional preliminary results will also be provided for CEMS data as measured by both Bostik and ENSR. The polyester burner DAS will also provide preliminary summaries for key operating data such as waste distillate feed rate, natural gas feed rate, heat input rate, combustion chamber temperature and flue gas flowrate.

13.4.2 Preliminary Reporting of Results

In the weeks following test conclusion, all field data will be reviewed and spreadsheet data entry will be checked for accuracy and completeness. As laboratory data become available, emission calculations will be performed and results will be provided to Bostik and EPA. Most importantly, the results of any failed tests will be provided as soon as the data are thoroughly checked for accuracy and associated QC data are determined to be acceptable.

13.4.3 Final Data Report

The final report for this project will be a comprehensive data compilation that properly and logically documents and certifies all required test results. The report will include all of the required elements of a MACT NOC as outlined in Section 1.4.2 of the CPT Plan. ENSR plans to follow the guidance provided by EPA that defers to the suggested format as offered by the Louisiana Department of Environmental Quality (LDEQ) for a combined NOC and CPT report. As such, the report would be structured in a similar manner with sections delineated as follows:

- Summary of Test Results
- Introduction and Process Description
- Process Operating Conditions

- Feed Stream Sampling and Analysis
- Performance Test Results
- Risk Assessment Test Results
- Quality Assurance / Quality Control Documentation
- Proposed Permit Limits and OPLs
- Continuing Compliance Methods

Report appendices will also provide detailed supporting documentation as delineated in the above referenced LDEQ guidance. Appendices for the project report would include:

- Stack Sampling Report
- Feed Stream Sampling Report
- QA/QC Data Report
- Process Operating Data
- Sample Emission Calculations
- Field Logs
- Analytical Data Packages
- CMS / CEMS performance Evaluation Test Reports

13.4.4 Management of Non-Detects

There are several different scenarios regarding the handling of analytical data reported as ND in this program. First, for the purposes of determining compliance with feed rate limits that are calculated from analytical data, the full ND value (reporting limit) will be used.

Second, there is a special consideration regarding the handling of ND values when calculating PCDD/PCDF emission rates for purposes of compliance. As per the requirements outlined at 40 CFR 63.1208(b)(1)(iii) and 40 CFR 266, Appendix IX, Section 4.0, ND values in this case are treated as zero.

In general, the emission tables to be generated for the final report will perform all calculations using either a real value or the detection limit (i.e. reporting limit) for those parameters reported as ND, with the exception noted above for PCDDs/PCDFs. In essence, using the full detection limit in an emission calculation provides a worst-case assessment. Data to be used in the SLRA, however, may ultimately treat the data differently in accordance with specific risk guidance.

13.4.5 Oxygen Correction

In accordance with 63.1206(c)(2)(iii), the facility is required to identify a projected oxygen correction factor based on normal operations to be used during periods of startup and shutdown. Bostik does not presently envision the need to project any alternative correction factor, but this will be addressed in the SSMP to be prepared prior to the MACT compliance date. It should also be noted that all concentration-based emission results will be corrected to 7% oxygen in accordance with both the RCRA and MACT regulations.

13.4.6 Sampling Times and Calculation of Results

Stack gas concentrations for each applicable parameter will be calculated from laboratory results and field sampling data. The total weight of the analyte detected will be divided by the volume of gas sampled to provide emission concentrations. As stated above, all emission concentrations are further corrected to 7% oxygen for comparison to published standards.

13.4.7 Blank Correction

Two types of samples collected on this type of program are allowed to be blank-corrected in accordance with method-specific procedures. First, gravimetric analyses for PM follow the procedures outlined in EPA Reference Method 5. Section 3.2 of Method 5 allows acetone residue blank correction up to a maximum of 0.001% of the weight of the acetone (0.01 mg/g). Second, blank correction for reagent contamination for all metals analyzed is allowed as per Sections 9.1.6 and 12.0 of Method 29.

13.4.8 Rounding and Significant Figures

For purposes of final data reporting, we propose to follow the procedures outlined under 40 CFR 63.1217(d) with respect to rounding of emission results and use of significant figures. This regulation notes that for all emission parameters except DRE, you must perform intermediate calculations using at least three significant figures, but that you may round the resultant emission levels to two significant figures to document compliance. No rounding is permitted with respect to the reporting of DRE results.

14.0 Quality Assurance Reports

Each LSC will prepare a case narrative on QC activities associated with this project for inclusion with the raw data report. This report will detail the results of quality control procedures, problems encountered and any corrective actions taken which may have been required.

The project QAO will be responsible for ensuring the validity of all report discussions dealing with data quality and data usability. The final report will include a section summarizing QA/QC activities during the program. The QAO will be primarily responsible for preparing this section. This section will provide summary QA/QC results for method blanks, surrogate spikes and laboratory control spike recoveries. This section will evaluate overall data quality in terms of accuracy, precision and completeness. Any discrepancies or difficulties noted in program work, protocol deviations or documentation gaps will be identified and discussed. If any data are determined to be invalid for whatever reason, this will be stated in the executive summary portion of the report and also fully explained in the appropriate sections of the document. In addition, any data deemed to be invalid or suspect will be footnoted in the specific summary tables in the report.

ATTACHMENT A

Projected Emission Rates at Expected Reporting Limits

Table A-1
Facility: Bostik, Inc. - Middleton, MA
TEQ Emission Rate Predictions for PCDDs / PCDFs at their EDLs [a]

	Units	Stack Gas Assumptions		
Sample Volume	dscf	120.0		
Sample Volume	m^3	3.40		
O_2 Conc.	% v/v (dry)	5.00		
CO_2 Conc.	% v/v (dry)	11.50		
Stack Flowrate	dscfm	2,000		
PCDD / PCDF Parameters	TEF [b]	MDL pg/sample	ng/m^3 TEQ	% Contribution to ND TEQ
2,3,7,8-TCDD	1.0	< 4.96	< 1.5E-03	37%
TOTAL TCDD	0	< 4.96	< 0.0E+00	0%
1,2,3,7,8-PeCDD	0.5	< 3.69	< 5.4E-04	14%
TOTAL PeCDD	0	< 3.69	< 0.0E+00	0%
1,2,3,4,7,8-HxCDD	0.1	< 7.14	< 2.1E-04	5%
1,2,3,6,7,8-HxCDD	0.1	< 6.60	< 1.9E-04	5%
1,2,3,7,8,9-HxCDD	0.1	< 6.74	< 2.0E-04	5%
TOTAL HxCDD	0	< 6.82	< 0.0E+00	0%
1,2,3,4,6,7,8-HpCDD	0.01	< 5.29	< 1.6E-05	0.4%
TOTAL HpCDD	0	< 5.29	< 0.0E+00	0%
OCDD	0.001	< 14.1	< 4.1E-06	0.1%
2,3,7,8-TCDF	0.1	< 3.85	< 1.1E-04	3%
TOTAL TCDF	0	< 3.85	< 0.0E+00	0%
1,2,3,7,8-PeCDF	0.05	< 5.73	< 8.4E-05	2%
2,3,4,7,8-PeCDF	0.5	< 5.28	< 7.8E-04	19%
TOTAL PeCDF	0	< 5.49	< 0.0E+00	0%
1,2,3,4,7,8-HxCDF	0.1	< 5.10	< 1.5E-04	4%
1,2,3,6,7,8-HxCDF	0.1	< 2.19	< 6.4E-05	2%
2,3,4,6,7,8-HxCDF	0.1	< 2.33	< 6.9E-05	2%
1,2,3,7,8,9-HxCDF	0.1	< 2.55	< 7.5E-05	2%
TOTAL HxCDF	0	< 5.26	< 0.0E+00	0%
1,2,3,4,6,7,8-HpCDF	0.01	< 3.88	< 1.1E-05	0.3%
1,2,3,4,7,8,9-HpCDF	0.01	< 4.62	< 1.4E-05	0.3%
TOTAL HpCDF	0	< 4.22	< 0.0E+00	0%
OCDF	0.001	< 10.5	< 3.1E-06	0.1%
TOTAL TEQs (ng/m^3)		=	< 0.0040	100%
TOTAL TEQs (ng/m^3 @ 7 % O_2)		=	< 0.0035	
TOTAL TEQs (g/s)		=	< 3.8E-12	

[a] EDL = Sample specific estimated detection limit

[b] U.S.EPA (1989) Toxic Equivalency Factor

Table A-2
Facility: Bostik, Inc. - Middleton, MA
Emission Rate Predictions for PAH's at their QLs [a]

	HRMS CARB 429 PAH MDLs via HRMS Run Date: 990525 VG70			**Sample Volume** 3.4 m^3 **Stack Flowrate** 2,000 dscfm					
Component	**MDL/split** ng	**QL** Low Level Standard pg/uL	FV = 1 mL Split = **3** **QL/train** Quant. Limit ng		g/sec		µg/m³		lb/hr
Naphthalene	500*	10	2000	<	5.55E-07	<	0.588	<	4.41E-06
2-Methylnaphthalene	50*	10	200	<	5.55E-08	<	0.059	<	4.41E-07
Acenaphthylene	0.6	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Acenaphthene	1.1	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Fluorene	1.8	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Phenanthrene	1.9	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Anthracene	1.2	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Fluoranthene	1.5	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Pyrene	1.4	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Benz(a)anthracene	1.0	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Chrysene	1.1	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Benzo(b)fluoranthene	1.2	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Benzo(k)fluoranthene	0.8	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Benzo(e)pyrene	0.5	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Benzo(a)pyrene	0.6	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Perylene	0.7	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Indeno(1,2,3-cd)pyrene	0.6	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Dibenzo(ah)anthracene	0.7	10	30	<	8.33E-09	<	0.009	<	6.61E-08
Benzo(ghi)perylene	0.7	10	30	<	8.33E-09	<	0.009	<	6.61E-08

[a] QL - quantitation limit based on the low level standard

* Practical limit of detection based on typical XAD2 resin background levels.

Table A-3
Facility: Bostik, Inc. - Middleton, MA
Example Semivolatile Organic PICs and Anticipated Method Detection Limits

SVOC via 0010/8270C MDLs
Run Date: 990223
HP-5972

Sample Volume 3.4 m^3
Stack Flowrate 2,000 dscfm

				FV = 1mL			
				Split =	3		
Component	**RDL** µg	**MDL/split** µg	Low Level Standard ng/µL	**QL/train** Quatitation Limit µg	g/s	µg/m^3	lb/hour
N-Nitrosodimethylamine	10	0.29	5	15	4.2E-06	4.41	3.30E-05
Phenol	1.1	0.67	10	30	8.3E-06	8.82	6.61E-05
Bis(2-chloroethyl)ether	1.8	0.21	5	15	4.2E-06	4.41	3.30E-05
2-Chlorophenol	2.7	0.84	10	30	8.3E-06	8.82	6.61E-05
1,3-Dichlorobenzene	2	0.18	5	15	4.2E-06	4.41	3.30E-05
1-4-Dichlorobenzene	2	0.17	5	15	4.2E-06	4.41	3.30E-05
1,2-Dichlorobenzene	2	0.16	5	15	4.2E-06	4.41	3.30E-05
Bis(2-chloroisopropyl)ether	1.5	0.2	5	15	4.2E-06	4.41	3.30E-05
Hexachloroethane	2	0.17	5	15	4.2E-06	4.41	3.30E-05
N-Nitroso-di-N-Propylamine	2.1	0.2	5	15	4.2E-06	4.41	3.30E-05
Nitrobenzene	2	0.47	5	15	4.2E-06	4.41	3.30E-05
Isophorone	4	0.14	5	15	4.2E-06	4.41	3.30E-05
2-Nitrophenol	1.4	0.57	10	30	8.3E-06	8.82	6.61E-05
2,4-Dimethylphenol	1.8	2	10	30	8.3E-06	8.82	6.61E-05
Bis(2-chloroethoxy)methane	1.3	0.17	5	15	4.2E-06	4.41	3.30E-05
2,4-Dichlorophenol	1.2	0.73	10	30	8.3E-06	8.82	6.61E-05
1,2,4-Trichlorobenzene	2	0.15	5	15	4.2E-06	4.41	3.30E-05
Naphthalene	0.44	0.21	5	15	4.2E-06	4.41	3.30E-05
Hexachlorobutadiene	2	0.19	5	15	4.2E-06	4.41	3.30E-05
4-Chloro-3-Methylphenol	1.4	0.9	10	30	8.3E-06	8.82	6.61E-05
Hexachlorocyclopentadiene	2	0.12	5	15	4.2E-06	4.41	3.30E-05
2,4,6-Trichlorophenol	1.2	0.9	10	30	8.3E-06	8.82	6.61E-05
2-Chloronaphthalene	0.9	0.3	5	15	4.2E-06	4.41	3.30E-05
Acenaphthylene	0.4	0.16	5	15	4.2E-06	4.41	3.30E-05
Dimethyl phthalate	1.1	0.13	5	15	4.2E-06	4.41	3.30E-05
2,6-Dinitrotoluene	0.6	0.16	5	15	4.2E-06	4.41	3.30E-05
Acenaphthene	0.7	0.16	5	15	4.2E-06	4.41	3.30E-05
2,4-Dinitrophenol	4.8	NA	20	60	1.7E-05	17.64	1.32E-04
2,4-Dinitrotoluene	0.5	0.13	5	15	4.2E-06	4.41	3.30E-05
4-Nitrophenol	1.4	0.3	20	60	1.7E-05	17.64	1.32E-04
Fluorene	0.3	0.13	5	15	4.2E-06	4.41	3.30E-05
4-Chlorophenylphenylether	0.9	0.17	5	15	4.2E-06	4.41	3.30E-05
Diethyl phthalate	1.1	0.17	5	15	4.2E-06	4.41	3.30E-05
4,6-Dinitro-2-methylphenol	1.5	0.95	10	30	8.3E-06	8.82	6.61E-05
N-Nitrosodiphenylamine	1.9	0.16	5	15	4.2E-06	4.41	3.30E-05
4-Bromophenylphenylether	0.3	0.14	5	15	4.2E-06	4.41	3.30E-05

Table A-3
Facility: Bostik, Inc. - Middleton, MA
Example Semivolatile Organic PICs and Anticipated Method Detection Limits

SVOC via 0010/8270C MDLs
Run Date: 990223
HP-5972

Sample Volume 3.4 m^3
Stack Flowrate 2,000 dscfm

				FV = 1mL			
				Split =	**3**		
Component	**RDL**	**MDL/split**	Low Level Standard	**QL/train** Quatitation Limit			
	µg	µg	ng/µL	µg	g/s	µg/m³	lb/hour
Hexachlorobenzene	2	0.19	5	15	4.2E-06	4.41	3.30E-05
Pentachlorophenol	1.3	1.3	10	30	8.3E-06	8.82	6.61E-05
Phenanthrene	0.3	0.16	5	15	4.2E-06	4.41	3.30E-05
Anthracene	0.2	0.13	5	15	4.2E-06	4.41	3.30E-05
Di-n-butyl phthalate	1.1	0.34	5	15	4.2E-06	4.41	3.30E-05
Fluoranthene	0.2	0.09	5	15	4.2E-06	4.41	3.30E-05
Pyrene	0.3	0.18	5	15	4.2E-06	4.41	3.30E-05
Benzyl butyl phthalate	0.6	0.38	5	15	4.2E-06	4.41	3.30E-05
3,3-Dichlorobenzidine	1	0.8	5	15	4.2E-06	4.41	3.30E-05
Benzo(a)anthracene	0.2	0.13	5	15	4.2E-06	4.41	3.30E-05
Chrysene	0.3	0.08	5	15	4.2E-06	4.41	3.30E-05
Bis(2-ethylhexyl)phthalate	1.4	0.98	5	15	4.2E-06	4.41	3.30E-05
Di-n-octyl phthalate	1.1	0.12	5	15	4.2E-06	4.41	3.30E-05
Benzo(b)fluoranthene	0.4	0.15	5	15	4.2E-06	4.41	3.30E-05
Benzo(k)fluoranthene	0.4	0.25	5	15	4.2E-06	4.41	3.30E-05
Benzo(a)pyrene	0.5	0.1	5	15	4.2E-06	4.41	3.30E-05
Indeno(1,2,3-cd)pyrene	0.6	0.37	5	15	4.2E-06	4.41	3.30E-05
Dibenzo(a,h)anthracene	0.4	0.18	5	15	4.2E-06	4.41	3.30E-05
Benzo(ghi)perylene	0.4	0.13	5	15	4.2E-06	4.41	3.30E-05
Aniline	5	0.61	5	15	4.2E-06	4.41	3.30E-05
Carbazole	5	0.23	5	15	4.2E-06	4.41	3.30E-05
Benzyl alcohol	2	1.1	5	15	4.2E-06	4.41	3.30E-05
2-Methylphenol	1.7	1.3	5	15	4.2E-06	4.41	3.30E-05
3&4-Methylphenol	2.1	2.1	10	30	8.3E-06	8.82	6.61E-05
1-Chloronaphthalene	0.9	0.3	5	15	4.2E-06	4.41	3.30E-05
Benzoic acid	1	1.5	5	15	4.2E-06	4.41	3.30E-05
4-Chloroaniline	5	1.1	5	15	4.2E-06	4.41	3.30E-05
2-Methylnaphthalene	1	0.5	5	15	4.2E-06	4.41	3.30E-05
2,4,5-Trichlorophenol	0.6	1.4	5	15	4.2E-06	4.41	3.30E-05
2-Nitroaniline	5	0.5	5	15	4.2E-06	4.41	3.30E-05
3-Nitroaniline	5	0.7	5	15	4.2E-06	4.41	3.30E-05
Dibenzofuran	5	0.49	5	15	4.2E-06	4.41	3.30E-05
Benzidine	10	NA	5	15	4.2E-06	4.41	3.30E-05
4-Nitroaniline	5	0.7	5	15	4.2E-06	4.41	3.30E-05
1,2-Diphenylhydrazine	5	NA	5	15	4.2E-06	4.41	3.30E-05

Note: QLs for chlorobenzenes may be improved to about 80 ng if analyzed as separate fraction using CARB M428.

Table A-4

Facility: Bostik, Inc. - Middleton, MA

Example Volatile Organic PICs and Anticipated Reporting Limits

VOST via M 5041A / 8260B			▨
Sample Volume	20.0	dsL	
Sample Volume	0.02	m^3	
Stack Flowrate	2,000	dscfm	

Component	RL µg *	g/s	µg/m^3	lb/hr
Acetone	0.120	5.7E-06	6.00	4.49E-05
Benzene	0.009	4.1E-07	0.43	3.22E-06
Bromobenzene	0.011	5.2E-07	0.55	4.12E-06
Bromochloromethane	0.011	5.2E-07	0.55	4.12E-06
Bromodichloromethane	0.009	4.2E-07	0.44	3.30E-06
Bromoform	0.013	6.1E-07	0.65	4.87E-06
Bromomethane	0.016	7.4E-07	0.78	5.84E-06
2-Butanone	0.064	3.0E-06	3.20	2.40E-05
n-Butylbenzene	0.015	7.3E-07	0.77	5.77E-06
sec-Butylbenzene	0.019	9.1E-07	0.96	7.19E-06
tert-Butylbenzene	0.018	8.4E-07	0.89	6.67E-06
Carbon disulfide	0.008	4.0E-07	0.42	3.15E-06
Carbon tetrachloride	0.007	3.2E-07	0.34	2.55E-06
Chlorobenzene	0.009	4.1E-07	0.43	3.22E-06
Chlorodibromomethane	0.008	3.6E-07	0.38	2.85E-06
Chloroethane	0.008	3.7E-07	0.39	2.92E-06
Chloroform	0.009	4.3E-07	0.46	3.45E-06
Chloromethane	0.012	5.7E-07	0.60	4.49E-06
2-Chlorotoluene	0.011	5.1E-07	0.54	4.05E-06
4-Chlorotoluene	0.011	5.4E-07	0.57	4.27E-06
1,2-Dibromo-3-chloropropane	0.032	1.5E-06	1.60	1.20E-05
1,2-Dibromoethane	0.008	3.7E-07	0.39	2.92E-06
Dibromomethane	0.013	6.0E-07	0.64	4.79E-06
1,2-Dichlorobenzene	0.012	5.6E-07	0.59	4.42E-06
1,3-Dichlorobenzene	0.013	6.0E-07	0.64	4.79E-06
1,4-Dichlorobenzene	0.015	7.0E-07	0.74	5.54E-06
Dichlorodifluoromethane	0.010	4.9E-07	0.52	3.90E-06
1,1-Dichloroethane	0.007	3.2E-07	0.34	2.55E-06
1,2-Dichloroethane	0.010	4.5E-07	0.48	3.60E-06
cis-1,2-Dichloroethene	0.010	4.7E-07	0.50	3.75E-06
trans-1,2-Dichloroethene	0.008	3.6E-07	0.38	2.85E-06
1,1-Dichloroethene	0.011	5.4E-07	0.57	4.27E-06

*Reporting limits are presented on a "sample train" basis. These detection limits are based on multiplying the per analysis detection limits by a factor of two which assumes that the tenax tube and the tenax/charcoal tube are desorbed separately.

Table A-4 (continued)
Facility: Bostik, Inc. - Middleton, MA
<u>Example</u> Volatile Organic PICs and Anticipated Reporting Limits

VOST via 5041A/8260B				
Sample Volume	20.0	dsL		
Sample Volume	0.02	m^3		
Stack Flowrate	2,000	dscfm		

Component	**RL µg ***	**g/s**	**$\mu g/m^3$**	**lb/hr**
1,2-Dichloropropane	0.009	4.2E-07	0.45	3.37E-06
1,3-Dichloropropane	0.014	6.7E-07	0.71	5.32E-06
2,2-Dichloropropane	0.007	3.2E-07	0.34	2.55E-06
cis-1,3-Dichloropropene	0.008	3.7E-07	0.39	2.92E-06
trans-1,3-Dichloropropene	0.008	3.7E-07	0.39	2.92E-06
1,1-Dichloropropene	0.008	4.0E-07	0.42	3.15E-06
Ethylbenzene	0.006	3.0E-07	0.32	2.40E-06
Hexachlorobutadiene	0.008	3.9E-07	0.41	3.07E-06
2-Hexanone	0.036	1.7E-06	1.80	1.35E-05
Isopropylbenzene	0.012	5.5E-07	0.58	4.34E-06
p-Isopropyltoluene	0.017	8.0E-07	0.85	6.37E-06
Methylene chloride	0.024	1.1E-06	1.20	8.99E-06
4-Methyl-2-pentanone	0.058	2.7E-06	2.90	2.17E-05
Naphthalene	0.013	6.2E-07	0.66	4.94E-06
n-Propylbenzene	0.011	5.1E-07	0.54	4.05E-06
Styrene	0.010	4.6E-07	0.49	3.67E-06
1,1,1,2-Tetrachloroethane	0.009	4.3E-07	0.46	3.45E-06
1,1,2,2-Tetrachloroethane	0.013	6.3E-07	0.67	5.02E-06
Tetrachloroethene	0.009	4.2E-07	0.44	3.30E-06
Toluene	0.005	2.5E-07	0.27	2.02E-06
1,2,3-Trichlorobenzene	0.011	5.3E-07	0.56	4.20E-06
1,2,4-Trichlorobenzene	0.013	6.2E-07	0.66	4.94E-06
1,1,1-Trichloroethane	0.010	4.8E-07	0.51	3.82E-06
1,1,2-Trichloroethane	0.011	5.3E-07	0.56	4.20E-06
Trichloroethene	0.020	9.3E-07	0.99	7.42E-06
Trichlorofluoromethane	0.016	7.6E-07	0.80	5.99E-06
1,2,3-Trichloropropane	0.020	9.3E-07	0.99	7.42E-06
1,2,4-Trimethylbenzene	0.013	5.9E-07	0.63	4.72E-06
1,3,5-Trimethylbenzene	0.015	7.0E-07	0.74	5.54E-06
Vinyl chloride	0.006	3.0E-07	0.32	2.40E-06
m-Xylene & p-Xylene	0.014	6.8E-07	0.72	5.39E-06
o-Xylene	0.011	5.0E-07	0.53	3.97E-06

*Reporting limits are presented on a "sample train" basis. These detection limits are based on multiplying the per analysis detection limits by a factor of two which assumes that the tenax tube and the tenax/charcoal tube are desorbed separately.

Table A-5
Facility: Bostik, Inc. - Middleton, MA
Emission Rate Predictions for Metals at their RLs [a], [b]

	Units	Stack Gas Assumptions:
Sample Volume	dscf	85.0
Sample Volume	m³	2.41
O_2 Conc.	% v/v (dry)	5.00
CO_2 Conc.	% v/v (dry)	11.50
Stack Flowrate	dscfm	2,000

Target Metals:	MACT Categ.	RL µg	g/sec	µg/m³ at 7% O_2	lb/hr
Antimony (Sb)		< 0.68	< 2.7E-07	< 0.25	< 2.12E-06
Arsenic (As)		< 0.68	< 2.7E-07	< 0.25	< 2.12E-06
Barium (Ba)		< 0.34	< 1.3E-07	< 0.12	< 1.06E-06
Beryllium (Be)		< 0.34	< 1.3E-07	< 0.12	< 1.06E-06
Cadmium (Cd)	SVM	< 0.34	< 1.3E-07	< 0.12	< 1.06E-06
Total Chromium (Cr)	LVM	< 0.68	< 2.7E-07	< 0.25	< 2.12E-06
Lead (Pb)	SVM	< 0.34	< 1.3E-07	< 0.12	< 1.06E-06
Mercury (Hg)		< 1.41	< 5.5E-07	< 0.51	< 4.39E-06
Nickel (Ni)		< 0.68	< 2.7E-07	< 0.25	< 2.12E-06
Silver (Ag)		< 0.34	< 1.3E-07	< 0.12	< 1.06E-06
Thallium (Tl)		< 0.34	< 1.3E-07	< 0.12	< 1.06E-06

LVM = Low Volatile Metals = Chromium only **Subtotal =** < **0.25**

SVM = Semivolatile Metals = Cadmium & Lead **Subtotal =** < **0.25**

[a] RL = Reporting Limit

[b] Applicable Sampling and Analytical Methods:

Parameter	Sampling Method	Analytical Method
Hg	EPA Method 29/0060	EPA Method 7470A (CVAAS)
All Other Metals	EPA Method 29/0060	EPA Method 6020 (ICP-MS)

Table A-6
Facility: Bostik, Inc. - Middleton, MA
Emission Rate Predictions for PM, HCl and Cl_2 at their RLs [a], [b]

	Units	**Stack Gas Assumptions:**			
Sample Volume	dscf	85.000			
Sample Volume	m^3	2.41			
O_2 Conc.	% v/v (dry)	5.00			
CO_2 Conc.	% v/v (dry)	11.50			
Stack Flowrate	dscfm	2,000			
HCl and Cl_2:	M.W.	RL µg	g/sec	ppm at 7% O_2	lb/hr
HCl	36.5	< 100	< 3.9E-05	< 0.024	< 3.11E-04
Cl_2	71.0	< 100	< 3.9E-05	< 0.012	< 3.11E-04
Particulate Matter:		RL mg	gr/dscf at 7% O_2	mg/m^3 at 7% O_2	lb/hr
Particulate Matter (PM)		< 0.30	< 4.8E-05	< 0.109	< 9.34E-04

[a] RL = Reporting Limit

[b] Applicable Sampling and Analytical Methods:

Parameter	Sampling Method	Analytical Method
HCl	EPA Method 26A	EPA Method 26A (IC)
Cl_2	EPA Method 26A	EPA Method 26A (IC)
PM	EPA Method 26A	EPA Method 5 (Gravimetric)

ATTACHMENT B

Example Field Data Sheets

ENSR | AECOM

Field Log - Bostik, Inc. - Middleton, MA

Date	Time	Description of Events and Activities

page ____ of ____

ENSR | AECOM

METHOD 2 GAS VELOCITY AND VOLUME DATA SHEET

Facility :
Date :
Operator(s) :

Port Length:
Port Diam.:

Monorail ?
Y or N

Platform Width
Railing Ht. :

Stack Diameter (in.) :
Bar. Press. (in. Hg) :
Static Press. (in. H_2O)

Cp : 0.84 or 0.99 (Circle one)

O_2 (%)
CO_2 (%)
Wet Bulb Temp. (°F) :
Dry Bulb Temp. (°F) :

SCHEMATIC OF STACK CROSS SECTION

Pt. #	Pos. (in.)	Vel. DP (in. H_2O)	Stack Temp. (°F)	Flow Angle that Yields a Null DP		Pt. #	Pos. (in.)	Vel. DP (in. H_2O)	Stack Temp. (°F)	Flow Angle that Yields a Null DP

R:\Air_Tox\COMMON\Source Test Templates\DataSheets\[Method2Form.xls]A

ENSR | AECOM

NOZZLE CALIBRATION FORM

Client: ______________________ Project #: ______________________

Date: ____________ Calibrated by: ______________________

Nozzle ID #	D_1, in.	D_2, in.	D_3, in.	Delta D, in.	D_{avg}, in.

Where:

$D_{1,2,3}$ = Nozzle diameter measured on a different diameter to the nearest 0.001 in.

Delta D = Maximum difference between any two measurements, in.
Tolerance = 0.004 in.

D_{avg} = Average of $D_{1,2,3}$

R:\Air_Tox\COMMON\Source Test Templates\DataSheets\[NozzleCalibForm.xls]A

ENSR | AECOM

EPA ISOKINETIC SAMPLING - FIELD DATA SHEET

Sampling Train ____________________
Run Number ____________________
Client ____________________
Facility Location ____________________
Source ____________________
Date ____________________
Operator ____________________
Stack Dia. - in. ____________________
Start Time ____________________
Stop Time ____________________

Barometric Pressure ______________
Static Pressure (+/-) ______________
Probe/Pitot Number ______________
Pitot Coefficient ______________
Filter Box No. ______________
Meter Box No. ______________
Orifice Coefficient (Y) ______________
Delta H @ ______________
Nozzle Size/No. ______________
XAD Thermocouple ID: ______________
Imp Outlet TC ID:

LEAK CHECKS in "Hg
INITIAL VAC. ________ **in.** **CFM** ________
MID VAC. ________ **in.** **CFM** ________
FINAL VAC. ________ **in.** **CFM** ________
\+ - \+ -
INIT. PITOT ________ **FINAL PITOT** ________

FILTER DATA

NUMBER	TARE

IMPINGER VOL'S.		
INIT.	FINAL	RINSE
SILICA GEL		

Orsat	
CO2	O2

Final Purge Rate ______________
Final PH

SAMPLE POINT	CLOCK TIME	VELOCITY HEAD DeltaP, in wc	ORIFICE METER VOL. DeltaH, in wc	GAS METER VOLUME ft³	TEMPERATURE READINGS, °F							PUMP VACUUM in. Hg	COMMENTS
					STACK	PROBE	OVEN	ORGANIC MODULE	IMPINGER	GAS METER IN	GAS METER OUT		

ENSR | AECOM

K-FACTOR FORM -- ISOKINETIC SAMPLING TRAINS

Meter Box #: ________ Delta H@: ________ D_{nozz} : ________

K-Factors :

Stack Temp, °F	**Meter BoxTemp, °F**					

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ENSR | AECOM

SOLID/LIQUID GRAB SAMPLING FIELD DATA SHEET

ENSR Project No.

Client: **Facility:**

Stream Sampled:

Sampling Location:

Date:		**Date:**		**Date:**	
Condition:		**Condition:**		**Condition:**	
Run No.		**Run No.**		**Run No.**	
Start Time:		**Start Time:**		**Start Time:**	
Stop Time:		**Stop Time:**		**Stop Time:**	
Grab Interval	Clock Time (actual)	Grab Interval	Clock Time (actual)	Grab Interval	Clock Time (actual)
Beginning		Beginning		Beginning	
Middle		Middle		Middle	
End		End		End	

Comments :

Signature of Sampler:

VOST DATA SHEET

PROJECT NO.

DATE

CLIENT

OPERATOR

FACILITY

BAR. PRESSURE, in. Hg

SOURCE

PROBE LENGTH (ft)

SAMPLING LOCATION

DESIRED PROBE TEMP.

METER CALIBRATION FACTOR (Y)

PROBE PURGED ?

DRY GAS METER NO.

DESIRED FLOW RATE (Lpm)

RUN NO.

DESIRED SAMPLE VOLUME (dsL)

TX & TX/C TUBE NO'S.

DGM PRESSURE, in. H_2O

Train Leak Check -- INITIAL VACUUM (in. Hg):

Leak Rate : in. Hg in 60 sec.

Train Leak Check -- FINAL VACUUM (in. Hg):

Leak Rate : in. Hg in 60 sec.

ACCEPTANCE CRITERIA : Leak Rate < 2.5 mm Hg (0.1 in. Hg) after 60 sec.

SAMPLING TIME (min)	CLOCK TIME (24-hr)	FLOW RATE (Lpm)	GAS METER READING (L)	TEMPERATURE READINGS			PUMP VAC. (in. Hg)
				PROBE (°C or °F)	DRY GAS METER (°C or °F)	TRAP (°C or °F)	

COMMENTS :

Laboratory Lot #:

VOST AUDIT DATA SHEET

PROJECT NO.	DATE
CLIENT	OPERATOR
FACILITY	BAR. PRESSURE, in. Hg
SOURCE	PROBE LENGTH (ft)
SAMPLING LOCATION EPA Audit Cylinder	DESIRED PROBE TEMP.
METER CALIBRATION FACTOR (Y)	PROBE PURGED ?
DRY GAS METER NO.	DESIRED FLOW RATE (Lpm)
RUN NO.	DESIRED SAMPLE VOLUME (dsL)
TX & TX/C TUBE NO'S.	DGM PRESSURE, in. H_2O

Train Leak Check -- INITIAL VACUUM (in. Hg):	Leak Rate : in. Hg in 60 sec.
Train Leak Check -- FINAL VACUUM (in. Hg):	Leak Rate : in. Hg in 60 sec.

ACCEPTANCE CRITERIA : Leak Rate < 2.5 mm Hg (0.1 in. Hg) after 60 sec.

SAMPLING TIME (min)	CLOCK TIME (24-hr)	FLOW RATE (Lpm)	GAS METER READING (L)	TEMPERATURE READINGS			PUMP VAC. (in. Hg)
				PROBE (°C or °F)	DRY GAS METER (°C or °F)	TRAP (°C or °F)	

COMMENTS :

AUDIT CYLINDER NO.:

Initial Pressure: Final Pressure:

ATTACHMENT C

Isokinetic Sampling Train Setup and Recovery Schematics

ENSR | AECOM

SAMPLE TRAIN MOISTURE RECOVERY DATA SHEET

Reference Method / Sampling Train :

Recovered by :				**Recovered by :**				**Recovered by :**			
Run No.	**Date :**			**Run No.**	**Date :**			**Run No.**	**Date :**		
XAD Module No. :				**XAD Module No. :**				**XAD Module No. :**			
Filter No. :				**Filter No. :**				**Filter No. :**			
Impinger No. and Volume				**Impinger No. and Volume**				**Impinger No. and Volume**			
No.	Initial (mL)	Final (mL)	Rinse (mL)	No.	Initial (mL)	Final (mL)	Rinse (mL)	No.	Initial (mL)	Final (mL)	Rinse (mL)
1				1				1			
2				2				2			
3				3				3			
4				4				4			
5				5				5			
6				6				6			
7			**DIFF :**	7			**DIFF :**	7			**DIFF :**
Totals				**Totals**				**Totals**			
	Initial (g)	Final (g)	**DIFF :**		Initial (g)	Final (g)	**DIFF :**		Initial (g)	Final (g)	**DIFF :**
Silica Gel				Silica Gel				Silica Gel			
Final Net Moisture Gain:				**Final Net Moisture Gain:**				**Final Net Moisture Gain:**			

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ENSR | AECOM

SAMPLE TRAIN SETUP
PCDDs / PCDFs / PAHs
(as Per EPA Methods 0023A / 0010)

IMPINGERS --

1st -- empty
2nd -- 100 mL HPLC Water
3rd -- 100 mL HPLC Water
4th -- empty
5th -- Silica Gel

FIELD BLANKS -- (No Volumes Specified in Methods)

FH Rinse -- ~ 50 mL Acetone
~ 50 mL MeCl
~ 50 mL Toluene

Filter -- One unused filter

XAD Trap -- One unused XAD Trap

HPLC Water -- ~ 200 mL

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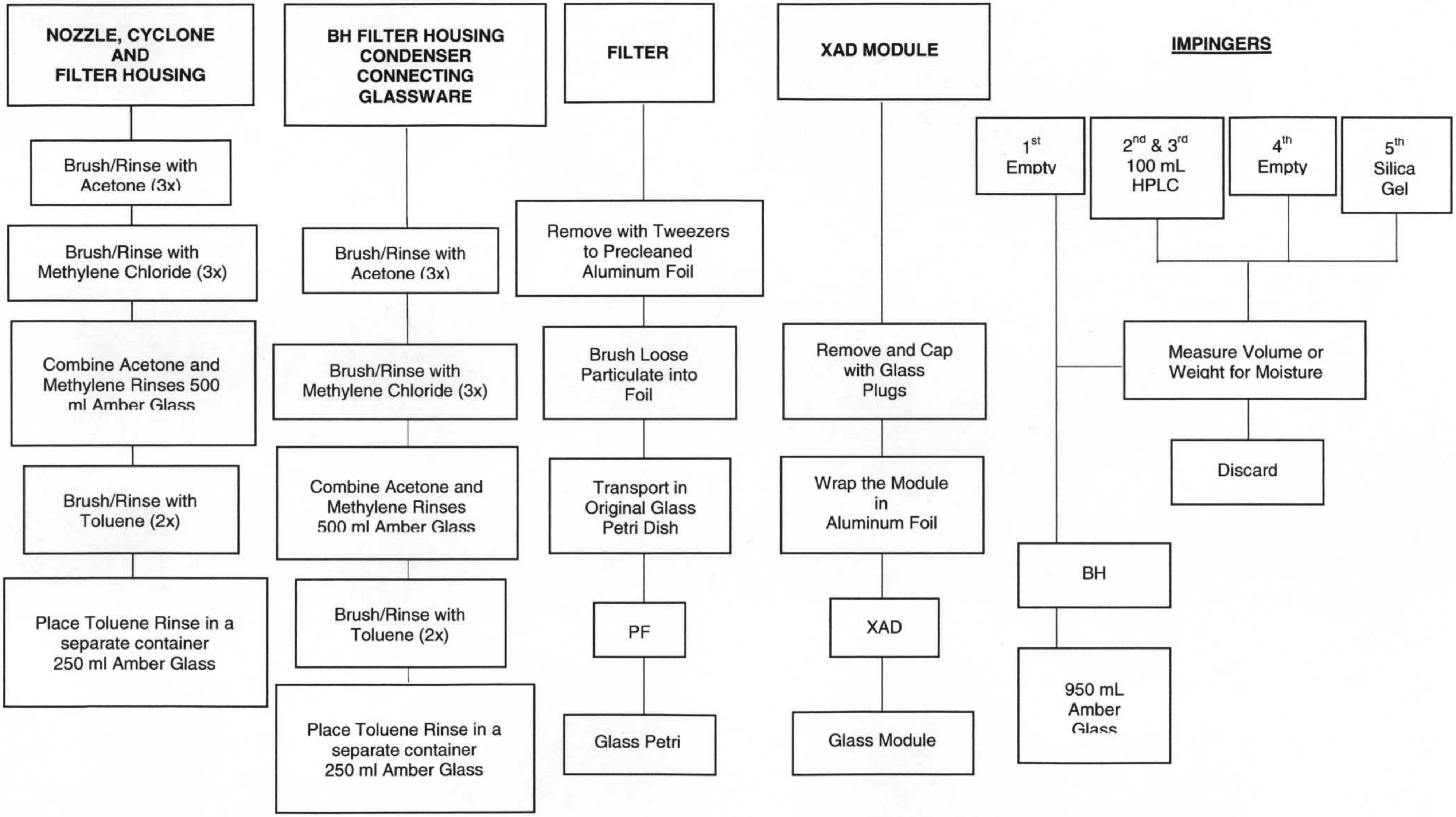


M0023A/0010 RECOVERY SCHEMATIC

ENSR | AECOM

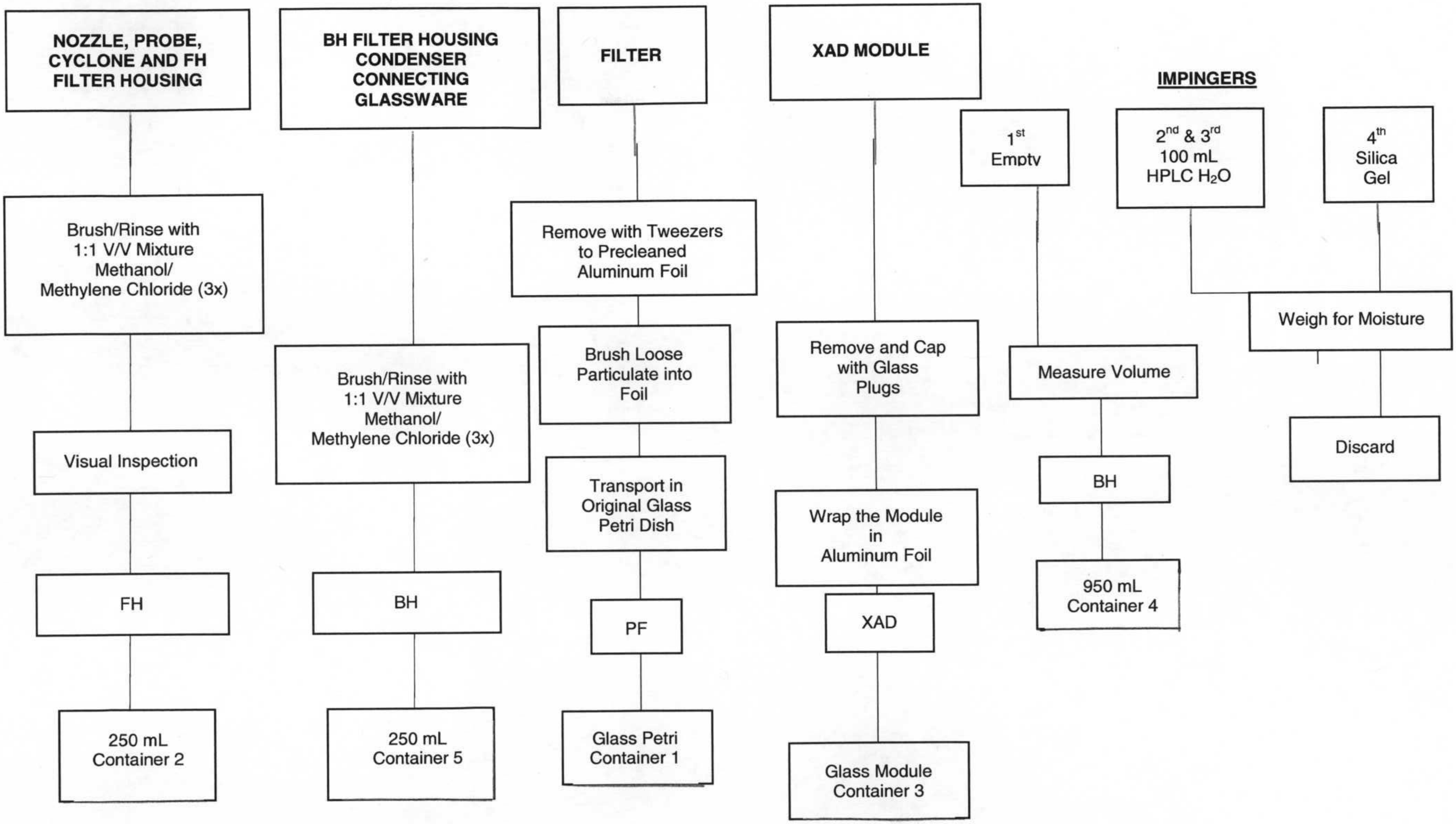
SAMPLE TRAIN SETUP
TARGET SEMIVOLATILE ORGANICS (SVOCs)
(as Per EPA Method 0010)

IMPINGERS --

1st	--	empty
2nd	--	100 mL HPLC Water
3rd	--	100 mL HPLC Water
4th	--	empty
5th	--	Silica Gel

FIELD BLANKS -- (No Volumes Specified by Method)

FH Rinse	--	~ 100 mL Methanol ~ 100 mL MeCl
Filter	--	One unused filter
XAD Trap	--	One unused XAD Trap
HPLC Water	--	~ 200 mL



METHOD 0010 (ORGANICS) RECOVERY SCHEMATIC

SAMPLE TRAIN SETUP
MULTIMETALS
(as Per EPA Method 29)

IMPINGERS --

1st	--	100 mL 5% HNO_3 / 10% H_2O_2
2nd	--	100 mL 5% HNO_3 / 10% H_2O_2
3rd	--	empty
4th	--	100 mL 10% H_2SO_4 / 4% $KMnO_4$
5th	--	100 mL 10% H_2SO_4 / 4% $KMnO_4$
6th		Silica Gel

FIELD BLANKS -- (Exact Volumes Specified by Method)

0.1 N HNO_3	--	300 mL
5% HNO_3 / 10% H_2O_2	--	200 mL
10% H_2SO_4 / 4% $KMnO_4$	--	100 mL
DI Water	--	100 mL
8 N HCl	--	25 mL (added to 200 mL water)
Filter	--	One unused filter

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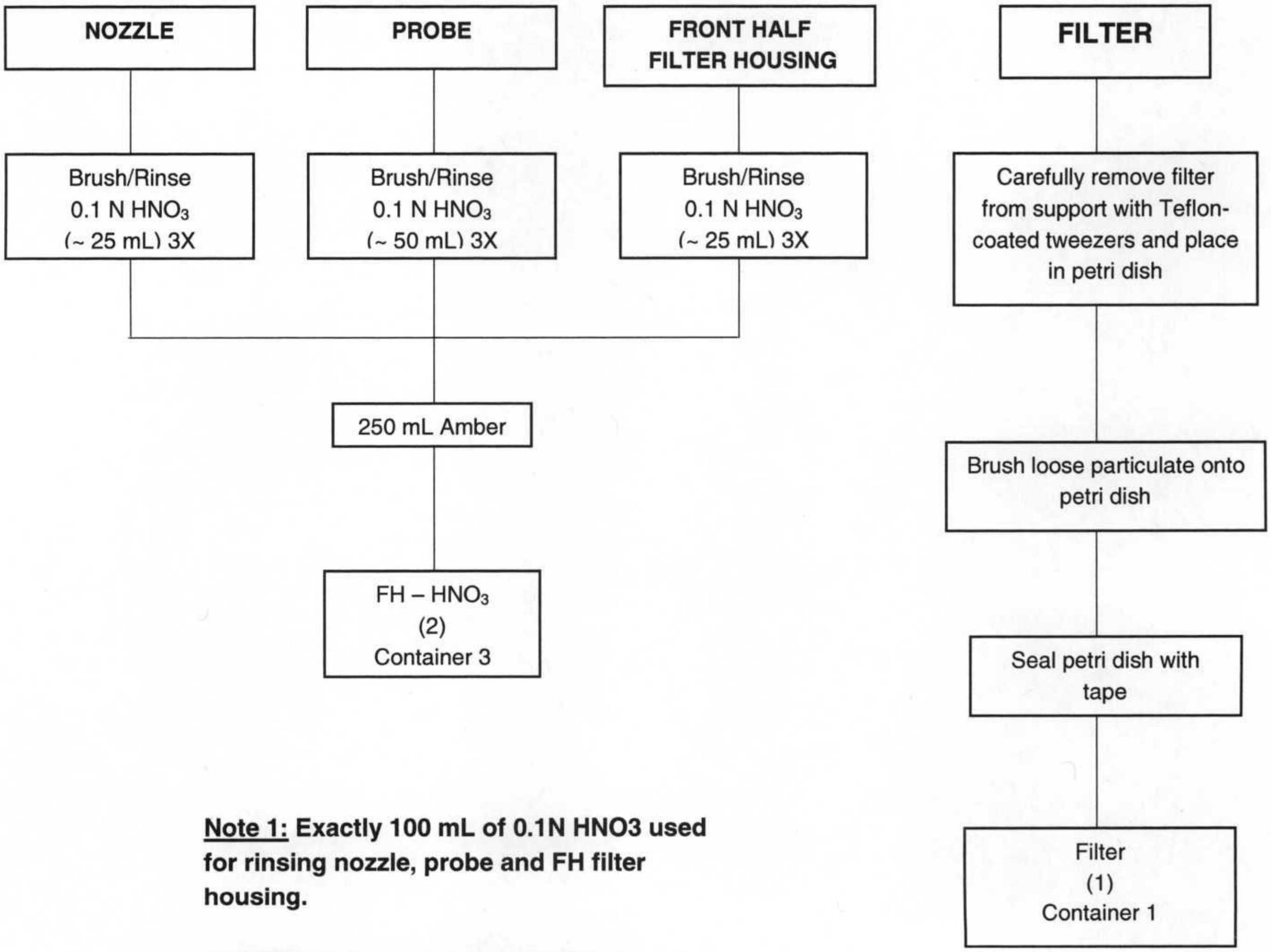

<u>Note 1:</u> Exactly 100 mL of 0.1N HNO3 used for rinsing nozzle, probe and FH filter housing.

<u>Note 2:</u> Nozzle, probe and FH filter housing are rinsed with water followed by acetone after the nitric acid rinse. The water and acetone are then discarded.

METHOD 29 (METALS) RECOVERY SCHEMATIC – FRONT HALF RECOVERY

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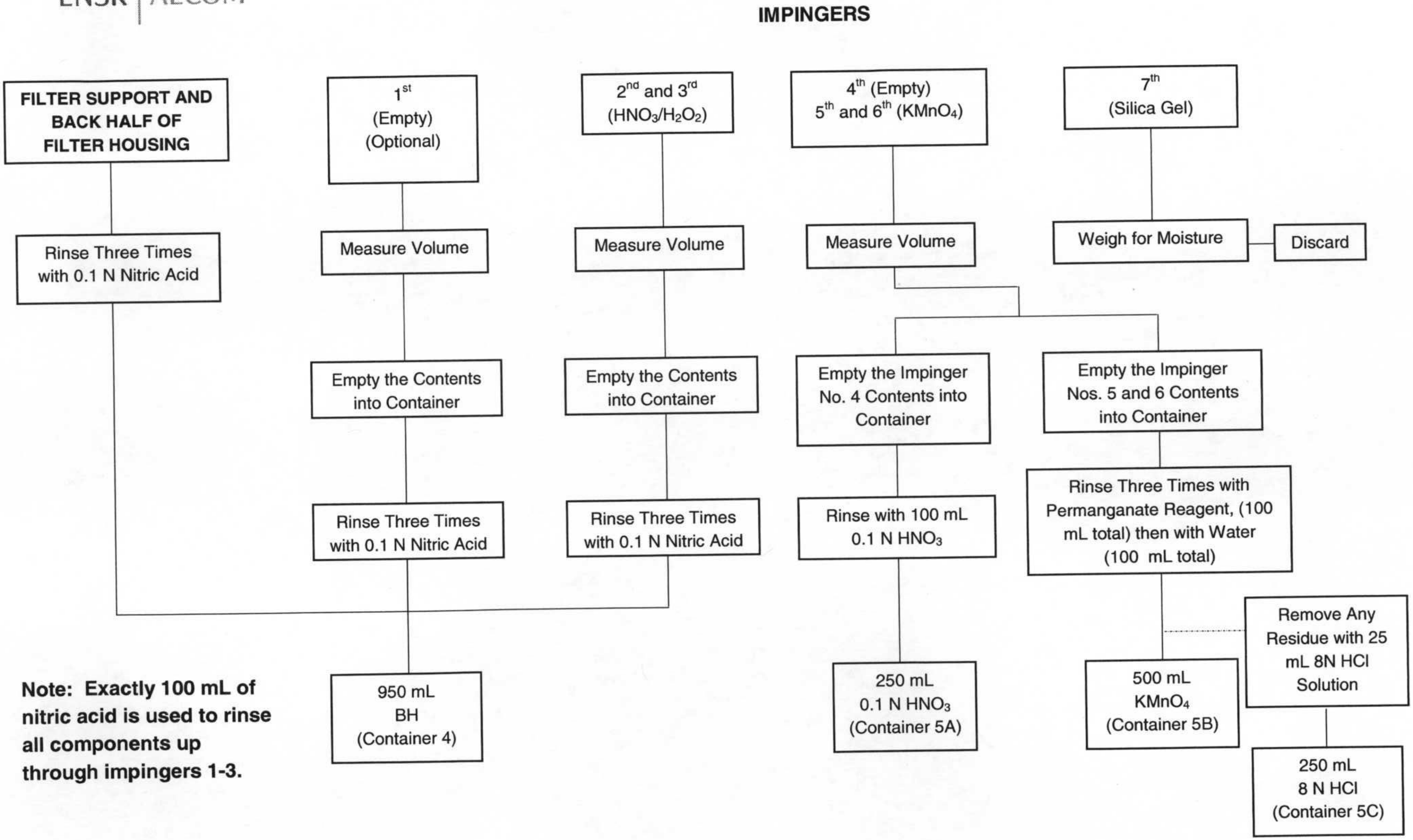

Note: Exactly 100 mL of nitric acid is used to rinse all components up through impingers 1-3.

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METHOD 29 (METALS) RECOVERY SCHEMATIC – BACK HALF RECOVERY

ENSR | AECOM

SAMPLE TRAIN SETUP
PARTICULATE MATTER / HCl / Cl_2
(as Per EPA Method 26A)

IMPINGERS --

1st	--	100 mL 0.1N H_2SO_4
2nd	--	100 mL 0.1N H_2SO_4
3rd	--	empty
4th	--	100 mL 0.1 N NaOH
5th	--	100 mL 0.1 N NaOH
6th		Silica Gel

FIELD BLANKS -- (Volumes Specified by Method)

0.1N H_2SO_4	--	200 mL
0.1 N NaOH	--	200 mL
DI Water	--	~ 200 mL
Acetone	--	~ 200 mL

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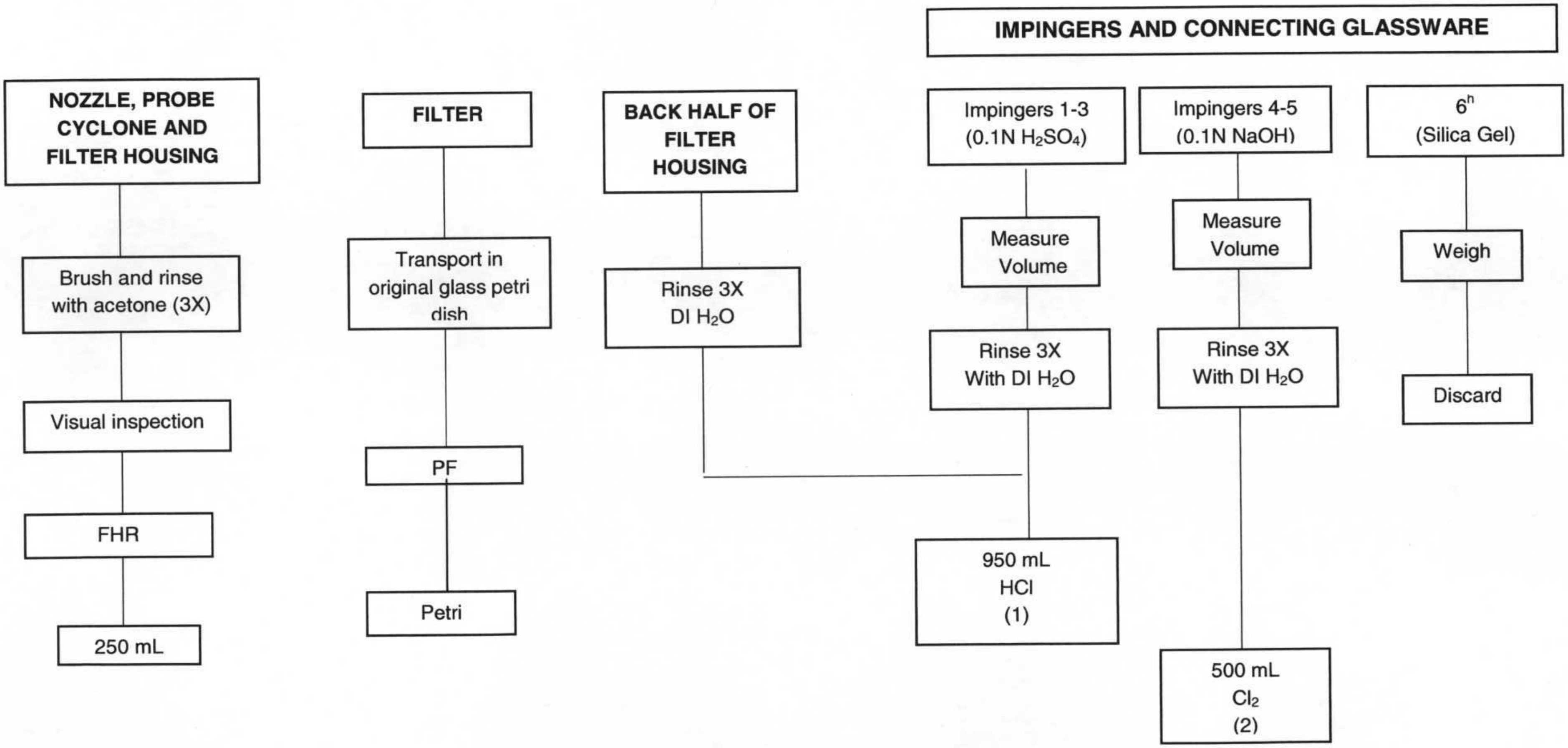
ENSR | AECOM



METHOD 26A (PM/HCl/Cl_2) RECOVERY SCHEMATIC

ATTACHMENT D

Detailed Analytical SOPs

Controlled Copy
Copy No. Uncontrolled Copy
Implementation Date: 10/20/06

STL KNOXVILLE

STANDARD OPERATING PROCEDURE

TITLE: Sample Preparation for Total Halogen Determination

(SUPERSEDES: KNOX-WC-0016, Rev. 2)

Prepared By: ______________________

Reviewed By: Jon M Humphrey 9-5-06
Technical Specialist

Approved By: 09/05/06
Quality Assurance Manager

Approved By: 9-5-06
Environmental, Health and Safety Coordinator

Approved By: 9-5-06
Laboratory Director

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1. Scope and Application

1.1. The purpose of this procedure is to describe the preparation steps followed to determine total chlorine, fluorine, bromine and iodine in waste feeds and related materials.

1.2. This procedure is applicable to the analysis of a wide variety of waste feed materials for total bromine, chlorine, fluorine and iodine. The determination will include bromine, chlorine, fluorine and iodine from both inorganic and organic materials present in the sample. It is based on ASTM Method E442, *Standard Test Method for Chlorine, Bromine and Iodine in Organic Compounds by Oxygen Flask Combustion* and SW-846 Method 5050, *Bomb Preparation Method for Solid Waste.*

1.2.1. For this procedure, the reporting limits are as follows:

- Total Bromine, 80 mg/kg.
- Total Chlorine, 200 mg/kg.
- Total Fluorine, 150 mg/kg
- Total Iodine, 500 mg/kg

2. Summary of Method

2.1. The sample is oxidized by combustion in an oxygen bomb containing oxygen at 30 atm or higher. The liberated halogen compounds are absorbed primarily as halides in a sodium carbonate/sodium bicarbonate buffer solution. The combustion products are collected by repeated rinsing of the combustion apparatus, and analyzed in accordance with SOP KNOX-WC-0005, current revision, based on SW-846 Method 9056.

3. Definitions

3.1. Total Bromine: The total bromine content of the sample, both from inorganic and organic sample constituents, expressed in terms of mg/kg.

3.2. Total Chlorine: The total chlorine content of the sample, both from inorganic and organic sample constituents, expressed in terms of mg/kg.

3.3. Total Fluorine: The total fluorine content of the sample, both from inorganic and organic sample constituents, expressed in terms of mg/kg.

3.4. Total Iodine: The total iodine content of the sample, both from inorganic and organic sample constituents, expressed in terms of mg/kg.

3.5. Total Halogens: For the purposes of this procedure, the total bromine, chlorine, fluorine and iodine content of the sample.

3.6. Additional definitions can be found in the STL Knoxville LQM glossary and in the STL Quality Management Plan.

4. Interferences

4.1. Samples with very high water content (> 25%) may not burn efficiently and require the addition of combustion aids to facilitate combustion.

5. Safety

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. Procedures shall be carried out in a manner that protects the health and safety of all associates. Exposure to chemicals and samples will be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. The preparation of all standards, reagents and glassware cleaning procedures that involve solvents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.3. All work must be stopped in the event of a known or potential compromise to the health and safety of any associate. The situation must be reported immediately to a laboratory supervisor.

5.4. Oxygen vigorously accelerates combustion. Keep all oil and greases away, do not use on regulators or gauges. Keep all combustibles away from oxygen and eliminate ignition sources. Use common safety practices for handling gas cylinders and ensure cylinder is secured at all times.

5.5. Primary materials used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Oxygen, Compressed	Oxidizer	NA	Exposure to Oxygen will not result in exposure symptoms unless the individual is exposed to high levels for more than 17 hours. However, exposure of flammable or combustible materials to Oxygen can create an extremely dangerous situation and result in an explosion,
2,2,2-Trichloroethanol	Corrosive	Not available	Causes burns. Harmful by inhalation, in contact with skin and if swallowed.
Methanol	Flammable, Poison, Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hydrazine Sulfate	Toxic	Not available	May cause allergic skin reaction. Causes burns. Toxic if absorbed through the skin. Toxic if inhaled. Material is extremely destructive to the tissue of the mucous membranes, upper respiratory tract, eyes and skin. Inhalation may result in spasm, inflammation and edema of the larynx, bronchi, chemical pneumonitis and pulmonary edema.
Hydrogen Peroxide, 30%	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
1-octanol	Flammable	TWA Not established.	May be harmful if swallowed or inhaled. Causes irritation to skin, eyes and respiratory tract. Affects the central nervous system. Combustible liquid and vapor.
2,3-dibromopropanol	Toxic	TWA Not established.	Harmful if swallowed. Toxic by inhalation and in contact with skin. Limited evidence of a carcinogenic effect. Risk of serious damage to eyes.
2,2,2-trifluoroethanol	Flammable Toxic	TWA Not established.	Harmful by inhalation, in contact with skin and if swallowed. Irritating to respiratory system and skin. Risk of serious damage to eyes.
2-iodoethanol	Toxic Combustible	TWA Not established.	Toxic if swallowed.
1-Exposure limit refers to the OSHA regulatory exposure limit.			

6. Equipment and Supplies

6.1. Parr Instrument Company Oxygen Bomb, Model 1108CL or equivalent.

6.2. Fuse Wire, Nickel-Chromium Alloy, 34 ga, Parr Instrument Company Cat. No. 45C10 or equivalent.

6.3. Parr Instrument Company Igniter, Cat. No. 2901EB or equivalent.

6.4. High density polyethylene (HDPE) sample containers, 125-mL capacity.

6.5. Analytical balance capable of weighing to 0.0001 g.

6.6. 3M Transparent Tape, Parr Instrument Company Cat. No. 517A or equivalent.

6.7. Sample capsule—an open crucible of stainless steel.

6.8. Top loading balance, capable of weighing to 0.01 grams.

7. Reagents and Standards

7.1. Deionized water.

7.2. Oxygen, free of combustible material and halogen compounds, available at a pressure of 40 atm.

7.3. Buffer solution, 0.048M Na_2CO_3/ 0.038M $NaHCO_3$. Dissolve 4.03 g Na_2CO_3 and 4.03 g $NaHCO_3$ in reagent water and dilute to l L. Store in HDPE bottle.

7.4. 1-Octanol, 99% purity.

7.5. 2,2,2-Trifluoroethanol, 99% purity.

7.6. 2,2,2,-Trichloroethanol, 99% purity.

7.7. 2,3-Dibromopropanol, 98% purity.

7.8. 2-Iodoethanol, 99% purity.

7.9. Hydrazine sulfate, ACS Certified, 2% w/v.

  7.9.1. Add 10 g hydrazine sulfate to 500 mL deionized water in a glass sample bottle. Shake to dissolve.

7.10. Eluate stock solution: Dissolve 6.30 g $NaHCO_3$ and 63.594 g Na_2CO_3 in 1L deionized water.

7.11. Methanol, 99% purity.

7.12. Acetone, Reagent grade.

7.13. LCS and MS/MSD Standard Solution: Prepare a solution of 2,3-dibromopropanol, 2-iodoethanol, 2,2,2-trichloroethanol and 2,2,2-trifluoroethanol to contain 20,000 mg/L chlorine and iodine and 7500 mg/L fluorine and bromine in methanol. Spiking solutions may be prepared that contain some or all of these components, as required. Stock solutions of each component should be prepared as a basis for the final spiking solution.

7.13.1. A solution containing chlorine and fluorine is sufficient for most testing. An additional solution containing all four halogens should be readily available at the concentrations listed. Please note that the concentrations given are for the elemental halogens, and the percentage of each halogen in the spiking compound must be taken into account.

8. Sample Collection, Preservation and Storage

8.1. Sampling is not performed for this method by STL Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, Receipt and Log In of Commercial Samples, current revision.

MATRIX	CONTAINER	PRESERVATION	HOLDING TIME
Aqueous Waste Feed Samples	Amber glass with Teflon™-lined closure (minimum: 1 x 250 mL)	None Required. Cool, 4°C if high volatile content is suspected.	None Specified. Analyze in a timely manner, preferably less than 30 days.
Organic Waste Feed Samples	Amber glass with Teflon™-lined closure (minimum: 1 x 250 mL)	None Required. Cool, 4°C if high volatile content is suspected.	None Specified. Analyze in a timely manner, preferably less than 30 days.
Soil, Sediment, Sludge or Other Solid Waste Feed Samples	Wide-mouth glass with Teflon™-lined closure (minimum 4 oz.)	None Required. Cool, 4°C if high volatile content is suspected.	None Specified. Analyze in a timely manner, preferably less than 30 days.

9. Quality Control

9.1. Method Blanks: Add 500 µL of methanol and 700 µL 1-octanol to a taped Parr sample capsule. Combust the sample in a Parr Bomb according to section 11.3. Analyze the collected combustate solution for chloride and fluoride. Calculate the concentration based on a 1.0-gram sample.

9.2. Laboratory Control Samples: Prepare a sample capsule by covering it with 3M tape, sealing the tape firmly around the edges. Cut away the excess tape with a sharp knife or razor blade. Using a syringe, add 700 µL of 1-octanol by puncturing the tape. Add 500 µL of LCS spiking solution, using a syringe to measure, by injecting the liquid through the hole used to inject the methanol. Immediately place the capsule in the capsule holder and assemble the oxygen bomb. Combust the contents of the sample capsule without delay according to sections 11.2.3 - 11.2.4. Collect the combustate solution (section 11.2.5 - 11.2.7), and analyze for bromide, chloride, fluoride and/or iodide, as required.

9.3. Matrix Spike/Matrix Spike Duplicate Samples: Add 500 µL of LCS/MS/MSD spiking solution to an aliquot of the sample equivalent to that analyzed for the unspiked sample. For samples diluted in methanol or acetone, add the same amount of diluted sample to a taped sample capsule as was used for the unspiked sample. Assemble the bomb and combust without delay according to section 11.2.4 - 11.2.5. Collect the combustate solution (11.2.5- 11.2.7), and analyze for bromide, chloride, fluoride and/or iodide.

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	The result should be less than or equal to the RL. Sample results greater than 20x the blank concentration or samples for which the contaminant is < RL do not require reprep.	Reprep and reanalyze samples.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	80-120% recovery for F, Cl; 70-130% recovery for Br, I.	Reprep and/or reanalyze all samples associated with the LCS.
Laboratory Duplicate	One per sample preparation batch of up to 10 samples. (Minimum one duplicate per trial burn)	RPD ≤ 10%	Flag the data if RPD > 10%. Note in the narrative.

Matrix Spike	One per sample preparation batch of up to 20 samples. For trial burn samples, one per trial burn.	80-120% recovery for F, Cl; 70-130% recovery for Br, I.	Flag the data if % recovery is outside QC acceptance limits. Note in the narrative.
Matrix Spike Duplicate	One per sample preparation batch of up to 20 samples. For trial burn samples, one per trial burn.	80-120% recovery for F, Cl; 70-130% recovery for Br, I. RPD ≤ 10%	Flag the data if RPD > 10%. Note in the narrative.

10. Calibration and Standardization

10.1. The analytical balance must have the calibration checked each day that analytical mass measurements are made. Calibrate the balance according to the manufacturer's instructions whenever the calibration check does not pass the daily criteria.

11. Procedure

11.1. All samples must be analyzed by combustion in an oxygen bomb. Higher concentration samples should not be combusted in an oxygen bomb unless the sample size is reduced to keep the amount of halide low organic. If a liquid sample is expected to contain > 5% halogen, dilute the sample in methanol or acetone prior to combustion.

11.2. Sample Preparation

11.2.1. Cleaning procedure for bomb and sample capsule: Remove any residual fuse wire from the terminals and the cup. Scrub the bomb with residue-free soap solution (for example, Conrad 70, Decon Labs). Using hot water, thoroughly rinse the interior of the bomb, the sample capsule, the terminals, and the inner surface of the bomb cover. Copiously rinse the bomb, cover, and cup with deionized water. Fill the bomb completely with deionized water and place the bomb head in bomb. Push into flask valve, and then leach for 15 minutes with deionized water.

11.2.2. Preparation of bomb and sample: Weigh and record the empty sample capsule weight.

11.2.2.1. Cut a piece of firing wire approximately 100 mm in length and attach the free ends to the terminals. Arrange the wire so that it is touching the sample.

11.2.2.2. Pipette 10 mL of the $NaHCO_3/Na_2CO_3$ solution into the bomb, wetting the sides. Add 50 uL hydrogen peroxide, 30%. Hydrogen peroxide is not necessary if neither bromine nor iodine is required to be determined.

11.2.2.3. Take an aliquot of the sample of 1.00 g or less, using an appropriate device (syringe, disposable pipette, spatula, etc.) and place in a tared sample capsule. If the sample contains volatile components, cover the capsule with 3M™ tape. Add 700 µL of 1-octanol. Obtain the exact weight of sample by first taring the capsule to zero, then adding the sample to the capsule and re-weighing.

11.2.2.3.1. Samples with high (5-25%) halogen content: Weigh 0.5-1.0 g sample into a clean 10, 25 or 50 mL volumetric flask. Samples should be diluted according to good judgment based on screening tests and historical data from repeat projects. Dilute to the mark with acetone or methanol. If the solutions are cloudy or otherwise heterogeneous, make a new solution a different solvent. Add 0.5 mL sample to a taped capsule. Assemble the bomb and combust without delay. For samples with very high halogen content (>25%), dilute 1 g sample to 50 mL.

11.2.2.3.2. Loose Solid Samples: Large particles may not burn completely and small particles are easily swept out of the capsule by turbulent gases during rapid combustion. Crush solid materials to obtain more surface area for combustion. If the material is very loose (e.g., fluffy organic crystals similar to benzoic acid, use the pellet press to make a pellet out of the loose solid material. Measure out approximately 1 gram of the sample and make a pellet out of it. Determine the weight of the pellet.

11.2.2.3.3. Combustion Aids: 700 µL of 1-octanol shall be routinely added to each sample, including those samples that are diluted in methanol or acetone. If a non-aqueous sample is difficult to ignite under these conditions, 0.5 mL acetone or methanol can be mixed with the sample. Combustion aids add to the total energy released in the bomb and the amount of sample may have to be reduced to compensate for the added charge.

11.2.2.3.4. Volatile Samples: Volatile samples can be handled in a standard sample capsule. These capsules can be sealed with a disc of plastic adhesive tape. Use the following procedure when filling and handling any of these tape-sealed sample capsules:

- Cover the sample capsule with tape and seal the tape on the edges. Trim the excess tape with a sharp knife or razor blade.
- Add the sample with a syringe.
- Set the cup in the electrode loop and arrange the wire fuse so that it touches the center of the tape disc.
- Fill the bomb with the usual oxygen charging pressure.

11.2.2.3.5. Aqueous Samples: Water inhibits combustion, and it is difficult to ignite samples that are composed of combustible materials dissolved in water. Use ashless (~0.3 to 0.5 g) filter paper to absorb the sample in the sample capsule. Add 700 µL of octanol and 500 µL of acetone or methanol to obtain complete combustion of the sample.

11.2.3. Oxygen Bomb Assembly and the Addition of Oxygen: Place the sample capsule in position and arrange the thread so that the end dips into the sample. Assemble the bomb and tighten the cover securely. Admit oxygen slowly (to avoid blowing the sample from the cup) until a pressure of 450 psi (~30 atm) is reached. Do not add oxygen or ignite the sample if the bomb has been jarred, dropped, or tilted.

11.2.4. Sample combustion: Completely immerse the bomb in a cool water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. Remove the bomb from the bath after immersion for at least 5 minutes.

11.2.5. At least 15 minutes after ignition, release the pressurized gas. Slowly release the pressurized contents of the bomb in a fume hood.

11.2.6. Open and inspect the bomb: After all of the pressurized gas has been released, open the bomb while rinsing the inner surface of the bomb head. As the bomb head is raised, rinse the electrodes of the sample capsule. Examine the contents. If traces of unburned oils or sooty deposits are found, discard the sample, and thoroughly clean the bomb before using it again. Process a new sample, taking care that the sample capsule is positioned properly and the ignition wire is in contact with the sample. If combustion is incomplete, vary the sample conditions and repeat.

11.2.7. Collection of halide solution: Tare a 125 mL wide-mouth HDPE sample bottle. Using deionized water, thoroughly rinse the interior of the bomb, the sample capsule, the terminals, and the inner surface of the bomb into the bomb casing. Decant the bomb contents into the tared HDPE sample container. Continue to rinse with deionized water rinses, decanting the rinse into the sample container each time. Weigh the sample container between rinses until the sample weight is 75-80 grams.

11.2.8. If bromine or iodine are to be determined, add 10 mL of hydrazine sulfate (2% solution).

11.2.9. pH adjustment

- Test and record the pH in the comments section of the sample preparation log.
- Raise the pH to 5 to 7 with 2N NaOH.
- Add 4.0 mL eluate stock solution and mix well.
- Test and record the final pH in the sample preparation log.

11.2.10. Transfer the contents of the sample collection bottle to a 500 mL graduated cylinder. Raise the volume to 400 mL (0.400 L) and transfer to a 500 mL HDPE sample bottle.

11.3. Sample Analysis: Analyze the combustate for the requested halides according to SOP KNOX-WC-0005, current revision, Anion Analysis. This preparation is applicable to analysis using the Dionex AS_14A column. It may be necessary to dilute the samples so that the concentration will fall within the range of standards.

Note: Care should be taken if a low-level sample is analyzed after a high level sample. If the preparation was performed in the oxygen bomb, repeat the low level sample preparation if carryover is suspected.

11.4. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure, except those specified by project specific instructions, shall be completely documented and approved by a Technical Specialist, Project Manager and QA Manager. If contractually required, the client shall be notified.

11.5. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. Data Analysis and Calculations

12.1. Calculations. Calculate the concentrations of each element detected in the sample according to the following equation:

$$C = \frac{C_{com} \times V_{com}}{W} \times 1000$$

where:

C = concentration of analyte in the sample, mg/kg
C_{com} = concentration of analyte in the combustate, mg/L (corrected for bench dilutions
V_{com} = total volume of combustate, L
W = weight of sample combusted, g.

12.1.1. Calculate the sample weight in the final prep as follows:

$$W = \frac{SW \, x \, V_{com}}{DV}$$

where:

W = sample weight in final prep, g
SW = sample weight before dilution (if any), g
DV = total dilution volume, mL
V_{com} = volume of sample dilution combusted, mL

12.2. Report the concentration of each halide detected in the sample in milligrams per kilogram (mg/kg).

12.3. The Percent Recovery is calculated using the following equation:

$$Percent\ Recovery\ (\%) = \frac{(X - S)}{T} \times 100$$

where:

X = Experimentally determined concentration of the spiked sample
S = Sample concentration before spiking
T = True concentration of the spike

12.4. The Percent Difference is calculated using the following equation:

$$\text{Relative Percent Difference (RPD)(\%)} = \left[\frac{(D_1 - D_2)}{\left(\frac{D_1 + D_2}{2} \right)} \right] \times 100$$

where: D_1 and D_2 = Results of duplicate measurements.

12.5. Refer to Appendix I for an example data review checklist used to perform and document the review of the data. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data.

13. Method Performance

13.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. Method Detection limits are determined and verified as specified in the current revision of SOP S-Q-003 (and attachment) based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit. MDL summaries are stored on the local area network.

13.2. Initial Demonstration of Capability: Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in STL Knoxville SOP KNOX-QA-0009.

13.3. Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009 current revision for further requirements for performing and documenting initial and on-going demonstrations of capability.

14. Pollution Prevention

14.1. All procedures shall be conducted in a manner to minimize, as far as practical, the use of solvents, reagents and other chemicals.

15. Waste Management

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2. Waste streams produced by the procedure: Excess sample waste will be collected and placed in containers specifically designated for this type of waste material.

16. References

16.1. STL Quality Management Plan, current revision.

16.2. STL Knoxville Laboratory Quality Manual (LQM), current revision.

16.3. Method 5050, Bomb Preparation Method for Solid Waste, USEPA SW-846, Test Methods for Evaluating Solid Waste, Third Edition.

16.4. Method 9056, Determination of Inorganic Anions by Ion Chromatography, USEPA SW-846 Test Methods for Evaluating Solid Waste, Third Edition.

16.5. ASTM Method E442-74 (1981). Standard Test Method for Chlorine, Bromine or Iodine in Organic Compounds by Oxygen Flask Combustion.

17. Miscellaneous

17.1. Modifications from the referenced method

17.1.1. Method 442

17.1.1.1. This method applies to total halogen determination in solid and liquid samples that are non-volatile at room temperature. All combustion is normally carried out in an oxygen flask at 1 atm. This SOP may also be used for volatile samples and reports results for total chloride and fluoride (not bromide and iodide). Also, all samples processed by this procedure will be combusted in an oxygen bomb at high pressure.

17.1.1.2. The analytical finish listed in the method is by titration, not ion chromatography as specified in this SOP.

17.1.1.3. The method uses KOH solution and H2O2. This SOP uses a bicarbonate buffer solution based on EPA Method 5050.

17.1.2. Method 5050

17.1.2.1. This method does not give any guidance for adding hydrogen peroxide to the buffer solution or using hydrazine sulfate to reduce more highly oxidized forms of bromine or iodine to bromide or iodide.

17.1.2.2. The buffer concentration has been increased from the promulgated method.

17.2. Appendix I: Example Project Narrative

17.3. Appendix I: Example Data Review Checklist

17.4. Appendix III: Example Spreadsheet

Appendix I: Example Project Narrative

PROJECT NARRATIVE

Lot Number:

Physical Testing

Total Chlorine:

The samples were prepared for total chlorine using SOP number KNOX-WC-0016 (based on ASTM Method E442 and SW-846 Method 5050). The sample is oxidized by combustion in an oxygen flask at atmospheric pressure or a bomb containing oxygen under pressure. The liberated halogen compounds are absorbed primarily as halides in a sodium carbonate/sodium bicarbonate buffer solution. The combustion products are collected by repeated rinsing of the combustion apparatus, and analyzed by ion chromatography in accordance with SOP KNOX-WC-0005 (based on SW-846 Method 9056). The results are calculated using the following equation:

$$C = \frac{C_{com} \times V_{com}}{W} \times 1000\, g/kg$$

Where:

C = concentration of analyte in the sample, mg/kg.
C_{com} = concentration of analyte in the combustate, mg/L.
V_{com} = total volume of combustate, L.
W = weight of sample combusted, g.

Appendix II: Example Data Review Checklist

STL Knoxville Total Halogens Data Review / Narrative Checklist
Methods: 5050 and 9056, SOPs: KNOX-WC-0016, Rev. 3 and KNOX-WC-0005, Rev. 8 **Page 1 of 1**

Batch Number		**Analytes:**	☐ Total Chlorine ☐ Total Fluorine ☐ Total Bromine ☐ Total Iodine		
Lot Numbers:					
Analysis Date:		**File ID:**		**ICAL File ID:**	

Review Items	NA	Y	N	**If No, why is data reportable?**	**2nd √**
1. Were PM checklists (LIM L40), Lot Summary and any applicable QAS reviewed?					
2. ICV within 90-110%R and ICB/CCB < 1/2 RL?					
3. CCVs/CCBs run after every 10 samples & end of run?					
4. Is %D ≤10% for each CCV?					
5. If CCV failed, was it rerun only once?					
6. LCS/LCSD analytes within 80-120%R? *If no, list LCS ID:*				☐ **[lcs3] LCS recovery >120% and sample results <RL.***	
7. Method blank < RL? *If no, list blank ID:*				☐ **[mb3] No analyte > RL in associated samples.*** ☐ [mb8] Sample results > 20x higher than blank.	
8. MS/MSD done per prep batch?					
9. MS/MSD within 80-120% recovery and ≤10 RPD? *If no, list ID:*				☐ [ms3] LCS acceptable indicating sample matrix effects. ☐ [ms4] Native analyte concentration >4x spike level.	
10. DUP done per 10 samples or per trial burn?					
11. DUP RPD ≤ 10%? *If no, list ID:*				☐ [rpd] OS and/or DUP < RL. ☐ [rpd2] LCS acceptable. Sample heterogeneity.	
12. Were MS run #'s assigned correctly?					
13. Sample analyses done within holding time (HT)? *If no, list samples:*				☐ [ht1] HT expired upon receipt. ☐ *[ht2] Analysis requested after HT expired.**	
14. Were results processed using correct ICAL?					
15. Are positive results within the calibration range?					
16. Is integration acceptable for all samples, QC samples and standards?					
17. For manual integrated standards and QC samples, are before/after chromatograms provided with initials/date/reason?				Reasons: S=Split peak, U=Undetected peak, I=Incorrect peak integration, B=Baseline correction, W=Wrong peak chosen by data system.	
18. Calculations checked for error? (Document manual calculation checks.)					
19. Were spreadsheets checked for transcription errors?					
20. Are results below RL obtained from undiluted IC runs?				☐ [elev1] Elevated RLs due to matrix interfernces.	
21. For results below RL, were samples prepared using at least 0.5 g sample weight in final prep?					
22. F6 report correct? (Verify results, RLs, units, qualifiers, DFs, dates, spikes.)					

Reviewed by:	**Date:**	**2nd Level Reviewed by:**	**Date:**
Comments:		**Comments:**	

Final Review by:	**Date:**	NA	Y	N	**If No, why is data reportable?**
1. Are all NCMs documented and discussed in narrative?					List NCM #:
2. Narrative correct? (Appropriate autotext included and all deviations noted.)					☐ [chlor] Total chlorine. ☐ [hal] Total chlorine, fluorine, bromine, iodine.
3. For trial burn samples, are sample results from the same waste feed consistent? (Spread of values < 20% of avg.)					☐ [tb1] Related PT data show similar variation. ☐ [tb2] Solid samples likely to be heterogeneous. ☐ [tb3] Reactive matrix. ☐ [tb4] Mutiphasic samples run as one analysis per client.
4. For trial burn samples, do the results agree within 10% of the known or presumed values?					☐ [tb5] Related PT data show similar variation. ☐ [tb6] Extremely volatile materials suspected. ☐ [tb7] Samples obviously impure (e.g., cloudy, biphasic).
Comments:					

* Such action must be taken in consultation with client. WC084R3.DOC, 9/01/06

Nonconformance memos are required for **bold** and *italicized* [autotext] statements: **Bold** = deficiency, *italicized* = anomoly.

Appendix III: Example Spreadsheet

STL Knoxville
SOP KNOX-WC-0016
Total Halogen Data Worksheet

Batch # : ______________
Prep Date: ______________
Comp Date: ______________

Analyte:
CAS #:
Quantims #:

Analyst Initials: ________
Analyst LIMS Code: ________

Reviewed by: ________
Instr. ID: ________

Date: ______________

Lot Number	Lot Sample Number	Suffix	Method Code	QuantIMS LDL (mg/kg)	WO #	Sample Weight (g)	Dilution Volume (mL)	Diluted Aliquot Combusted (mL)	Sample Weight in Final Prep (g)	Final Prep Volume (L)	Bench DF	IC Conc. (mg/L)	Total Halide in Prep (mg)	Sample Conc. (mg/kg)	LIMS DF	Analysis Date (mmddyy)	Analysis Time (hhmm)

Total Halogens (Rev 2)1

Total Halogens rev 2.xlt

WC087R2, 04/26/06

Appendix III: Example Spreadsheet, cont'd.

STL Knoxville
SOP KNOX-WC-0016
Total Halogen Data Worksheet

Spike Information:

Spike	**Standard ID**	**Spike Vol. (mL)**	**Std. Conc. (ug/mL)**	**Work Order**	**Sample Weight in Final Prep (g)**	**True Spike Concentration (mg/kg)**
LCS						
LCSD						
MS (1)						
MSD (1)						
MS (2)						
MSD (2)						
MS (3)						
MSD (3)						
MS (4)						
MSD (4)						
MS (5)						
MSD (5)						

Comments:

True Spike Concentration (mg/kg or ug/g) = Standard Concentration (ug/mL) x Spike Volume (mL) / Sample Weight in Final Prep (g)

CONFIDENTIAL

Vista
Analytical Laboratory

SOP 36	**Revision: 0**	**Supersedes: 1CC, Rev2**

AIR TOXICS

SAMPLE PREPARATION OF MM5 TRAIN FOR ANALYSIS OF PCDDs/PCDFs/PCBs/PAHs by EPA METHOD 0023A/CARB 428/CARB 429

Analyst review:

Management:

Quality Assurance:

Effective Date: 29 January 2008

Revision No.	Description of Revision

1 PURPOSE

This analytical procedure (AP) describes the preparation of Modified Method 5 (MM5) sampling trains for the analysis of polychlorinated dibenzo-dioxins and furans (PCDDs/PCDFs) by USEPA Method 0023A, polychlorinated biphenyls (PCBs) by California Air Resources Board (CARB) Method 428, and polycyclic aromatic hydrocarbons (PAHs) by CARB Method 429.

2 SCOPE

2.1 All differences between the above EPA and CARB Methods and actual laboratory techniques have been developed to reduce interferences and increase sensitivity.

2.2 Data determined to be out-of-control from criteria stated within this AP is handled according to procedures addressed within the applicable section. Other options of handling out-of-control data are discussed in SOP 7H.

3 SUMMARY OF THE METHOD

3.1 This procedure uses air-specific extraction, analyte-specific cleanup, and HRGC/HRMS analysis techniques.

3.2 Detection limits are sample-specific based on factors in the formula (does not apply to PCBs and PAHs).

3.3 The sampling train consists mainly of five components:

3.3.1 Component 1 includes the filter with sampled particulate matter.

3.3.2 Component 2 is the XAD-2 sorbent.

3.3.3 Component 3 includes the solvent rinses from the front half of the train.

3.3.4 Component 4 includes the solvent rinses from the back half of the train.

3.3.5 Component 5 includes the impinger contents and rinses.

3.4 After sampling, the following components are combined to begin extraction:

3.4.1 The filter (component 1) is combined with the front half rinses (component 3) and placed into the soxhlet and spiked with the appropriate internal standards.

3.4.2 The XAD-2 sorbent (component 2) is combined with the back half rinses (component 4) and placed into a separate soxhlet and spiked with the appropriate internal standards.

3.4.3 Component 5 (impinger contents and rinses) is spiked with appropriate alternate standards and extracted using the separatory funnel shake-out.

4 APPARATUS AND MATERIALS

4.1 CTC Autosampler Model A200S.

4.2 Alpha Station Model 500 with Opus Data System.

4.3 Neslab HX200 and HX500 Water Cooler.

4.4 Micromass Ultima Autospec High Resolution Mass Spectrometer.

4.5 Pipet, disposable, serological, 10mL.

4.6 Amber glass bottles 500mL (teflon-lined screw cap).

4.7 Two-liter separatory funnels.

4.8 Soxhlet extractor.

4.9 Teflon boiling chips.

4.10 Glass Thimbles.

4.11 Organomation 24-Station N-Evaporator with Teflon tubing connection to trap and gas regulator.

4.12 Conical vials, 2mL.

4.13 Glass fiber filters and glass wool plugs.

4.14 Funnels 100mL (CMS).

4.15 Graduated cylinders, 500mL.

4.16 Buchler Rotary Evaporator.

4.17 Round bottom flasks, 50mL and 500mL.

4.18 Fischer Scientific Top-Loader Balance, Model XL-3000.

4.19 Injection vial inserts, 100µL (Sun International).

4.20 Electrothermal Electromantle six sample and 3000mL capacity.

4.21 VWR Oven.

5 REAGENTS, STANDARDS AND SOLVENTS

5.1 Reagents

5.1.1 Sulfuric acid concentrated.

5.1.2 Silica gel. Highest purity grade.

5.1.3 Water, distilled.

5.1.4 Florisil. Supelclean LC-Florisil.

5.1.5 Prepurified nitrogen gas.

5.1.6 Anhydrous sodium sulfate. Pour several portions of methylene chloride through the anhydrous sodium sulfate under vacuum.

5.1.7 Sodium Hydroxide. Highest available purity.

5.2 Solvents

5.2.1 Methylene chloride. Highest available purity.

5.2.2 Hexane. Highest available purity.

5.2.3 Tetradecane. Highest available purity.

5.2.4 Acetone. Highest available purity.

5.2.5 Toluene. Highest available purity.

5.2.6 Methanol. Highest available purity.

5.3 Standards

5.3.1 Analytical standards obtained from a certified vendor.

6 COLLECTION, PRESERVATION, and HANDLING

6.1 Glass XAD_2 resin traps and glass fiber filters are used for the collection of air toxins during the operation of a sampling train.

6.2 Amber glass bottles and jars must be used for collection of solvent rinses and impinger waters.

6.3 All sample components should be stored at 4° C in the dark, extracted within 21 days and completely analyzed within 40 days from extraction.

7 QUALITY CONTROL

7.1 Method Blank (MB): A method blank is an XAD and filter preparation that is free of native analyte and has been prepared and analyzed using the same procedures followed for the rest of the analytical batch.

7.1.1 A method blank is analyzed with every analytical batch.

7.1.2 Method 0023A/CARB 428: If the amount found is greater than the minimum level or one-third the regulatory compliance limit, whichever is greater; or if any potentially interfering compound is found in the blank at or above the minimum level for each congener, the data must be evaluated to determine whether the batch shall be re-extracted or the data is qualified appropriately.

7.1.3 Method 429: The laboratory method blank must not contain any of the target analytes listed in Table 3b. at levels exceeding the Quantitation Limit or 5 percent of the analyte concentration in the field sample. If the levels in the method blank exceed the acceptance criteria, then the data must be evaluated to determine whether the batch shall be re-extracted or the data is qualified appropriately

7.2 An LCS pair or OPR is analyzed with every analytical batch depending upon the method requested. An OPR is required for EPA Method 0023A Dioxin/Furan and CARB 428 for PCB analyses. An LCS pair is required for CARB 429.

7.2.1 Laboratory Control Sample (LCS): A pair of laboratory control samples is prepared by adding a known quantity of native standards to a native free matrix and used to assess method performance (precision and accuracy).

7.2.1.1 A pair of LCSs is analyzed with every set of samples for CARB 429.

7.2.1.2 The LCS results for each native isomer should have a relative percent difference (%RPD) less than 50%.

7.2.1.3 The acceptance criteria for percent recovery is 50-150% for native and internal standards. If all compounds do not meet acceptance criteria, re-analyze LCSs. If the LCS pair fails again, the archive extracts are worked up and analyzed.

7.2.2 Ongoing Precision and Recovery (OPR): An ongoing precision and recovery sample is prepared by adding a known quantity of native standard to a native free matrix and used to assess method performance (precision and recovery).

7.2.2.1 An OPR is analyzed with every analytical batch or 20 samples (whichever is less) per matrix type.

7.2.2.2 Method 0023A: The acceptance criteria for percent recovery is 70-130% for natives and 40-135% for internal standards.

7.2.2.3 CARB 428: The acceptance criteria for percent recovery is 70-130% for natives and 40-120% for internal standards.

8 EXTRACTION

8.1 Each sample is comprised of a filter, an XAD_2 resin trap, solvent rinses and aqueous impinger contents. The resin and filter are extracted once with methylene chloride and once with toluene. See attached flow charts outlining extraction sequences for each method.

8.2 Impinger Contents

8.2.1 Transfer aqueous impinger contents to an appropriate separatory funnel. Spike the appropriate amount of PAH AS into the separatory funnel. Rinse all containers with $MeCl_2$ and add to the separatory funnel. Drain any organic solvent into a 500mL round bottom flask and combine this with the solvent rinses discussed in 9.2.3.

8.2.2 Extract aqueous fraction in the separatory funnel with 60mL of methylene chloride. Drain methylene chloride into a 500mL amber glass jar. Repeat extraction step twice.

8.2.3 Impinger rinses are combined and roto-evaporated down and combined with the impinger aqueous fraction solvent.

8.3 Solvent Rinses

8.3.1 Roto-evaporate the back half rinses to less than 5mL and add to the round bottom with the XAD-2 sorbent.

8.3.2 Repeat 9.3.1 for the front half rinses in a separate soxhlet extractor to be use with the filter in a separate round bottom.

8.4 Resin and Filter (separate methylene chloride and toluene extractions)

8.4.1 Rinse XAD-2 sorbent into a glass thimble. Place a glass wool plug into the thimble on top of the resin. Carefully slide the thimble into a pre-cleaned soxhlet extractor. Place extractor atop the 500mL round bottom flask from 9.3.1. Add Teflon boiling chips and 300mL of methylene chloride (MeCl2).

8.4.2 Repeat Step 9.4.1 for the filter and the front half rinse.

8.4.3 Spike the appropriate amount of D/F, PCB and PAH internal standards indicated on the process sheet into the thimble. Into the LCSs/OPR, also spike the D/F, PCB and PAH native spikes.

8.4.4 Assemble the soxhlet apparatus to the condenser and place upon a heating mantle. Turn on heating units and coolers and set temperature for methylene chloride to boil. Allow refluxing for 16 hours at the rate of 3 cycles per hour.

8.4.5 After allowing soxhlet apparatus to cool, drain all solvent remaining in the extractor into the round bottom flask.

8.4.6 Remove the round bottom flask without overturning the soxhlet apparatus, and carefully drain the extract through $MeCl_2$ rinsed sodium sulfate (Na_2SO_4) into a pre-cleaned 500mL amber glass jar. Rinse the funnel and sodium sulfate with $MeCl_2$ and allow to drain completely into the jar. Save funnel and sodium sulfate. Cap and shake jar. Place soxhlet extractor back atop the flask and add more boiling chips if necessary. Add 300mL of toluene. Turn on heating units and coolers and set temperature for toluene to boil. Allow refluxing for 16 hours at the rate of 3 cycles per hour. Spike the toluene portion of the front half with D/F alternate standard in the amount indicated on the process sheet.

8.4.7 Repeat process in 9.4.6 but discard resin/filter/sodium sulfate into lab pack.

9 SPLITTING/COMBINING

9.1 Using graduated cylinders, measure volumes of the $MeCl_2$ soxhlet extract, the toluene soxhlet extract, and the impinger extract. Split each of the fractions into the following four equal parts.

9.1.1 ¼ of each MeCl2 soxhlet fraction combined with ¼ of the impinger extract for PAH analysis.

9.1.2 ¼ of each MeCl2 soxhlet fractions are combined with ¼ of each toluene soxhlet fractions, with ¼ impinger extract for PCB analysis.

9.1.3 ¼ of the MeCl2 soxhlet fraction from the filter soxhlet is combined with ¼ of the toluene fraction from the filter soxhlet for PCDD/PCDF analysis for the front half part of the train.

9.1.4 ¼ of the MeCl2 soxhlet fraction from the XAD-2 soxhlet is combined with ¼ of the toluene soxhlet fraction for PCDD/PCDF analysis for the back half part of the train.

9.1.5 The remaining ¼ fractions will be stored as the archive portions.

10 CLEANUP

10.1 Dioxin/Furan Cleanup

10.1.1 Add 100-150µL of tetradecane (C_{14}) to the extract to be processed. Roto-evaporate the extract until only the C_{14} remains. Proceed with the following clean-ups.

10.1.2 Florisil Modified Silica Gel Filtration (Figures 1)

10.1.2.1 Pack a 15mm x 200mm column and a 11mm x 160mm column as per Figures 2 and 3. Pre-rinse both columns with ~20mL of hexane

10.1.2.2 Place top column into bottom column and transfer C_{14} sample extract to the top column. Let sample elute through both top and bottom columns

10.1.2.3 Rinse the round bottom flask with hexane and elute the rinse through both top and bottom columns.

10.1.2.4 Discard rinses.

10.1.2.5 Elute with ~ 100mL of hexane through both top and bottom column and discard.

10.1.2.6 Remove top column and elute bottom column with ~25mL of hexane and discard.

10.1.2.7 Elute ~45mL of methylene chloride through bottom column and collect into a 50mL round bottom flask.

10.1.2.8 When column stops dripping remove round bottom flask and roto-evaporate to ~1mL. Transfer extract to a 2mL conical vial containing 10µL of C_{14} and 10µL of D/F Recovery Standard. Rinse flask with ~1mL of methylene chloride and transfer rinse to vial.

10.1.2.9 Using Nitrogen evaporation, concentrate the extract until only the C_{14} remains. Transfer the extract using a 20µL micropipet into an autoinjection vial and cap vial with a crimp top cap. Final volume is 20µL. Submit for analysis.

10.2 PCB Cleanup

10.2.1 For the PCB portion, concentrate the extract to be processed to less than 5mL. Add 60mL hexane to the extract and roto-evaporate to less than 5mL. Add an additional 60mL of hexane and roto-evaporate again to less than 5mL. Proceed with the following cleanup.

10.2.2 Silica Gel Filtration (Figure 2)

10.2.2.1 Pack a 11mm x 160mm column as shown in Figure 2. Pre-rinse the column with 20mL of hexane. Discard the rinse.

10.2.2.2 Transfer the sample extract onto the column using a disposable pipette. Rinse the container twice with additional hexane and add to the column. Begin collecting the eluate into a 250mL round bottom flask.

10.2.2.3 Just prior to the exposure of the sodium sulfate layer to air, begin elution of the column with 25mL of hexane. After the hexane has passed, elute with 30mL of 40% $MeCl_2$/Hexane. Continue collection of these eluates into the 250mL round bottom flask.

10.2.2.4 Roto-evaporate the eluate to less than 5mL. Quantitatively transfer to an 8mL test tube, using a hexane rinse.

10.2.2.5 Concentrate to 0.5mL using a gentle stream of nitrogen. The nitrogen for PCB concentration must be purity grade 5.0 and charcoal filtered.

10.2.2.6 Add 10uL of PCB-Recovery standard to a conical vial containing 100uL nonane. Transfer the concentrated extract to the vial and continue to nitrogen-evaporate until only the nonane remains. Transfer the extract using a 50µL micropipet into a pre-cleaned amber autoinjection vial and cap vial with a crimp top cap. Final volume is 100µL. Submit for analysis.

10.3 PAH Cleanup

10.3.1 For the PAH portion, concentrate the extract to less than 5mL. Add 60mL hexane to the extract and roto-evaporate to less than 5mL. Proceed with the following clean-up.

10.3.2 Silica Gel Filtration (Figure 2)

10.3.2.1 Pack a 11mm x 160mm column as shown in Figure 2. Pre-rinse the column with 20mL of hexane. Discard the rinse.

10.3.2.2 Transfer the sample extract onto the column using a disposable pipette. Rinse the container twice with additional hexane and add to the column. Begin collecting the eluate into a 250mL round bottom flask.

10.3.2.3 Just prior to the exposure of the sodium sulfate layer to air, begin elution of the column with 25mL of hexane. After the hexane has passed, elute with 30mL of 40% $MeCl_2$/Hexane. Continue collection of these eluates into the 250mL round bottom flask.

10.3.2.4 Roto-evaporate the eluate to less than 5mL. Quantitatively transfer to an 8mL test tube, using a hexane rinse.

10.3.2.5 Concentrate to 0.5mL using a gentle stream of nitrogen. The nitrogen for PAH concentration must be purity grade 5.0 and charcoal filtered.

10.3.2.6 Add 50µL of PAH-Recovery standard to a pre-cleaned amber autoinjection vial. Transfer the concentrated extract to the vial and cap vial with a crimp top cap. Final volume is 500µL. Submit for analysis.

11 POLLUTION PREVENTION

11.1 The solvents used in this method pose little threat to the environment when managed properly. The solvent evaporation techniques used in this method are amenable to solvent recovery, and the laboratory shall recover solvents wherever feasible.

11.2 Standards should be prepared in volumes consistent with laboratory use to minimize disposal of standard.

12 WASTE MANAGEMENT

12.1 Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. Safety officer should be contacted if additional information is required.

12.2 The laboratory waste management is in compliance with all federal, state, and local regulations to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations.

13 REFERENCES

13.1 California Air Resources Board (CARB) Method 428

13.2 California Air Resources Board (CARB) Method 429

13.3 U.S. Environmental Protection Agency (USEPA) Method 0023A.

Figure 1

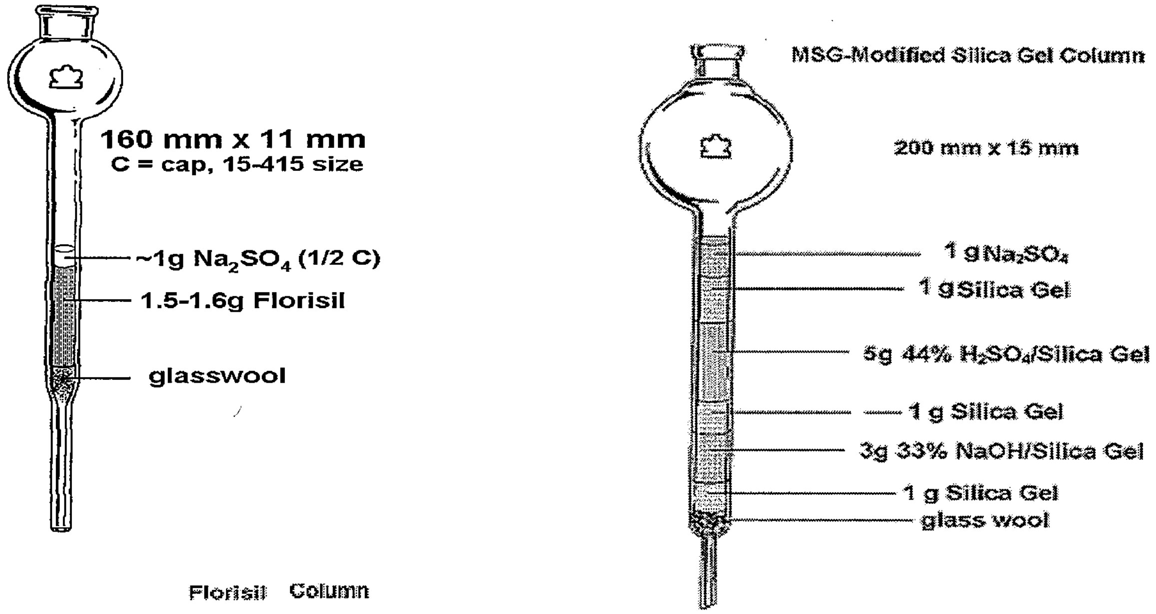


Figure 2

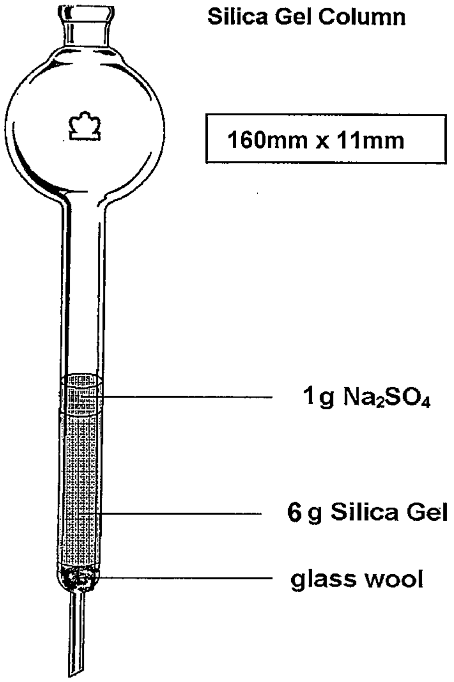


Table 1. Concentrations of PCDD/PCDFs in Calibration Solutions (pg/uL)

Compound PCDD/PCDF	Calibration Solutions (pg/uL)						
Calibration Standards	CS0	CS1	CS2	CS3	CS4*	CS5	CS6
2,3,7,8-TCDD	0.25	0.50	1.0	5.0	50	100	500
2,3,7,8-TCDF	0.25	0.50	1.0	5.0	50	100	500
1,2,3,7,8-PeCDD	1.25	2.5	5.0	25	250	500	2500
1,2,3,7,8-PeCDF	1.25	2.5	5.0	25	250	500	2500
2,3,4,7,8-PeCDF	1.25	2.5	5.0	25	250	500	2500
1,2,3,4,7,8-HxCDD	1.25	2.5	5.0	25	250	500	2500
1,2,3,6,7,8-HxCDD	1.25	2.5	5.0	25	250	500	2500
1,2,3,7,8,9-HxCDD	1.25	2.5	5.0	25	250	500	2500
1,2,3,4,7,8-HxCDF	1.25	2.5	5.0	25	250	500	2500
1,2,3,6,7,8-HxCDF	1.25	2.5	5.0	25	250	500	2500
1,2,3,7,8,9-HxCDF	1.25	2.5	5.0	25	250	500	2500
2,3,4,6,7,8-HxCDF	1.25	2.5	5.0	25	250	500	2500
1,2,3,4,6,7,8-HpCDD	1.25	2.5	5.0	25	250	500	2500
1,2,3,4,6,7,8-HpCDF	1.25	2.5	5.0	25	250	500	2500
1,2,3,4,7,8,9-HpCDF	1.25	2.5	5.0	25	250	500	2500
Internal Standards							
13C-2,3,7,8-TCDD	100	100	100	100	100	100	100
13C-2,3,7,8-TCDF	100	100	100	100	100	100	100
13C-1,2,3,7,8-	100	100	100	100	100	100	100
PeCDD	100	100	100	100	100	100	100
13C-1,2,3,7,8-PeCDF	100	100	100	100	100	100	100
13C-1,2,3,6,7,8-	100	100	100	100	100	100	100
HxCDD	100	100	100	100	100	100	100
13C-1,2,3,6,7,8- HxCDF	100	100	100	100	100	100	100
Surrogate Standards							
37Cl4-2,3,7,8-TCDD	10	15	20	25	30	35	40
13C-2,3,4,7,8-PeCDF	40	60	80	100	120	140	160
13C-1,2,3,4,7,8-	40	60	80	100	120	140	160
HxCDD	40	60	80	100	120	140	160
13C-1,2,3,4,7,8-	40	60	80	100	120	140	160
HxCDF							
13C-1,2,3,4,7,8,9- HpCDF							
Recovery Standards							

13C-1,2,3,4-TCDD	100	100	100	100	100	100	100
13C-1,2,3,4-TCDF	100	100	100	100	100	100	100
13C-1,2,3,4,7,8,9-	100	100	100	100	100	100	100
Alternate Standard							
13C-1,2,3,7,8,9-HxCDF	100	100	100	100	100	100	100

* Calibration Verification Solution

Table 2. Concentration of PCBs in Calibration Solutions (pg/uL)

2a. PCB Native Standards	IUPAC	CS1	CS2	CS3	CS4	CS5
Monochlorobiphenyl (MCB[1])		0.25	2.5	25	125	500
Dichlorobiphenyl (DiCB)		0.25	2.5	25	125	500
Trichlorobiphenyl (TriCB)		0.25	2.5	25	125	500
Tetrachlorobiphenyl (TCB)		0.50	5.0	50	250	1000
Pentachlorobiphenyl (PeCB)		0.50	5.0	50	250	1000
Hexachlorobiphenyl (HxCB)		0.50	5.0	50	250	1000
Heptachlorobiphenyl (HpCB)		0.50	5.0	50	250	1000
Octachlorobiphenyl (OCB)		0.75	7.5	75	375	1500
Nonachlorobiphenyl (NCB)		0.75	7.5	75	375	1500
Decachlorobiphenyl (DeCB)		0.75	7.5	75	375	1500
2b. PCB Internal Standards						
^{13}C-2-Mono-CB	1	100	100	100	100	100
^{13}C-4-Mono-CB	3	100	100	100	100	100
^{13}C-2,2'-Di-CB	4	100	100	100	100	100
^{13}C-2,5-Di-CB	9	100	100	100	100	100
^{13}C-2,2',6-Tri-CB	19	100	100	100	100	100
^{13}C-2,4,4'-Tri-CB	28	100	100	100	100	100
^{13}C-2,4',6-Tri-CB	32	100	100	100	100	100
^{13}C-3,4,4'-Tri-CB	37	100	100	100	100	100
^{13}C-2,2',6,6'-Tetra-CB	54	100	100	100	100	100
^{13}C-3,3',4,4'-Tetra-CB	77	100	100	100	100	100
^{13}C-3,4,4',5'-Tetra-CB	81	100	100	100	100	100
^{13}C-2,2',3,5',6-Penta-CB	95	100	100	100	100	100
^{13}C-2,2',4,5,5'-Penta-CB	101	100	100	100	100	100
^{13}C-2,2',4,6,6'-Penta-CB	104	100	100	100	100	100
^{13}C-2,3,3',4,4'-Penta-CB	105	100	100	100	100	100
^{13}C-2,3,4,4',5-Penta-CB	114	100	100	100	100	100
^{13}C-2,3',4,4',5-Penta-CB	118	100	100	100	100	100
^{13}C-2',3,4,4',5-Penta-CB	123	100	100	100	100	100
^{13}C-3,3',4,4',5-Penta-CB	126	100	100	100	100	100
^{13}C-2,2',4,4',5,5'-Hexa-CB	153	100	100	100	100	100
^{13}C-2,2',4,4',6,6'-Hexa-CB	155	100	100	100	100	100
^{13}C-2,3,3',4,4',5-Hexa-CB	156	100	100	100	100	100
^{13}C-2,3,3',4,4',5'-Hexa-CB	157	100	100	100	100	100
^{13}C-2,3',4,4',5,5'-Hexa-CB	167	100	100	100	100	100
^{13}C-3,3',4,4',5,5'-Hexa-CB	169	100	100	100	100	100

2c. Internal Standards (cont.)						
^{13}C-2,2’,3,3’,4,4’,5-Hepta-CB	170	100	100	100	100	100
^{13}C-2,2’,3,4,4’,5,5’-Hepta-CB	180	100	100	100	100	100
^{13}C-2,2’,3,4’,5,6,6’-Hepta-CB	188	100	100	100	100	100
^{13}C-2,3,3’,4,4’,5,5’-Hepta-CB	189	100	100	100	100	100
^{13}C-2,2’,3,3’,4,4’,5,5’-Octa-CB	194	100	100	100	100	100
^{13}C-2,2’,3,3’,5,5’,6,6’-Octa-CB	202	100	100	100	100	100
^{13}C-2,2’,3,3’,4,4’,5,5’,6-Octa-CB	206	100	100	100	100	100
^{13}C-2,2’,3,3’,4,5,5’,6,6’-Nona-CB	208	100	100	100	100	100
^{13}C-Deca-CB	209	100	100	100	100	100
2d. Recovery Standards						
^{13}C-4,4’-Di-CB	15	100	100	100	100	100
^{13}C- 2,4’,5-Tri-CB	31	100	100	100	100	100
^{13}C- 2,3,4,4’-Tetra-CB	60	100	100	100	100	100
^{13}C-2,3,3’,5,5’-Penta-CB	111	100	100	100	100	100
^{13}C- 2,2’,3,4,4,’,5’-Hexa-CB	138	100	100	100	100	100
^{13}C- 2,3,3’,4,4’,5,5’,6-Octa-CB	205	100	100	100	100	100
2e. Prespike Standards						
^{13}C-2,2’,5,5’-Tetra-CB	52	100	100	100	100	100
^{13}C-2,2’,3,3’,5,5’,6-Hepta-CB	178	100	100	100	100	100

Table 3. Concentration of PAHs in Calibration Solutions (pg/uL)

PAH Calibration Standards	**CS1**	**CS2**	**CS3**	**CS4**	**CS5**
Naphthalene	25	125	250	500	1250
Napthalene-2nd	25	125	250	500	1250
2-Methylnaphthalene	10	50	100	200	500
Acenapthylene	10	50	100	200	500
Acenaphthene	10	50	100	200	500
Fluorene	10	50	100	200	500
Phenanthrene	25	125	250	500	1250
Phenanthrene-2nd	25	125	250	500	1250
Anthracene	10	50	100	200	500
Fluoranthene	10	50	100	200	500
Pyrene	10	50	100	200	500
Benzo(a)anthracene	10	50	100	200	500
Chrysene	10	50	100	200	500
Perylene	10	50	100	200	500
Benzo(b)fluoranthene	10	50	100	200	500
Benzo(k)fluoranthene	10	50	100	200	500
Benzo(e)pyrene	10	50	100	200	500
Benzo(a)pyrene	10	50	100	200	500
Benzo(g,h,i)perylene	10	50	100	200	500
Indeno(1,2,3-cd)pyrene	10	50	100	200	500
Dibenz(a,h)anthracene	10	50	100	200	500
Internal Standards					
d_8-Naphthalene	100	100	100	100	100
d_8-Acenapthylene	100	100	100	100	100
d_{10}-Acenaphthene	100	100	100	100	100
d_{10}-Fluorene	100	100	100	100	100
d_{10}-Phenanthrene	100	100	100	100	100
d_{10}-Fluoranthene	100	100	100	100	100
d_{12}-Benzo(a)anthracene	100	100	100	100	100
d_{12}-Chrysene	100	100	100	100	100
d_{12}-Benzo(b)fluoranthene	200	200	200	200	200
d_{12}-Benzo(k)fluoranthene	200	200	200	200	200
d_{12}-Benzo(a)pyrene	200	200	200	200	200
d_{12}-Benzo(g,h,i)perylene	200	200	200	200	200
d_{12}-Indeno(1,2,3-cd)pyrene	200	200	200	200	200
d_{14}-Dibenz(a,h)anthracene	200	200	200	200	200
Surrogate Standards					
d_{12}-Benzo(e)pyrene	200	200	200	200	200
d_{14}-Terphenyl	200	200	200	200	200
Alternate Standard					
d_{10}-Anthracene	100	100	100	100	100
Recovery Standards					
d_{10}-1-Methyl-naphthalene	100	100	100	100	100
d_{10}-Pyrene	100	100	100	100	100
d_{12}-Perylene	100	100	100	100	100

Table 4. Quantitation Limits for the Determination of Total PCBs Only

Total PCB Congeners	Quantitation Limits Air (pg/sample)
Monochlorobiphenyl (MCB[1]) Homologues[2]	100 – 200,000
Dichlorobiphenyl (DiCB) Homologues[2]	100 – 200,000
Trichlorobiphenyl (TriCB) Homologues[2]	100 – 200,000
Tetrachlorobiphenyl (TCB) Homologues[2]	200 – 400,000
Pentachlorobiphenyl (PeCB) Homologues[2]	200 – 400,000
Hexachlorobiphenyl (HxCB) Homologues[2]	200 – 400,000
Heptachlorobiphenyl (HpCB) Homologues[2]	300 – 600,000
Octachlorobiphenyl (OCB) Homologues[2]	300 – 600,000
Nonachlorobiphenyl (NCB) Homologues[2]	300 – 600,000
Decachlorobiphenyl (DeCB) Homologues[2]	300 – 600,000

[1] CB = Chlorobiphenyl

[2] Quantitation limits are based upon the single PCB congener, which is used to calibrate for the homologue series assuming a four-way split.

Table 4a. Quantitation Limits for the Determination of Dioxin/Furans

Dioxin/Furans	Quantitation Limits[3] Air (pg/sample)
Tetra	20 – 16,000
Penta-Hepta	100 – 80,000
Octa	200 – 160,000

[3] Quantitation limits are based upon a four-way split.

Table 4b. Quantitation Limits for the determination of PAHs

PAH	Quantitation Limits[3] Air (ng/sample)
Naphthalene	50 – 2500
2-Methylnaphthalene	20 – 1000
Acenapthylene	20 – 1000
Acenaphthene	20 – 1000
Fluorene	20 – 1000
Phenanthrene	50 – 2500
Anthracene	20 – 1000
Fluoranthene	20 – 1000
Pyrene	20 – 1000
Benzo(a)anthracene	20 – 1000
Chrysene	20 – 1000
Perylene	20 – 1000
Benzo(b)fluoranthene	20 – 1000
Benzo(k)fluoranthene	20 – 1000
Benzo(e)pyrene	20 – 1000
Benzo(a)pyrene	20 – 1000
Benzo(g,h,i)perylene	20 – 1000
Indeno(1,2,3-cd)pyrene	20 – 1000
Dibenz(a,h)anthracene	20 – 1000

[3] Quantitation limits are based upon a four-way split.

Table 5

Theoretical Ion Abundance Ratios and Control Limits for PCDDs/PCDFs and PCBs

DIOXINS AND FURANS

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits(1)	
			Lower	Upper
4(2)	M/M+2	0.77	0.65	0.89
5 (CDD)	M+2/M+4	0.65	0.55	0.75
5 (CDF)	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6(3)	M/M+2	0.51	0.43	0.59
7	M+2/M+4	1.05	0.88	1.20
7(4)	M/M+2	0.44	0.37	0.51
8	M+2/M+4	0.89	0.76	1.02

PCBs

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits(5)	
			Lower	Upper
1	M/M+2	3.13	2.66	3.60
2	M/M+2	1.56	1.33	1.81
3	M/M+2	1.04	0.88	1.20
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.56	1.33	1.79
6	M+2/M+4	1.25	1.06	1.44
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02
9	M+4/M+6	1.34	1.14	1.54
10	M+4/M+6	1.17	0.99	1.34

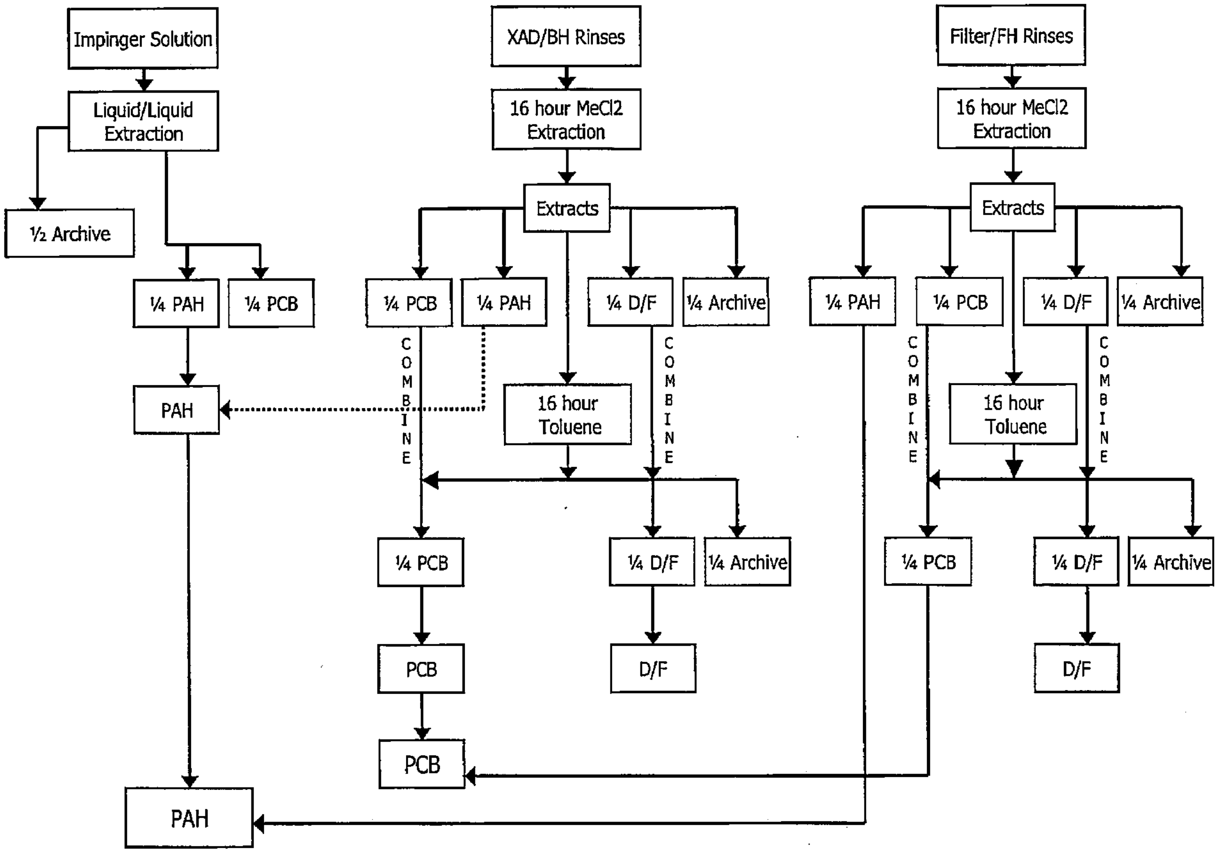
(1) Represents ± 15% windows around the theoretical ion abundance ratios

(2) Does not apply to $^{37}Cl_4$-2,3,7,8-TCDD (cleanup standard)

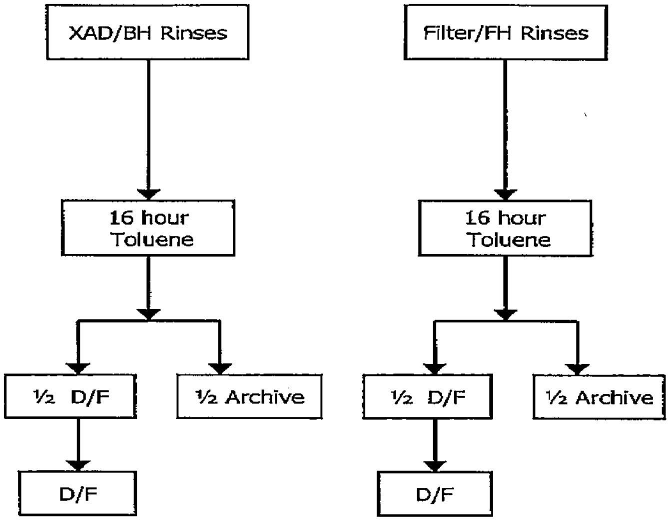
(3) Used for ^{13}C-HxCDF

(4) Used for ^{13}C-HpCDF

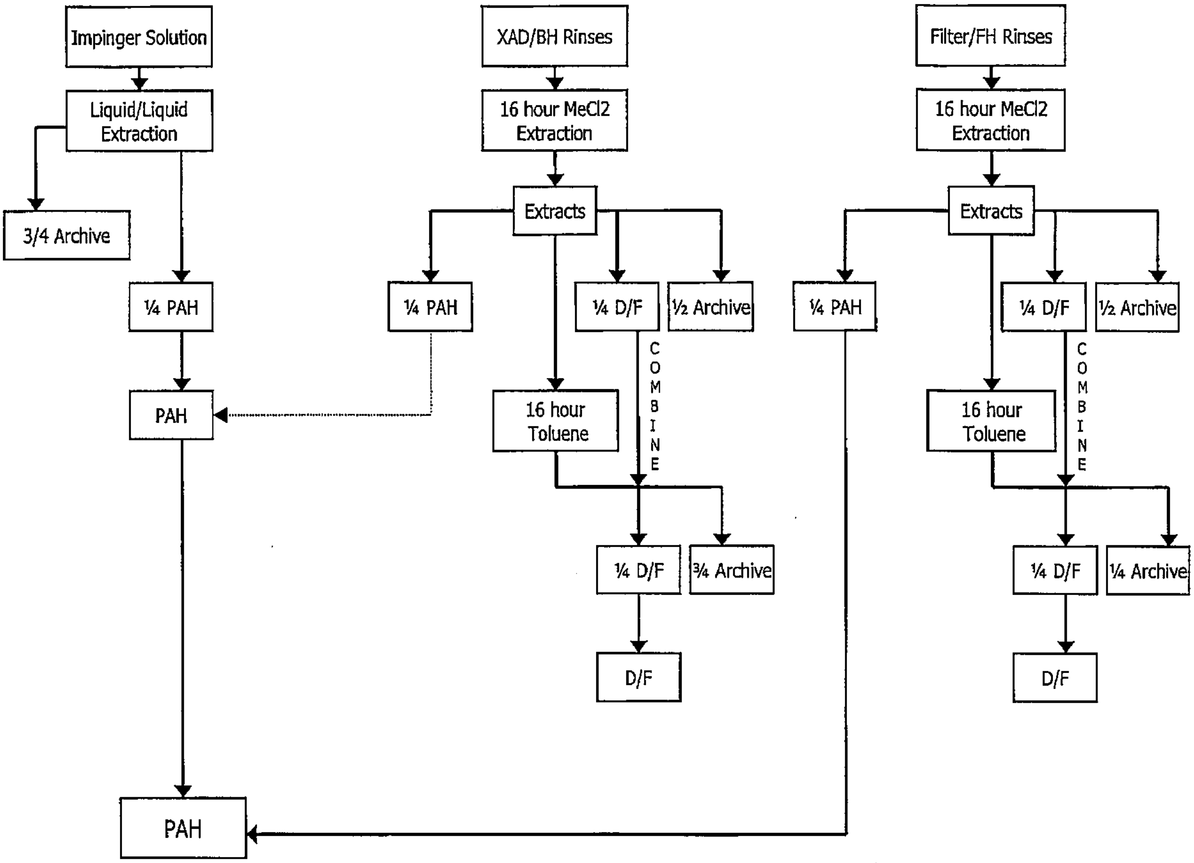
(5) Represents ± 15% windows around the theoretical ion abundance ratios.



Method 0023A, CARB Method 429 and 428
(Dioxin/Furan; PAHs; PCBs)



Method 0023A (Dioxin/Furan)



Method 0023A and CARB Method 429
(Dioxin/Furan; PAHs)

GLOSSARY

Symbols

°C	Celsius
µL	microliter
µm	micrometer
<	less than
>	greater than
%	percent

Alphabetical Abbreviations

cm	centimeter
g	gram
L	liter
M	molecular ion
m	meter
mg	milligram
ml	milliliter
mm	millimeter
m/z	mass-to-charge ration
N	normal
pg	picogram
ppb	part-per-billion
ppm	part-per-million
ppq	part-per-quadrillion
ppt	part-per-trillion
v/v	volume per unit volume
w/v	weight per unit volume

Definitions and Acronyms

Alternate Standard – An alternate standard is a labeled PAH/PCDD/PCDF compound which is added to the impinger contents prior to extraction to estimate the extraction efficiency for PAHs/PCDDs/PCDFs in the impinger sample.

Analyte – a PAH/PCB/PCDD/PCDF tested for by this method. The analytes are listed in Tables 1-3.

Calibration Standard – a solution prepared from a secondary standard and/or stock solutions and used to calibrate the response of the instrument.

Calibration Verification Standard – the mid-point calibration standard (CS-3) that is used to verify calibration.

CB – chlorinated biphenyl congener.

CS-1, CS-2, CS-3, CS-4, CS-5 – See calibration standards and Tables 1-3.

HRMS – High resolution mass spectrometry.

Internal Standard – an internal standard is a labeled PAH/PCB/PCDD/PCDF which is added to all field samples, blanks and other quality control samples before extraction. It is also included in the calibration solutions. Internal standards are used to measure the concentration of the analyte and surrogate compounds.

IPR – initial precision and recovery, four aliquots of a reference matrix spiked with the analytes of interest and labeled compounds and analyzed to establish the ability of the laboratory to generated acceptable precision and recovery.

Isotope Dilution – a means of determining a naturally occurring (native) compound by reference to the same compound in which one or more atoms has been isotopically enriched.

Matrix Spike (MS) – a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte is concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on the methods recovery efficiency.

Method Blank (MB) – an aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, regents, internal standards, and surrogates that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

Minimum Level - The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all Method-specified sample weights, volumes, and cleanup procedures have been employed.

OPR – ongoing precision and recovery standard, a method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

PFK – perfluorokerosene, a mixture of compounds used to calibrate the exact m/z scale in the HRMS.

Practical Quantitation Limit (PQL) – The PQL is a limit for each compound at or below which data must not be reported.

Reagent Water – water demonstrated to be free from the analytes of interest and potentially interfering substances at the method detection limit for the analyte.

Recovery Standard – A recovery standard is a labeled compound, which is added to the extracts of all samples, blanks, and QC samples before analysis. It is also included in the calibration solutions. The response of the internal standards relative to the recovery standard is used to estimate the recovery of the internal standards. The internal standard recovery is an indicator of the overall performance of the analysis.

Relative Response Factor – The relative response factor is the response of the mass spectrometer to a known amount of an analyte or labeled compound relative to a known amount of an internal standard or another labeled compound.

Signal-to-Noise Ratio (S/N) – the height of the signal as measured from the mean (average) of the noise to the peak maximum divided by the width of the noise.

Stock Solution – a solution containing an analyte that is prepared using a reference material traceable to a source that will attest to the purity and authenticity of the reference material.

1.0 VOST SW-846 5041A/8260B

This method involves GC/MS full scan analysis of volatile organic compounds in air samples collected on Tenax/Charcoal (VOST) cartridges. Samples are collected using SW-846 Method 0030/0031 Volatile Organic Sampling Train (VOST) protocols. The VOST cartridges are thermally desorbed by heating and purging with Ultra High Purity Helium. The resulting gaseous effluent is then bubbled through 5 ml of organic free reagent grade water and trapped on the sorbent trap of the purge and trap system. The trap is then thermally desorbed for GC/MS analysis. For condensate analysis, a 5 ml aliquot of condensate sample is placed directly in the sparge vessel of the purge and trap (P&T) system and analyzed in a similar manner.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 1-1. Summary of Method Modifications

Requirement	EPA Method 5041A/8260B	Air Toxics Ltd. Modifications
Method Blank	Cartridges from the same media batches as the samples.	Media batch is certified prior to use in the field. Method Blank is from a different batch unless requested by the client.
Connection between thermal desorption apparatus & purge vessel.	PTFE 1/16” Teflon tubing.	Heated, 1/16” silica lined stainless steel tubing.
Calibration Criteria for non-CCCs.	RSD ≤ 15 % for all non-CCCs.	RSD ≤ 30 % for Acetone, Bromoform, Vinyl Acetate, Bromomethane, Chloromethane, 1,1,2,2-Tetracholoroethane, & 1,2,3-Trichloropropane.

Table 1-2. SW-846 Modified Method 5041A Standard Analyte List

Analytes	RL (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
1,1,1-Trichloroethane	10	15	70 – 130	-
1,1,1,2-Tetrachloroethane	10	15	70 – 130	-
1,1,2,2-Tetrachloroethane – SPCC	10	30	70 – 130	RF > 0.30
1,1,2-Trichloroethane	10	15	70 – 130	-
1,1-Dichloroethane – SPCC	10	15	70 – 130	RF > 0.10
1,1-Dichloroethene – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
1,1-Dichloropropene	10	15	70 – 130	-
1,2,3-Trichlorobenzene	50	15	70 – 130	-
1,2,3-Trichloropropane	10	30	70 – 130	-
1,2,4-Trichlorobenzene	50	15	70 – 130	-
1,2,4-Trimethylbenzene	10	15	70 – 130	-
1,2-Dibromo-3-chloropropane	50	15	70 – 130	-
1,2-Dichlorobenzene	10	15	70 – 130	-
1,2-Dichloroethane	10	15	70 – 130	-
1,2-Dichloropropane – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
1,3,5-Trimethylbenzene	10	15	70 – 130	-
1,3-Butadiene[1]	50	30	50 – 150	-
1,3-Dichlorobenzene	10	15	70 – 130	-
1,3-Dichloropropane	10	15	70 – 130	-
1,4-Dichlorobenzene	10	15	70 – 130	-
2,2-Dichloropropane	10	15	70 – 130	-
2-Butanone[2]	50	30	50 – 150	-
2-Chloropropane	10	15	70 – 130	-
2-Chlorotoluene	10	15	70 – 130	-
2-Hexanone[2]	50	30	50 – 150	-
3-Chloropropene	10	15	70 – 130	-
4-Chlorotoluene	10	15	70 – 130	-
4-Methyl-2-pentanone[2]	50	30	50 – 150	-
Acetone[2]	50	30	50 – 150	-
Acrylonitrile	10	15	70 – 130	-
Benzene	10	15	70 – 130	-
Bromobenzene	10	15	70 – 130	-
Bromochloromethane	10	15	70 – 130	-
Bromodichloromethane	10	15	70 – 130	-
Bromoform – SPCC	10	30	70 – 130	RF > 0.10
Bromomethane[2]	10	30	50 – 150	-
Butylbenzene	10	15	70 – 130	-
Carbon Disulfide	10	15	70 – 130	-
Carbon Tetrachloride	10	15	70 – 130	-

Analytes	**RL (ng)**	**Acceptance Criteria**		
		ICAL (%RSD)	**LCS (% R)**	**CCV**
Chlorobenzene – SPCC	10	15	70 – 130	RF > 0.30
Chloroethane	10	15	50 – 150	-
Chloroform – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
Chloromethane – SPCC	10	30	50 – 150	RF > 0.10
cis-1,2-Dichloroethene	10	15	70 – 130	-
cis-1,3-Dichloropropene	10	15	70 – 130	-
cis-1,4-Dichloro-2-butene	50	15	70 – 130	-
Cumene	10	15	70 – 130	-
Dibromochloromethane	10	15	70 – 130	-
Dibromomethane	10	15	70 – 130	-
Ethylbenzene – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
Ethylene Dibromide	10	15	70 – 130	-
Freon 11	10	15	70 – 130	-
Freon 12	10	15	50 – 150	-
Freon 113	10	15	70 – 130	-
Hexachlorobutadiene	50	15	70 – 130	-
Hexane	10	15	70 – 130	-
Iodomethane	50	15	70 – 130	-
Methylene Chloride	10	15	70 – 130	-
Methyl t-butyl ether (MTBE)	10	30	70 – 130	-
Naphthalene	50	15	70 – 130	-
m,p-Xylene	10	15	70 – 130	-
o-Xylene	10	15	70 – 130	-
p-Cymene	10	15	70 – 130	-
Propylbenzene	10	15	70 – 130	-
sec-Butylbenzene	10	15	70 – 130	-
Styrene	10	15	70 – 130	-
tert-Butylbenzene	10	15	70 – 130	-
Tetrachloroethene	10	15	70 – 130	-
Toluene – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
trans-1,2-Dichloroethene	10	15	70 – 130	-
trans-1,3-Dichloropropene	10	15	70 – 130	-
trans-1,4-Dichloro-2-butene	50	15	70 – 130	-
Trichloroethene	10	15	70 – 130	-
Vinyl Acetate[1,2]	50	30	50 – 150	-
Vinyl Bromide[1] (Bromoethene)	50	30	50 – 150	-
Vinyl Chloride – CCC	10	30	50 – 150	%D ≤ 25% VOST tubes; ≤20% condensates

[1] *Independent source verification check not available for these compounds.*

[2] *Due to nature of these compounds, recoveries outside of noted limits do not result in re-calibration.*

Table 1-3. Matrix Spike/Matrix Spike Duplicate

Analyte	%R
1,1-Dichloroethene	60 – 140
Benzene	60 – 140
Trichloroethene	60 – 140
Toluene	60 – 140
Chlorobenzene	60 - 140

Table 1-4. Internal Standards

Analyte	CCV IS (%R)	Sample IS (%)R
1,4-Dichlorobenzene-d_4	50 – 200	60 – 140
Chlorobenzene-d_5	50 – 200	60 – 140
Fluorobenzene	50 – 200	60 – 140

Table 1-5. Surrogates

Analyte	%R
1,2-Dichloroethane-d_4	70 – 130
4-Bromofluorobenzene	70 – 130
Dibromofluoromethane	70 – 130
Toluene-d_8	70 – 130

Table 1-6. Summary of Calibration and QC Procedures for SW-846 Modified Method 5041A

Note: These criteria are used specifically for the standard list of analytes listed in Table 6-1.2.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at the start of every 12-hour clock.	Method 5041A tuning criteria.	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis.	SPCC criteria in Table 1-2, CCC and non-CCC compound criteria in Table 1-2.	Correct problem then repeat initial calibration.
Laboratory Control Sample (LCS)	Once per initial calibration, and with each analytical batch (maximum of 20 samples).	See Table 1-2.	Investigate the problem and if warranted, analyze a new analytical curve for the out-of-limits compound. (except for compounds noted in Table 1-2.)
Continuing Calibration Verification (CCV)	At the start of every shift immediately after the BFB tune check.	**For SPCCs:** see "CCV criteria" column **For CCCs:** %D ≤ 25% for VOST tubes and ≤ 20% for condensates.	Investigate and correct the problem, up to and including recalibration if necessary.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Internal Standards (IS)	As each standard, blank, and sample is being loaded.	**For CCVs:** area counts 50% - 200%, RT w/in 30 sec of mid-point in ICAL. **For blanks, samples and non-CCV QC Checks:** area counts 60 – 140%, RT w/in 20 sec. of RT in CCV.	**CCV:** inspect and correct system prior to sample analysis. **For blanks:** inspect the system and re-analyze the blank. **For condensates:** re-analyze; if out again, flag data. **For VOST:** flag the data, evaluate system and correct problem before proceeding.
Surrogates	With all samples and QC.	See Table 1-5.	Same as for Internal Standards.
Laboratory Blanks	Immediately after the calibration standard or after samples with high concentrations (≥ 5000 ng).	Results less than laboratory reporting limit	Inspect the system and re-analyze the blank.
(MS/MSD)	Once/batch of condensate samples.	See Table 1-3.	Q-flag and narrate.

Reviewed 2/7/2005
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Reviewed 3/9/2007
Revised by CF2, 3/13/2007

Title: DETERMINATION OF METAL EMISSIONS FROM STATIONARY SOURCE (EPA METHOD 29, EPA SW-846 METHOD 0060, CARB METHOD 436)

[EPA 29, EPA 0060, CARB 436]

Approvals (Signature/Date):

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This SOP was previously identified as SOP No. SAC-IP-0007.

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1. SCOPE AND APPLICATION

1.1. This method is an acid digestion procedure used to prepare air samples for analysis by Inductively Coupled Plasma spectroscopy (ICP), Cold Vapor Atomic Absorption Spectrometry (CVAAS), and Inductively Coupled Plasma - Mass Spectroscopy (ICP-MS). Samples prepared by this method may be analyzed by ICP, ICP-MS or CVAAS for the following metals: aluminum, antimony, arsenic, barium, beryllium, cadmium, total chromium, cobalt, copper, lead, manganese, mercury, nickel, phosphorous, selenium, silver, thallium, vanadium, and zinc. Additional elements may be requested but it should be understood that they are not specified in the method.

2. SUMMARY OF METHOD

2.1. Air samples are collected on heated quartz filter paper, and in a series of impinger solutions. These sampling trains are recovered and digested in separate Front and Back Half Fractions. Acid digestion is performed on the Front Half filter and probe rinse using a solution of both HNO3 and HF that is heated in a microwave. This digestate is made up to predetermined final volume and analyzed by ICP or ICP-MS, and CVAA (Hg). The Back Half contents and rinses are digested using HNO3 and H2O2. A separate aliquot is analyzed by Cold Vapor AAS for mercury determination.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual (LQM).

3.2. Air Train Front Half (FH). This contains the particulate filter, HNO3 probe rinses and, optionally the acetone rinse.

3.3. Air Train Back Half (BH). This contains the impinger contents 1 - 6 and rinsates.

3.4. Test Condition. This consists of three stack sampling runs and a field blank.

4. INTERFERENCES

4.1. Iron (Fe) can be a spectral interference during the analysis of As, Cr, and Cd by ICP. These interferences can be reduced by dilution of the analytical sample, but such dilution raises the in-stack detection limits.

4.2. Aluminum (Al) can be a spectral interference during the analysis of As and Pb by ICP. These interferences can be reduced by dilution of the analytical sample, but such dilution raises the in-stack detection limits.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1. Specific Safety Concerns or Requirements

5.1.1. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Coordinator or hazardous waste specialist for guidance. HF acid or solution may not be used for any purposes other than those described in this or other STL Sacramento SOPs. Contact with HF may not be painful at first. The flouride ion rapidly penetrates skin and may cause delayed effects including lasting ulcerations, bone degeneration, pulmonary edema, muscle paralysis and cardiac arrest.

5.1.1.1. Any suspected exposure to HF liquid or fumes must be immediately evaluated by appropriate medical staff (U.C. Davis Medical Center Emergency Room). Review Appendix 1 for a detailed first aid plan before work with HF begins.

5.1.1.2. Some metals react with HF to release flammable hydrogen gas.

5.1.1.3. Glass reacts with HF to produce toxic silicon tetraflouride.

5.1.1.4. Only associates who have received special training are permitted to use HF.

5.1.1.5. HF is classified as a poison, and must be stored in a locked cabinet when not in use.

5.1.1.6. Anytime HF is to be used in a laboratory, prepare a decontamination solution before starting work. Take a small bucket (2-3 gallons) and mix 1/2 cup of Epsom salts per quart of water in sufficient volume. When an employee has finished working with HF, carefully dip one gloved hand at a time into this bucket for a few seconds. Do not put the hand into the bucket any deeper than to bring the solution level to within 1-2 inches of the glove cuff. The Epsom salts solution will be checked each day prior to beginning work and will be prepared fresh when necessary before HF is used. The old Epsom salt solution will be disposed of down the laboratory sink.

5.1.1.7. The labels on containers for all digestates/extracts with any concentration of HF acid will be marked with a yellow highlighter.

5.1.2. Any HF spill requires activation of the Emergency Response Team.

5.1.3. The acidification of samples that are at a high pH or contain reactive materials may result in a violent reaction or the release of toxic gasses, such as cyanides or sulfides. Acidification of samples must be done in a fume hood.

5.1.4. When digesting samples on a hot plate or digestion block, heat protective gloves and/or hot tongs must be used when handling containers. Ensure that digestion containers are arranged safely and are not overcrowded.

5.1.5. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.1.6. Acid washing of glassware is classified as a high-risk activity. A face shield must be worn over safety glasses or safety goggles during this process.

5.1.7. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Vinyl and nitrile gloves provide satisfactory protection for the acids used in this process. Nitrile gloves must be worn when handling organic solvents.

5.1.8. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred, prepared, digested and cooled in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.9. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrofluoric Acid	Poison Corrosive Dehydrator	3 ppm-TWA	Severely corrosive to the respiratory tract. Corrosive to the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. **THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Volumetric flasks, 100 mL, 500 mL, and 1000 mL. For preparation of standards and sample dilution.

6.2. Graduated cylinders. For preparation of reagents. Note: Graduated cylinders used for measuring HF acid must have graduations that wrap around the entire cylinder, so as to minimize the need to handle the cylinder while pouring. They will also have the plastic collar inverted so that the concave side of the collar faces the top of the cylinder. This will help catch any HF that might drip down the outside of the cylinder.

6.3. Microwave pressure relief vessels (CEM Corporation model or equivalent).

6.4. Beakers and watch glasses. 1000, 250 and 125 mL Teflon beakers for sample digestion with watch glasses to cover the tops.

6.5. Filter funnels. For holding filter paper.

6.6. Whatman 41 filter papers (or equivalent). For filtration of digested samples.

6.7. Disposable Pasteur pipettes.

6.8. Volumetric pipettes.

6.9. Analytical balance. Accurate to within 0.1 mg.

6.10. Microwave oven. For heating samples at fixed power levels or temperatures.

6.11. Hot plates.

6.12. 100 mL and 300 mL snap top sample containers for analytical sample storage.

6.13. 500 mL poly bottles for analytical sample storage,

6.14. 500 mL and 1000 mL glass bottles with Teflon lined caps for analytical sample storage,

6.15. 1 L TriPour beaker,

6.16. Eppendorf pipettes (1-1,000 µL).

7. REAGENTS AND STANDARDS

7.1. Hydrochloric acid (HCl) concentrated.

7.2. Hydrochloric acid (HCl), 8 N. Carefully add, with stirring, 690 mL of concentrated HCl to a graduated cylinder containing 250 mL of deionized water. Dilute to 1000 mL with deionized water. Mix well. The reagent shall contain less than 2 ng/mL of Hg.

7.3. Laboratory double distilled water. All target metals should be less than 1 ng/mL.

7.4. Hydrofluoric acid (HF) concentrated.

7.5. Nitric acid, (HNO_3) concentrated. Baker Intra-analyzed or equivalent.

7.6. HNO_3, 50% (v/v). Carefully, with stirring, add 125 mL of concentrated HNO_3 to 100 mL of deionized water. Dilute to 250 mL with deionized water. Mix well. Reagent shall contain less than 2 ng/mL of each target metal.

7.7. AAS Grade mMetal Standards, multi-mix. One for each target metal.

7.8. Hydrogen peroxide (H_2O_2), 30% (v/v).

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in acid-rinsed glass, polyethylene, or polypropylene containers. Sample collection should be performed in accordance with the appropriate sampling method.

8.2. Samples may be stored at ambient temperature until the time of preparation. Store empty containers until all analyses have been performed.

8.3. Analytical hold times for all metals excluding mercury is 180 calendar days. Mercury analytical hold time is 28 calendar days. (Note- CARB Method 436 has a holding time of 60 days from sampling for all metals except mercury)

9. QUALITY CONTROL

9.1. A batch is defined as an analytical set-up of a given matrix, for a given method, not to exceed twenty samples.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. A preparation blank for the front-half fraction is a control sample that contains 20 mLs of distilled water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. A preparation blank for the back-half fraction is a control sample that contains 100 mLs of distilled water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, copper, iron, lead (trace only) or zinc) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of twenty times higher than the blank contamination level). Corrective actions must be documented and implemented when the method blank is deemed unacceptable. See SOPs for SW846 6010 for ICP and SW846 6020 for ICP-MS for further guidance. See Policy QA-003-SAC for specific acceptance criteria.

9.2.1. Label all prep blanks with the date of preparation, the type of analysis (ICP, ICP-MS, and CVAA), the matrix, and the project numbers associated with it. If a project must be split between more than one preparation blank, record the associated sample numbers on the bottle.

9.3. Duplicate Control Samples (DCS) - One pair of aqueous LCS/LCSD (DCS) must be processed with each preparation batch. The DCS must contain all analytes of interest and must be carried through the entire analytical procedure. The front-half DCS consists of 20 mLs of distilled water, and the back-half DCS each consist of 100 mL distilled water and the reagents used for the digestion, spiked with standards to contain a known concentration of each analyte being requested for a project or a group of projects. The DCS is used to monitor the accuracy of the analytical process. On going monitoring of the DCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. If the DCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the DCS is outside limits, corrective action must be implemented and documented. See SOPs for SW846 6010 for ICP and SW846 6020 for ICP-MS for further guidance. See Policy QA-003-SAC for specific acceptance criteria.

9.4. Mercury QC

9.4.1. All mercury samples will be analyzed in duplicate with the average result reported.

9.4.2. Matrix Spike (MS) - A matrix spike will be performed on the nitric/peroxide impinger fraction of the sampling train at a frequency of one sample per condition for mercury analysis only. The MS results are used to determine the effect of a matrix on the accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS analysis. If the DCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the DCS is outside limits, corrective action must be taken. Corrective action may include reanalysis, or re-preparation and analysis of the sample batch. MS results, which fall outside the control limits, must be addressed in the narrative. See SOPs for SW846 6010 for ICP and SW846 6020 for ICP-MS for further guidance. See Policy QA-003-SAC for specific acceptance criteria.

9.5. ICP or ICP-MS QC

9.5.1. A duplicate analysis will be performed per source test condition. A duplicate analysis of one of the front-half (FH), back-half (BH), or combined FH/BH digestates is performed to monitor the precision of the ICP or ICP-MS analysis.

Note- A Matrix Spike (MS) cannot be performed for the front or back-half fractions (Analytical Fractions 1A or 2A) for ICP or ICP-MS analysis because the entire sample is consumed in the initial digestion. A post-digestion spike may be performed based on project specific requirements.

10. CALIBRATION

10.1. On a monthly basis, calibrate any auto-pipettors to be used in accordance with SOP SAC-QA-004.

10.2. On a daily basis, calibrate any balances to be used in accordance with SOP SAC-QA-0041

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation of Field Samples and Field Blanks

Note the level of the liquid in each of the containers and determine if any sample was lost during shipment. If leakage has occurred, notify the project manager to determine if any corrective action is required. All sample volumes should be measured volumetrically or gravimetrically and recorded for the project record. Prepare each fraction as described in sections 11.4 – 11.8

11.4. Container No. 1 (Filter).

11.4.1. If particulate emissions are being determined, first desiccate the filter and filter catch at room temperature as described in SOP SAC IP-0006. The relative humidity in the desiccator should be 33 ± 5°C.

11.4.2. Weigh to a constant weight as described in Section 10.7 in SOP SAC-IP-0006 (Determination of Particulate Matter in Atmosphere). Following this procedure, or initially, if particulate emissions are not being determined along with metals analysis, a filter division may be needed based on the amount of particulate loading.

11.4.2.1. If the total filter weight exceeds 1.25 grams, or the filter appears to have heavy loading, it may be necessary to divide the filter across several microwave vessels. If the sample filter is divided across several vessels, the final digestate volume may need to be adjusted to maintain an acceptable level of HF in the final digestate. Notify the project manager if method variations are required.

11.4.3. If no division of the filter is required, place the filter into the individual acid-rinsed microwave vessels. The contents of the FH HNO3 rinse, and the re-solubilized particulate from the optional acetone rinse can also be added to the corresponding microwave vessel.

11.4.4. Add 6 mL of concentrated HNO_3 and 4 mL of concentrated HF to each vessel.

WARNING: The use of hydrofluoric acid requires special safety precautions. Review Appendix 1 for a detailed first aid plan before work with HF begins.

Note: Suggested microwave-heating times are approximate and are dependent upon the number of samples being digested. Ten to twelve minutes heating times have been found to be acceptable for simultaneous digestion of up to twelve individual samples. Newer microwave unit may have pre-set methods, based on the number of samples in the batch, that will provide the appropriate amount of power to perform the digestion. These systems monitor the pressure and temperature of the process through probes placed in one of the microwave vessels.

11.4.5. Heat the combined filter and FH rinse samples in the microwave oven for a total of 10 minutes using sufficient power to raise the temperature of each sample to 175 ^{0}C in less than 5.5 minutes and maintain the temperature between 170-180 ^{0}C for the remainder of the 10 minute period. The temperature is monitored by the temperature probe. The digestion program is a preset program (3051-H) in the CEM Mars-5 method library in the instrument.

WARNING: Should a vessel rupture during this process, lab employees are NOT authorized to attempt clean-up of the HF digestate. Turn the oven off and activate the emergency response team.

11.4.6. Allow samples to cool to ambient temperature.

Note: *If the microwave unit does not utilize a temperature monitoring probe it may be necessary to program alternating steps of heating and cooling as follows: heat for 2-3 minutes, turn off power for 2-3 minutes, then heat again for 2-3 minutes. Repeat this process until 12-15 minutes of total heating is completed.*

11.5. Container No. 2 (Acetone Rinse).

11.5.1. For the particulate determination, measure the liquid in the container either volumetrically within 1 mL or gravimetrically within 0.5 g.

11.5.2. Transfer the contents to a preweighed beaker and evaporate to dryness at ambient temperature and pressure.

11.5.3. If particulate emissions are being determined, desiccate for 24 hours at room temperature. The relative humidity in the desiccator should be 33 ± 5°C.

11.5.4. Weigh to a constant weight as described in Section 10.7 in SOP SAC-IP-0006 (Determination of Particulate Matter in Atmosphere) and record the results to the nearest 0.1 mg.

11.5.5. Redissolve the residue with 10 mL of 3M HNO_3.

11.5.6. Quantitatively combine the resultant sample with Container No. 3 and proceed to Section 11.6.

11.6. Container No. 3 (Probe Rinse).

11.6.1. Determine the pH of this sample. If the pH is higher than 2, acidify the sample with concentrated HNO_3 to pH 2 or lower.

11.6.2. Rinse the sample into a beaker with deionized water.

11.6.3. Reduce the sample volume to approximately 20 mL by heating on a hot plate at an approximate temperature of 70 - 75^{o}C. Care should be taken to ensure the sample does not boil or go to dryness.

11.6.4. Transfer the samples to microwave vessels containing the corresponding filter, using small water rinses. Go to section 11.4.3.

11.6.5. Filter the combined solution of the acid digested filter and probe rinse samples using Whatman 41 filter paper.

11.6.6. Dilute to 150 mL with deionized water.

11.6.7. Quantitatively remove a 30 mL aliquot for mercury analysis and label as "Analytical Fraction 1B". The remaining 120 mL portion is for ICP or ICPMS analysis. Label this "Analytical Fraction 1A". Proceed to SOP SAC-MT-0005 for the mercury digestion and analysis, SOP SAC-MT-0001 for ICPMS analysis, or SAC-MT-0003 for ICP analysis.

***Note**: The Analytical Fraction 1B is typically prepared for mercury analysis without dilution (30mls/30mls), using the appropriate volume of reagents for a 30 mls FV.*

11.7. Container No. 4 (HNO_3/H_2O_2 Impingers 1-3).

11.7.1. Measure and record the total volume of this sample "Fraction 2" to within 0.5 mL.

11.7.2. Determine the pH of "Sample Fraction 2". If necessary, acidify using concentrated HNO_3 to pH 2 or lower.

11.7.3. Remove an aliquot of 75-100 mL for mercury analysis and label as “analytical fraction 2B”. If there is less than 500 mLs of container No. 4 received, a smaller sample aliquot for mercury may be required. Label the remaining portion of container 4 as “sample fraction 2A”. Proceed to SOP SAC-MT-0005 for the mercury digestion and analysis of Analytical fraction 2B.

***Note**: A 10x dilution is routinely performed on the Analytical Fraction 2B aliquot (3mls/30mls FV) before mercury preparation and analysis to lower the concentration of* H_2O_2 *present in the solution. The appropriate volume of reagents are used for a 30 ml FV.*

11.7.4. Rinse “Sample Fraction 2A” into an acid-rinsed beaker with deionized water.

11.7.5. Reduce the sample volume to approximately 100 mL by heating on a hot plate at an approximate temperature of 70 - 75^{o}C. Care should be taken to ensure the sample does not boil or go to dryness.

11.7.6. Remove from heat and cool to ambient temperature.

11.7.7. Add 15 mL of conc. HNO_3 and heat for 30 minutes on a hot plate at an approximate temperature of 70 - 75°C. Care should be taken to ensure the sample does not boil or go to dryness.

11.7.8. Add 3 mL of 30% H_2O_2 and heat for 10 more minutes.

11.7.9. Cool, and filter the sample if needed through Whatman 41 filter paper and dilute to 150 mL. This "Analytical Fraction 2A" is used for determining Back-Half metals concentration through ICP/ICP-MS analysis. Proceed to SOP SAC-MT-0001 for ICPMS analysis, or SAC-MT-0003 for ICP analysis of Analytical fraction 2A.

11.8. Container No. 5A (Impinger 4- Optional empty impinger), Container No. 5B (Impingers 5 and 6- KmnO4/H2SO4) and 5C (HCl rinses of Impingers 5 and 6).

These fractions are for Hg analysis only. Keep these samples separate from each other.

11.8.1. Measure and record the total volume of 5A to within 0.5 mL. Label the contents of Container No. 5A as "Analytical Fraction 3A". Remove a 3 ml, or appropriate size aliquot for mercury and proceed to SOP SAC-MT-0005 for the mercury digestion and analysis of Analytical Fraction 3A.

Note: *A 10x dilution is routinely performed on the Analytical Fraction 3A aliquot (3mls/30mls FV) before mercury preparation and analysis to lower the concentration of* H_2O_2 *present in the solution. The appropriate volume of reagents are used for a 30 ml FV.*

11.8.2. If brown MnO_2 precipitate is still present in Container No. 5B after mixing, it should be removed by filtering the contents through Whatman 41 filter paper into a 500 or 1000 mL glass bottle and diluting to a volume of 500 mL with deionized water. Save the filter for digestion of the brown MnO2 precipitate. Label the 500 mL filtrate as "Analytical Fraction 3B". Analyze this fraction within 48 hours of filtration step. Proceed to SOP SAC-MT-0005 for the mercury digestion and analysis.

Note: *The Analytical Fraction 3B is typically prepared for mercury analysis without dilution (30mls/30mls), using the appropriate volume of reagents for a 30 ml FV.*

Note: *Method 0060 does not require the filtration of Container 5B, it only requires the volume of Fraction 5B to be measured, but the sample is not diluted to 500 mls.*

11.8.3. If the sample required filtration, place the filter and contents in a 100 mL snap vial.

11.8.4. Add 25-40 mL of 8N HCl and allow to digest at room temperature for 24 hours.

11.8.5. Filter the contents of Container No. 5C through Whatman 41 filter paper into a 500 mL poly bottle.

11.8.6. Filter the resulting digestate from the digestion of the brown MnO_2 precipitate (Section 11.8.4) into the same poly bottle.

11.8.7. Bring to a volume of 500 mL with deionized water and label as "Analytical Fraction 3C". Fractions 3A, 3B and 3C will be analyzed for Hg only. Proceed to SOP SAC-MT-0005 for the mercury digestion and analysis.

Note- The Analytical Fraction 3C is typically prepared for mercury analysis without dilution (30mls/30mls), using the appropriate volume of reagents for a 30 mls FV.

11.9. Field Reagent Blanks

11.9.1. Container No. 7 (Acetone Blank)

If particulate emissions are to be determined, a 100 mL portion of the acetone used for sample recovery should be in a container labeled as Container No.7 for shipment to the lab.

11.9.2. Container No. 8A (0.1N HNO_3 Blank)

A 300 mL aliquot of the 0.1N HNO3 solution used for the sample recovery process should be placed in a container and labeled as Container 8A.

11.9.3. Container No. 8B (Water Blank)

A 100 mL aliquot of the water used in the sample recovery process should be in a container labeled Container No. 8B

11.9.4. Container No. 9 (5 % HNO_3/10% H_2O_2 Blank)

A 200 mL aliquot of the 5 % HNO3/10% H2O2 solution used in the sample recovery process should be in a container labeled Container No. 9

11.9.5. Container No. 10 (Acidified $KMnO_4$ Blank)

A 100 mL aliquot of the Acidified KMnO4 used in the sample recovery process should be in a container labeled Container No. 10

11.9.6. Container No. 11 (8N HCl Blank)

A 200 mL aliquot of the reagent water and a 25 ml aliquot of the 8N HCl used in the sample recovery process should be combined in a container and labeled as Container No. 11

11.9.7. Container No. 12 (Filter Blank)

Three unused filters from the same lot as the sampling filters should be in a container labeled Container No. 12

11.10. Sample Preparation for Reagent Blanks

11.10.1. Reagent Blanks for Analytical Fractions 1A and 1B

11.10.1.1. Add 100 mLs of Container No. 8A to the beaker containing the desiccated residue of the Container No. 7 to resolubilize the residue.

11.10.1.2. Reduce the volume of the combined residue of Container No. 7 and the 100 mLs of Container 8A according to Section 11.6.3.

11.10.1.3. Transfer the sample to a microwave vessel and add one of the filters from Container No. 12. Proceed with the HF/Microwave digestion of the reagent sample according to section 11.4.4. This will produce the reagent blank fraction for analytical fractions 1A and 1B.

11.10.2. Reagent Blanks for Analytical Fractions 2A and 2B

A 100 mL aliquot of Container No. 8A is combined with a 200 mL aliquot of Container No. 9 to produce the Reagent Blanks for analytical fractions 2A and 2B. Proceed with sample preparation according to Section 11.7.

11.10.3. Reagent Blank for Analytical Fraction 3A

A 100 mL aliquot of Container No. 8A will be digested to produce the Reagent Blank for Analytical Fraction 3A. Proceed with sample preparation according to Section 11.8.1.

11.10.4. Reagent Blank for Analytical Fraction 3B

A 33 mL aliquot of Container No. 8B is combined with 100mls of Container No. 10 to produce the Reagent Blank for Analytical Fraction 3B. Filter the combined solution according to Section 11.8.2, but do not dilute to 500 mL. Analyze the Reagent Blank within 48 hrs of filtration, and use 400 mL as the blank volume when calculating the blank mass value. Proceed to SOP SAC-MT-0005 for the mercury digestion and analysis.

11.10.5. Reagent Blank for Analytical Fraction 3C

Digest the filter that was used to remove any brown MnO4 precipitate, if it was present, from the Reagent Blank for Analytical Fraction 3B according to Section 11.8.4.

***Note:** Method 0060 does not require the filtration of the $KMnO_4$ solution to remove the MnO_2 precipitate, so there will be no filter fraction to combine with the contents of Container No. 11.*

Filter the digestate from Section 11.10.1.1 and the contents of Container No. 11 according to Section 11.8.5 to produce the Reagent Blank for Analytical Fraction 3C. Proceed to SOP SAC-MT-0005 for the mercury digestion and analysis.

11.11. Analysis of the Front Half, Back Half, or combined Front Half/Back Half digestates for ICP, or ICP-MS analysis (See SOPs SAC-MT-0003 for 6010/ICP and SAC-MT-0003 6020/ICP-MS. Refer to Section 17)

11.11.1. Analytical Fractions 1A (Front Half) and Analytical Fractions 2A (Back Half) may be analyzed separately, or by client request, may be proportionally combined to a final volume for analyses by ICP or ICP-MS. Results calculated on μg/sample.

11.12. Mercury analysis: Label each fraction appropriately and analyze by Method SW846 7470 (Refer to SOP SAC-MT-0005, see Section 17).

- Analytical Fraction 1B (Front Half)
- Analytical Fraction 2B (Back Half)
- Analytical Fraction 3A (Condensates)
- Analytical Fraction 3B ($KMnO_4$)
- Analytical Fraction 3C (HCl)

12. CALCULATIONS

12.1. The analysis of metals by ICP or ICP-MS, for separate FH and BH fractions, is based on a final digestate volume of 150 mL. Use Equations 1 and 2 to determine the total mass of metals in each fraction. If Analytical fractions 1A & 2A are to be proportionally combined for a single analytical fraction, use Equations 3, 4, and 5 to calculate the results.

Note- Check instrument data system to confirm the units used for reporting (ug/L, ug/ml, etc) so the proper unit conversions can be applied.

12.2. Calculation for the FH metals (except Hg), based on a predetermined final volume:

Equation #1: $$\mathbf{M_{FH} = C_{1A} \times (DF) \times (V_{1A})}$$

Where:

M_{FH} = Total mass of each metal except Hg collected in the front half of the sampling train (Sample Fraction 1), µg

C_{1A} = Concentration of metal in Analytical Fraction 1A as read from the standard curve, µg/L

DF = The inverse of the fractional portion of the concentrated sample in the solution (Dilution Factor)

V_{1A}= Total volume of Analytical Fraction 1 digestate, L

12.3. Calculation for the BH metals (except Hg), based on a predetermined final volume minus the fraction taken out for the Mercury analysis is as follows:

Equation #2: $$\mathbf{M_{BH} = C_{2A} \times (AF) \times (V_{2A})}$$

Where:

M_{BH} = Total mass of each metal except Hg collected in the back half of the sampling train, µg

C_{2A} = Concentration of metal in Analytical Fraction 2A as read from the standard curve, µg/L

AF = Volume of Sample Fraction 2 ÷Volume of Sample Fraction 2A, L

V_{2A} = Total volume of digested sample solution (Analytical Fraction 2A), L

12.4. Calculated volume used for reporting FH/BH combined samples

Equation #3: $$\mathbf{V_{CAL} = [BH_{I} \div (BH_{I} - BH_{R\,Hg}) \times BH_{FD}] + FH_{FD}}$$

Where:

V_{CAL} = The calculated volume used to report samples in µg/sample, based on combined FH/BH samples, ml

BH_I = Initial back half volume, mL

$BH_{R\,Hg}$ = Back half volume taken out for Hg analysis, mL

BH_{FD} = Back half volume of the final digestate, mL

FH_{FD} = Front half volume of the final digestate, mL

12.5. Volume of FH digestate to be used for combined FH/BH analysis.

Equation #**4:** $$\mathbf{V_{FHD} = FH_{FD} \div \text{Result of Eq. \#3} \times \text{100 mL combined FV}}$$

Where:

V_{FHD} = Amount of volume to remove from Front Half Digestates to be combined with Back Half digestates, ml
FH_{FD} = Front half volume of the final digestate, mL

12.6. Volume of BH digestate to be used for combined FH/BH analysis.

Equation #5: $\mathbf{V_{BHD} = BH_{I} \div (BH_{I} - BH_{R\,Hg}) \times BH_{FD} \div}$ **Result of Eq. #3 × 100 mL FV**

Where:
V_{BHD} = Amount of volume to remove from the Back Half digestate to be combined with the Front Half digestates
BH_{I} = Initial back half volume, mL
$BH_{R\,Hg}$ = Back half volume taken out for Hg analysis, mL
BH_{FD} = Back half volume of the final digestate, mL

Note: Calculations from Sections 12.3 -12.5 are reflected on the Air Toxic Metals Prep Combined FH/BH Air Train Spreadsheet.

12.7. Mercury Calculations (based on the following volumes):

12.7.1. Mercury result for Analytical Fraction 1B (Front Half), based on 150 mL (0.150 L) (Analytical Fraction 1).

Equation #6: $\mathbf{M_{Hg1B} = C_{1B} \times VF_{1B} \text{ x } DF}$

Where:

M_{Hg1B} =Total mass of Hg collected in the front half, µg

$C1_{B}$ = Concentration of Hg in Fraction 1B as read from the standard curve, ug/L

VF_{1B} = Digestate Volume of Fraction 1, L

DF = Dilution Factor

12.7.2. Analytical Fraction 2B (Back Half) calculation based on volume received (Sample Fraction 2).

Equation #7: $\mathbf{M_{Hg2B} = C_{2B} \times VF2 \text{ x } DF}$

Where:

M_{Hg2B}=Total mass of Hg collected in the front half fraction, µg

C_{2B} = Concentration of Hg in Fraction 2B as read from the analytical curve (ug/L)

VF2= Volume of Fraction 2 received (L)

DF = Dilution Factor

12.7.3. Analytical Fraction 3A (Condensates) calculation based on volume received (Analytical Fraction 3A).

Equation #8: $\mathbf{M_{Hg3A} = C_{3A} \times VF_{3A} \text{ x } DF}$

Where:

M_{Hg3A}= Total mass of Hg in Fraction 3A as read from the analytical curve, µg

C_{3A} = Concentration of Hg in Fraction 3A as read from the analytical curve, ug/L

VF_{3A}= Volume of Analytical Fraction 3A (cond) received, L

DF = Dilution Factor

12.7.4. Analytical Fraction 3B ($KMnO_4$) based on 500 mL (Analytical Fraction 3B).

Equation #9: $\mathbf{M_{Hg3B} = C_{3B} \times VF_{3B} \text{ x } DF}$

Where:

M_{Hg3B}= Total mass of Hg collected in the $KmnO_4$, µg

C_{3B} = Concentration of Hg in Fraction 3B as read from the analytical curve, ug/L

VF_{3B}= Final volume of Analytical Fraction 3B ($KmnO_4$), L (0.5 L)

DF = Dilution Factor

Note- Method 0060 does not require the volume of Anal $Fract_{3B}$ to be adjusted to 500mls, so the volume used for VF_{3B} will be the volume received.

12.7.5. Analytical Fraction 3C (HCl) based on 500 mL (Analytical Fraction 3C).

Equation #10: $\mathbf{M_{Hg3C} = C_{3C} \times VF_{3C} \text{ x } DF}$

Where:

M_{Hg3C}= Total mass of Hg collected in the HCl rinse, µg

C_{3C} = Concentration of Hg in Fraction 3C as read from the analytical curve, ug/L

VF_{3C}= Final volume of Analytical Fraction 3C (HCL), L (0.5 L)

DF = Dilution Factor

Note: *Calculations from Sections 11.7 are reflected on the Air Toxic Mercury Spreadsheet.*

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. All methods performed at STL Sacramento must have an annual MDL study, a finalized SOP, and four acceptable LCS samples from each analyst performing the method.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15. WASTE MANAGEMENT

15.1. Contaminated disposable materials such as plastic vials, pipettes, and filters used during sample preparation and digestion. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES

16.1. Emissions Test Method 29. "Determination of Metals Emissions from Stationary Sources" (1994 Proposal)

16.2. Method 436, “Determination of Multiple Metals Emissions from Stationary Sources”, California Environmental Protection Agency, Air Resources Board (July 28, 1997)

16.3. Method 0060, “Determination of Metals in Stack Emissions”, U.S. Environmental Protection Agency, SW-846 Update III, 12/96

17. MISCELLANEOUS

17.1. Deviations from reference method.

17.1.1. The filter and probe rinse fractions are digested together in the same microwave vessel, using SW-846 Method 3051.

17.1.2. The Back-half (Fraction 2A) is reduced to 75-100 mls instead of the 20 mls referenced in Method 29 and 0060, prior to digestion. This is to reduce loss of more volatile metals.

17.1.3. The Back-half fraction is digested using 15 mls on conc HNO3 and 3 mls of 30% H2O2, instead of 30 mls of 50% HNO3 and 10 mls of 3% H2O2. The lab does not use the 50 mls of hot water in the digestion procedure.

17.1.4. Formulas for the calculation of proportionally combined Front Half and Back Half digestates are not referenced in the EPA Method 29.

17.1.5. The digestate and aliquot volumes from each Fraction listed in Section 11 have may be different from the final volumes listed in EPA method 29, EPA SW-846 Method 0060, or ARB Method 436.

17.1.6. Laboratory double distilled water is used rather than ASTM Type II.

17.2. List of other SOPs cross-referenced in SOP.

17.2.1. SAC-QA-0041 Calibration and Calibration Check of Balances.

17.2.2. QA-008-SAC Data Recording Requirements.

17.2.3. QA-003-SAC Quality Control Program.

17.2.4. SAC-MT-0003 Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis,
Method 6010A and Method 200.7.

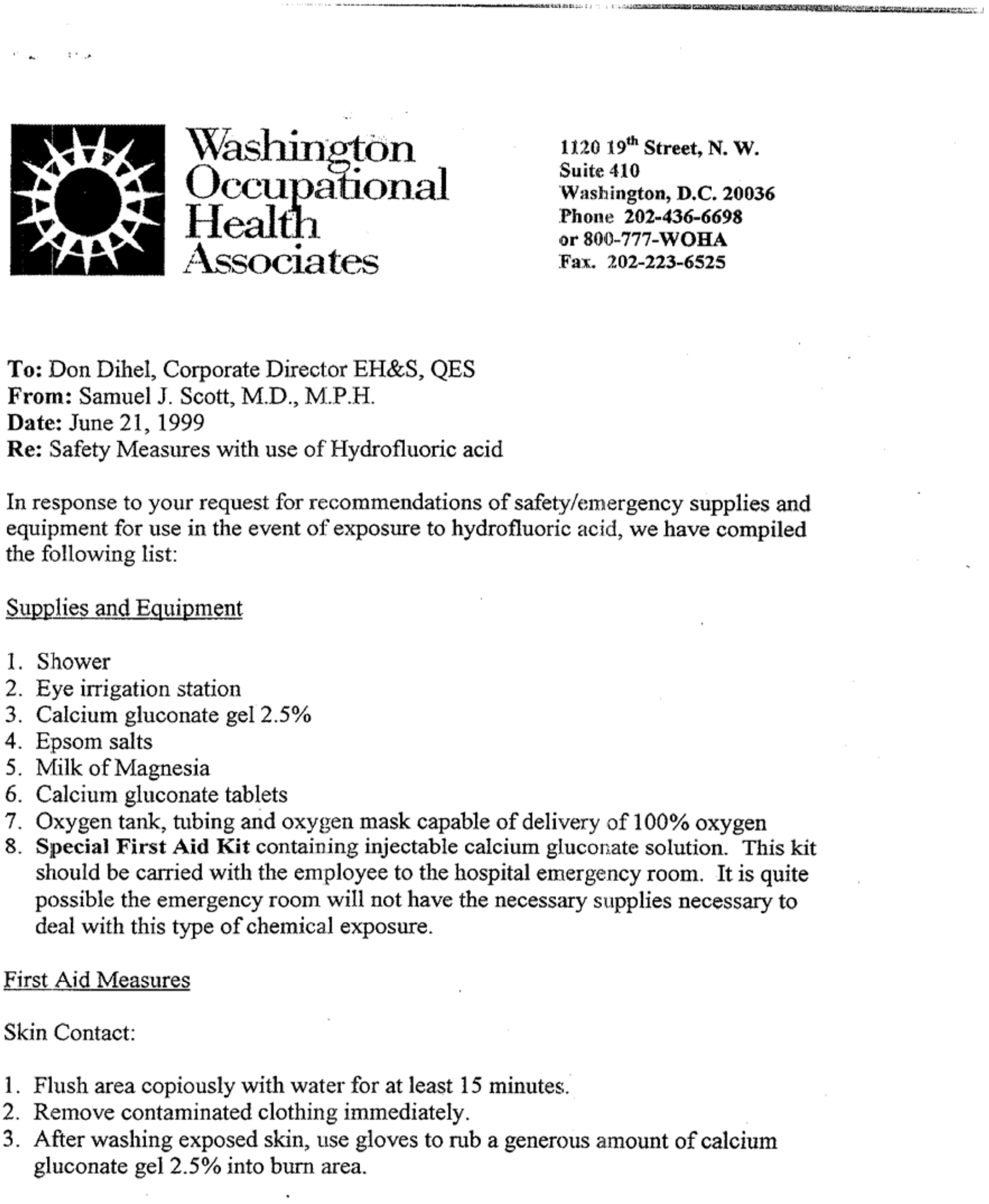
17.2.5. CORP-MT-0005 Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A and MCAWW 245.1.

17.2.6. SAC-MT-0001 Analysis of Metals by Inductively Coupled Plasma -Mass Spectrometry (ICP-MS)

17.3. Method appendices.

17.3.1. Appendix 1. Hydrofluoric acid Safety Guidelines.

Appendix 1 Hydrofluoric Acid Safety Guidelines



Washington Occupational Health Associates

1120 19th Street, N. W.
Suite 410
Washington, D.C. 20036
Phone 202-436-6698
or 800-777-WOHA
Fax. 202-223-6525

To: Don Dihel, Corporate Director EH&S, QES
From: Samuel J. Scott, M.D., M.P.H.
Date: June 21, 1999
Re: Safety Measures with use of Hydrofluoric acid

In response to your request for recommendations of safety/emergency supplies and equipment for use in the event of exposure to hydrofluoric acid, we have compiled the following list:

Supplies and Equipment

1. Shower
2. Eye irrigation station
3. Calcium gluconate gel 2.5%
4. Epsom salts
5. Milk of Magnesia
6. Calcium gluconate tablets
7. Oxygen tank, tubing and oxygen mask capable of delivery of 100% oxygen
8. **Special First Aid Kit** containing injectable calcium gluconate solution. This kit should be carried with the employee to the hospital emergency room. It is quite possible the emergency room will not have the necessary supplies necessary to deal with this type of chemical exposure.

First Aid Measures

Skin Contact:

1. Flush area copiously with water for at least 15 minutes.
2. Remove contaminated clothing immediately.
3. After washing exposed skin, use gloves to rub a generous amount of calcium gluconate gel 2.5% into burn area.

4. For areas too large to apply the gel, use an Epsom salts solution in a concentration of ½ to 1 cup of Epsom salts in one quart of iced water. Immerse the limb into a bucket of solution or soak the solution into gauze and apply to the wound. This dressing should be replaced or re-soaked every two minutes.
5. If area affected is greater than 2 inches by 2 inches, give 6 tablets of calcium gluconate orally.
6. If area affected is greater than 4 inches by 4 inches, assume significant inhalation injury and treat accordingly.
7. Except for small burns, refer to the emergency room and continue applying calcium gluconate gel or Epsom salt soaks en route.

Caution: Hydrofluoric acid causes delayed burns over several hours so the immediate care is essential to prevent further harm.

Inhalation:

1. Administer oxygen 100%.
2. Resuscitate as necessary.
3. Transport to the nearest emergency room.

Eye Contact:

1. Immediately flush the eyes at the irrigation station.
2. Get medical assistance immediately.

Ingestion:

1. Do NOT induce vomiting.
2. Have the victim drink two large glasses of water or milk.
3. Give two ounces of milk of magnesia.
4. Give six tablets of calcium gluconate.

I hope this provides the information you requested. Should you have further questions or require additional information, please do not hesitate to contact one of the physicians at WOHA.

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Title: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption
[EPA 7470A, EPA 245.1]

Approvals (Signature/Date):

	1/25/08	Richard Kester for Joe Schairer	1-21-08
Barry Votaw Technical Manager	Date	Joe Schairer Health & Safety Manager / Coordinator	Date
Pamela Schemmer	1/23/08	Karla Buechler	1/25/08
Pamela Schemmer Quality Assurance Manager	Date	Karla Buechler Laboratory Director	Date

This SOP was previously identified as SOP No. SAC-MT-0005.

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1. Method 7470A is applicable to the preparation and analysis of mercury in ground water, wastewater, wastes, wipes, TCLP, SPLP, and STLC leachates. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline water, domestic and industrial wastes. All matrices require sample preparation prior to analysis

2. SUMMARY OF METHOD

2.1. Samples are digested using a combination of acids and strong oxidizers, converting all forms of mercury to elemental mercury. The samples are treated with hydroxylamine hydrochloride solution to remove excess oxidizing reagents. The digestates are analyzed using Leeman Labs automated mercury analyzers. The mercuric ions are reduced to atomic mercury vapor with the addition of stannous chloride into a gas-liquid separator. The mercury vapor is purged into an absorption cell with nitrogen gas. The relative absorbance of the mercury vapor is detected at a wavelenth of 253.7- nm via a lamp/detector system

2.2. Reporting limit: 0.2 µg/L

2.3. Calibration Range: 0.2 µg/L to 10 µg/L

2.4. This analytical method is restricted to use by, or under the supervision of an analyst experience in the operation of cold vapor analysis and the evaluation of the resulting data.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. Potassium permanganate, which is used to breakdown organic mercury compounds also eliminates possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. High levels of free chlorine can cause a positive interference. Seawaters, brines and industrial effluents high in chlorides require additional permanganate (as much as 5.0 mL) since chloride is converted to free chlorine during oxidation. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide satisfactory protection.

5.2.2. Latex and vinyl gloves provide adequate protection against the chemicals and reagents typically used during this process. However, if any organic solvents are used, or any sample matrix contains organic solvents, only nitrile gloves should be used.

5.2.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.2.4. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2.5. Mercury is a highly toxic element that must be handled with care. Mercury vapor is toxic, so all work must be done in a functioning fume hood and vapors must be vented into a hood. If any volume of mercury reagent or liquid mercury is spilled, the Emergency Response Team must be activated, code yellow, for spill clean-up.

5.2.6. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2.7. Do not look directly into the beam of the mercury lamp. The UV light from the lamp is harmful to the eyes.

5.3. PRIMARY MATERIALS USED

5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hydroxylamine Hydrochloride	Corrosive Poison	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Corrosive to the eyes. Irritant and possible sensitizer. May cause burns to the skin.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (1)	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. An autoclave that is able to obtain conditions of 15 psi, at 120 °C for 15 minutes. The temperature of the autoclave is to be verified quarterly. Checks can be performed more frequently if the performance of the autoclave is in question.

6.2. Leeman Labs PS 200II and Leeman Labs Hydra AA automated mercury analyzers.

6.2.1. Leeman autosampler.

6.2.2. Liquid-gas separator.

6.2.3. Peristaltic pump.

6.2.4. Dehydration line: prevents condensation in the cold vapor absorption cell.

6.2.5. Cold vapor absorption cell.

6.2.6. Mercury hollow cathode lamp.

6.3. Computer with a printer.

6.4. Top-loading balance capable of accurately weighing 0.01 g.

6.5. 14 mL polystyrene test tubes for the autosampler.

6.6. 50 mL centrifuge tubes.

6.7. Nitrogen gas supply.

6.8. Pump windings – dimensions per instrument manufacturer requirements.

6.9. Bottle-top dispenser: re-pipetters.

6.10. Volumetric adjustable air displacement pipets.

6.11. Class A volumetric flasks.

6.12. pH indicator strips (pH range 0-14).

7. REAGENTS AND STANDARDS

7.1. Reagent water is produced by a Millipore nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Nitric acid (HNO_3), concentrated, analytical reagent grade.

7.3. Sulfuric acid (H_2SO_4), concentrated, analytical reagent grade.

7.4. Potassium permanganate, 5% solution (w/v): Dissolve 5.0 g of potassium permanganate for each 100 mL of reagent water.

7.5. Potassium persulfate, 5% solution (w/v): Dissolve 5.0 g of potassium persulfate for each 100 mL of reagent water.

7.6. Stannous chloride solution: Add 25 g of stannous chloride and 15 mL concentrated HCl to 250 mL of reagent water. This mixture is a suspension and should appear cloudy. This solution must be made every 12 hours.

7.7. Sodium chloride-hydroxylamine hydrochloride solution: Add 12.0 g of sodium chloride and 12.0 g of hydroxylamine hydrochloride for each 100 mL of reagent water.

7.8. All standards must be stored in polyethylene or polypropylene bottles.

7.9. 10 ppm mercury standards: Two separate stock standards purchased from separate manufacturers or different lots from the same manufacturer. One standard is used to make the ICV standard and the other for instrument calibration, CCVs, LCSs, and MS/SDs. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year from opening, and must be replaced sooner if verification from an independent source indicates a problem.

Note: Upon receipt of any stock standard, the “Certificate of Analysis” should be immediately filed in the proper location labeled with the receipt date. The receipt date and date the container is opened must be labeled on the container.

7.10. 0.1 ppm working mercury standards (for each stock): add 0.5 mL of stock 10 ppm source standard and 1 mL HNO_3 to a 50 mL volumetric flask and dilute to volume with reagent water. These standards must be made daily.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.2. Aqueous samples must be preserved with nitric acid to a pH of <2 at the time of sampling and may be stored in either plastic or glass. Refrigeration is not required. Preservation **must** be verified prior to analysis

9. QUALITY CONTROL

9.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be analyzed within the same time frame. A method blank (MB), laboratory control sample (LCS) or duplicate control sample (DCS) are distilled and analyzed as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD), or in some instances a sample/sample duplicate. An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples (see policy QA-003-SAC (Quality Control Program) for more details).

9.2. One method blank must be processed and reported for every 20 samples. If a method blank exceeds +/- the reporting limit, then the samples must be re-extracted. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo (see policy QA-003-SAC (Quality Control Program) for further details).

9.3. A laboratory control sample (LCS) must be processed and reported for every 20 samples distilled and analyzed. A LCS consists of reagent water spiked with the analyte of interest and processed through all of the steps, and at the same time as the associated samples. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-extracted. One exception is when a LCS exhibits high recovery, those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo (see policy QA-003-SAC (Quality Control Program) for further details).

9.4. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. MS/MSD pairs are aliquots of a selected field sample spiked with a known concentration of the analyte of interest. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the blank, LCS, selected field samples, and/or the MS/MSD may be required after evaluation and review.

9.4.1. Samples identified as field blanks, equipment blanks, or trip blanks should not be used for sample/sample duplicate nor MS/MSD analysis.

9.4.2. A duplicate control sample (DCS or LCS/LCSD) must be substituted when insufficient volume is provided to process a sample/sample duplicate or MS/MSD pair. The LCS and LCSD are evaluated independently for acceptance (see policy QA-003-SAC (Quality Control Program) for further details.

9.5. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard immediately upon completion of instrument calibration. This standard must be at a concentration different from that used to calibrate the instrument and different from the CCV standard. The ICV must fall within +/- 10% of the true value of the standard solution. An ICB prepared the same as the calibration blank must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit from zero. If either the ICV or ICB fail to meet acceptance criteria the analysis must be terminated, the problem corrected, and the instrument re-calibrated.

9.6. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. Ten samples include any analysis that registers with a result, even if not used. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument re-calibrated, the calibration verified, and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action includes re-preparation of the associated samples.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument calibration must be performed daily (once every 24 hours) or each time the instrument is off or non-operational for more than two hours.

10.2. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.3. Calibration standards must be processed through the preparation procedure as described in section 11.9. Calibration standards must be prepared daily.

10.4. Calibrate the instrument according to instrument manufacturer’s instructions, using a minimum of five standards and a calibration blank. One standard must be at the reporting limit. Analyze standards in ascending order beginning with the blank

10.4.1. Calibration standard concentrations and the volume of 0.1 ppm working standard required to make 30 mL of standard:

0.1 ppm Hg Volume (mL)	0.06	0.15	0.3	1.5	3.0
Final Concentration (µg/L)	0.2	0.5	1.0	5.0	10.0

10.4.2. 30 g of 2% HNO_3 is added to each digestion vessel. An adjustable volumetric pipette is used to remove a volume of 2% HNO_3 corresponding to the spiking volume for that standard. The standard is then spiked with the appropriate volume of 0.1 ppm Hg standard.

10.5. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and re-calibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

10.6. The concentration of the ICV (section 9.5) is 2.0 ppb made by diluting 0.6 mL of the 0.1 ppm alternate source intermediate to 30 mL total volume with 2% HNO_3. See section 10.4.2 for the spiking procedure.

10.7. The 5.0 ppb CCV is the same as the calibration standard.

10.8. The autoclave is monitored daily to assure samples are digested for the proper time interval at the appropriate temperature. An “ERTCO” temperature logging device is put in the autoclave and undergoes the same program as the samples. This device can be connected to a computer and a program generates a graph of time vs temperature. These are submitted with the raw data.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a nonconformance memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The nonconformance memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. All preparation procedures must be carried out in a properly functioning hood.

11.4. All samples are to be checked out and back into sample control with the chain of custody documentation filled out completely. Samples are to be returned to the sample control area once all of the digestions have been initiated.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be accurate and legible. Always set the samples up on the sample cart in the order with which they are to be dispensed. Double-check the sample bottle IDs, once prior to pouring them into the digestion vessels and again before they are returned to the sample cart.

11.6. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludge and tissues (animal/plant) are usually logged in with solid test codes. When initiating preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge-like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.7. **Always** read the QAS for every project prior to establishing batch associations. This is meant to assure that all client requirements are satisfied and it also aids in creating more efficient batches. Setting up batches to minimize QC and meet all client requirements is a skill that must be developed by all new personnel with the aid of skilled sample preparation and analytical staff.

11.8. The following procedure must be followed for all aqueous sample preparations:

11.8.1. Use a small aliquot of each sample and measure the pH with pH indicator strips. If the bottles have an “X” on the tops, they have already been tested for other tests and do not require this step.

11.8.1.1. For samples with pH<2 put an “X” on top of the bottle indicating the samples were properly preserved. For these samples, record pH<2 on the preparation log.

11.8.1.2. For samples that test pH>2, notify the project manager immediately so they can contact the client. If the client requests the sample be preserved by the laboratory, add HNO_3 in 1.0 mL aliquots until the sample remains at pH<2 for at least 10 minutes. Let the sample stand for 24 hours and re-test the pH prior to preparation. As long as the pH<2, the sample can then be digested. A non-conformance memo must be created with an accounting of the anomalous event and the corrective action. A “Sample Preservation Log” must also be completed and filed with the raw data. Record “AF” for “Anomaly Filed” on the digestion log. Put an “X” on top of the bottle indicating the samples were properly preserved.

11.9. Sample Preparation:

11.9.1. All calibration and QC standards must be prepared in the same manner as the samples and be processed through all steps of the preparation procedure.

11.9.2. Transfer 30 mL of **well-mixed sample** to a 50 mL graduated centrifuge tube. The MS/SD sample should be shaken between pouring the separate aliquots.

11.9.3. TCLP extracts are diluted 5X and STLC extracts diluted 10X prior to digestion. LCS and MS/SD spiking levels are 1.0 ppb times the dilution factor.

11.9.4. For each method blank and LCS required, weigh out 30 g of water. For the LCS, remove 0.3 mL with an adjustable volumetric pipette. Add 0.3 mL of the 0.1 ppm Hg intermediate used to make the calibration standards. The LCS mercury concentration is 1.0 ppb.

11.9.5. For each MS/SD, spike the samples directly with 0.3 mL of the 0.1 ppm Hg intermediate used to make the calibration standards. The MS/SD mercury concentration is 1.0 ppb.

11.9.6. Add 2.0 mL of concentrated H_2SO_4 and 1.0 mL of concentrated HNO_3, to all samples and standards, mixing after each addition.

11.9.7. Add 5.0 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 5 mL additional permanganate the color does not persist, sample dilution may be required.

Note: When reporting analyses, the addition of excess reagent must be addressed through mathematical correction of the results to account for the resultant dilution effect.

11.9.8. Add 2.5 mL of potassium persulfate solution.

11.9.9. Autoclave for 25 minutes at 120 °C and 15 lbs (to maintain temperature for 15 minutes).

11.9.9.1. Allow the autoclave to cool to less than 80°C where there is no psi of pressure remaining.

Warning: ***Caution must be used when opening the door of the autoclave as superheated steam may be present and can cause significant burns.***

11.9.10. Allow all of the samples to cool to room temperature.

11.10. Sample Analysis:

11.10.1. When ready to begin analysis, add 2mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Mercury analysis is run using a Leeman PS200II or Hydra AA automated mercury analyzer. Results are reported as ug/L Hg.

11.10.1.1. Make certain the lamp is on and the pump windings are working properly. Replace any pump windings that do not exhibit a smooth flow of liquid or look stretched or flattened.

Warning: ***Do not look directly into the beam of the mercury lamp. The UV light from the lamp is harmful to the eyes.***

11.10.1.2. Load the samples into the automated sampler racks according to the sample lists entered into the computer. Do a final check once all samples have been loaded on to the racks to assure proper sequencing.

11.10.1.3. Stannous chloride is automatically added to the samples by the instrument via the peristaltic pump and mixed with the sample in a liquid-gas separator. This must be freshly prepared every 12 hours.

11.10.2. Dilute and reanalyze all samples that exceed the 10 ppb linear range of the calibration.

11.10.3. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.10.4. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.10.5. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.10.6. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

11.10.6.1. recovery of the analyte in the matrix spike is less than 50%;

11.10.6.2. the concentration of the analyte does not exceed the regulatory level;

11.10.6.3. the concentration of the analyte is within 20% of the regulatory level.

11.11. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data is reviewed periodically throughout the run.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV/CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found_{(ICV\ /\ CCV)}}{True_{(ICV\ /\ CCV)}}\right)$$

12.2. Matrix spike recoveries are calculated according to the following equation:

$$\%\ R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.3. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{\left|MSD - MS\right|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{\left|DU1 - DU2\right|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.4. The final concentration for an aqueous sample is calculated as follows:

$$\mu g/L = C \times D$$

Where:

C = Concentration (*µg/L*) from instrument readout
D = Instrument dilution factor

12.5. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found_{(LCS)}}{True_{(LCS)}}\right)$$

12.6. Appropriate factors must be applied to sample values if dilutions are performed.

12.7. Sample results should be reported with up to three significant figures in accordance with the STL Sacramento significant figure policy (see QA-004-SAC, Rounding and Significant Figures).

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.2. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SAC-QA-006 and policy S-Q-003.

13.3. Initial Demonstration

Each analyst must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration standard.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the historical acceptance criteria.

13.3.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.4. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

14.2. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. The following waste streams are produced when this method is performed.

15.1.1. Unused acidic digestate from the preparation process and remaining in the plastic tubes on the instrument. This acidic liquid is consolidated into a plastic LLE drum. When the drum is full or after no more than 75 days, move this drum to the main waste area for shipment

15.1.2. Aqueous acidic waste from the auto-analyzer. This is collected in a 1-gallon carboy. When the carboy is full, or after no more than one year, consolidate it into a plastic LLE drum for shipment.

15.1.3. Contaminated plastic vials from digestion and analysis. Pour any excess/unused sample from the vial into the aqueous acidic waste drum. Put the plastic vial into the contaminated lab trash. Collect all of the contaminated vials in a plastic bag, and move it to the waste collection area for shipment.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

16.2. “Methods for the Chemical Analysis of Water and Wastes”, EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.1.

16.3. QA-003-SAC, Quality Control Program.

16.4. QA-004-SAC, Rounding and Significant Figures.

16.5. SAC-QA-006, Method Detection Limits and Instrument Detection Limits.

16.6. SAC-QA-023, Nonconformance and Corrective Action System.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modification from Method 7470A and 245.1.

17.1.1. An autoclave is used for sample preparation rather than a hot plate (7470A) or water bath (245.1). See Appendix C.

17.1.2. The method has been modified for use with Leeman automated analyzers. The samples are prepared to a total volume of 30 mL versus the 100 mL volume specified in both methods. The addition of all reagents is modified relative to this decrease in volume. Stannous chloride in HCl is used in place of Stannous sulfate in H_2SO_4 (per instrument instructions). The analytical method is automated, whereas both methods are written for manual CVAA analysis.

17.1.3. STLC leachate analysis has been added to the methods.

17.1.4. The five point curve used by STL Sacramento has a 0.2 ppb Hg standard to accommodate analysis of one standard at our 0.2 ppm reporting limit. Both reference methods start with a 0.5 ppb Hg standard and include a 2.0 ppb Hg standard. We do not run the 2.0 ppb Hg standard.

17.2. Modifications from previous SOP

17.2.1. The Hydra AA unit recently purchased is referenced as a second analyzer with the PS200II.

17.2.2. 40 mL plastic test tubes for autosampler changed to 14 mL polystyrene.

17.2.3. Matrix spike and LCS preparation procedures were moved from section 7 to section 11.

17.2.4. Removed Table I from the SOP. Parameters are already defined in the SOP.

17.2.5. Updated the Quality Control section to current configuration requirements.

17.2.6. Added more detailed description of autoclave calibration.

17.2.7. Added sections 11.3 – 11.8 to section 11.

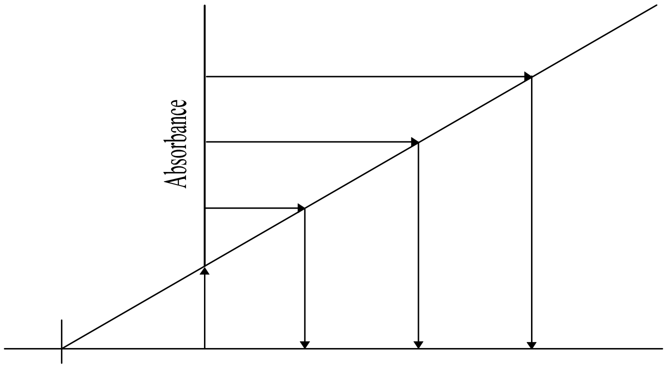
17.2.8. Updated the Method Performance section to current configuration requirements.

APPENDIX A. MSA GUIDANCE
Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause-cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F. AUTOCLAVE USAGE LETTER

Ms. Debra K. White
Principal Inorganic Scientist
Quanterra Incorporated
4955 Yarrow Street
Arvada, CO 80002

Dear Deb,

Thank you for your letters of July 10, 1995 and September 25, 1995, seeking clarification on several issues regarding RCRA testing and analysis. As the Office of Solid Waste updates SW-846 methods, we will take every opportunity to incorporate your comments and suggestions. Our goal is to remove overly restrictive language from SW-846 methods, which does not effect method performance and to continue to move toward truly performance based methods.

Your first request is for a clarification regarding the acceptance of the autoclave to prepare aqueous samples for mercury analysis under EPA SW-846 Method 7470A. The heating device in Section 4.10 of the Method 7470A specifies "Hot plate or equivalent." An autoclave would classify as an equivalent heating device and should give equivalent results to the hot plate/water bath protocol called for in Sections 7.1 and 7.2 of Method 7470A. Method 245.1 CLP-M is analogous to Method 7470A and allows the autoclave option for sample preparation.

Your second request is for a clarification regarding scaling of sample size for RCRA analysis. In particular, you request processing 50 mL aliquots for aqueous metal digestion rather than the 100 mL sample size specified in the methods. This should not present any problems for pure aqueous samples (no solids) because of their homogeneous nature. As a "representative sample" can be assured, scaling causes no loss of precision or accuracy in the analysis. Solid samples on the other hand are frequently heterogeneous in nature and changing sample size presents a big problem in obtaining a "representative sample" and should not be allowed without proper sample preparation (ie. crushing, grinding, mixing, and splitting).

I hope that this information is helpful to your analytical program. If you have any questions, please feel free to call me at (202) 260-4778.

Sincerely,

Oliver M. Fordham, Jr.

National Inorganic Program
Manager for RCRA

ATTACHMENT E

Target Analytes for the Test Program

TARGET PCDDs/PCDFS and PAHs

BOSTIK, INC. - MIDDLETON, MA

2008 RCRA TRIAL BURN / MACT CPT

Methods 0023A / 0010 / CARB 429 - Vista Analytical

Target PAH Compounds

Noncarcinogenic PAHs:
Naphthalene
2-Methylnaphthalene
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Benzo(e)pyrene
Perylene
Benzo(g,h,i)perylene
Carcinogenic PAHs:
Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-c,d)pyrene
Dibenz(a,h)anthracene

Target PCDD/PCDF Congeners

Chlorinated Dioxins:
2,3,7,8-TCDD
1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDD
1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD
1,2,3,4,6,7,8-HpCDD
OCDD
Chlorinated Furans:
2,3,7,8-TCDF
1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDF
1,2,3,6,7,8-HxCDF
2,3,4,6,7,8-HxCDF
1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF
OCDF

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TARGET SEMIVOLATILE ORGANICS

BOSTIK, INC. - MIDDLETON, MA

2008 RCRA TRIAL BURN / MACT CPT

Methods 3542 / 8270C Semivolatiles - Test America

SVOCs (excl. PAHs)	Filter/Rinse		XAD/Rinse		Condensate		Train Total	
	Rept Limit	MDL	Rept Limit	MDL	Rept Limit	MDL	Rept Limit	MDL
Benzyl alcohol	100 μg	35 μg	100 μg	35 μg	10 μg	0.76 μg	210 μg	70.76 μg
bis(2-Chloroethoxy)methane	10 μg	0.59 μg	10 μg	0.5 μg	10 μg	0.76 μg	30 μg	1.85 μg
bis(2-Chloroethyl) ether	10 μg	0.76 μg	10 μg	0.56 μg	10 μg	0.64 μg	30 μg	1.96 μg
bis(2-Ethylhexyl) phthalate	10 μg	3.3 μg	20 μg	10 μg	10 μg	0.8 μg	40 μg	14.1 μg
4-Bromophenyl phenyl ether	10 μg	0.53 μg	10 μg	0.5 μg	10 μg	0.54 μg	30 μg	1.57 μg
Butyl benzyl phthalate	10 μg	1.1 μg	10 μg	0.61 μg	10 μg	0.87 μg	30 μg	2.58 μg
4-Chloroaniline	10 μg	1.2 μg	20 μg	6 μg	10 μg	3.04 μg	40 μg	10.24 μg
4-Chloro-3-methylphenol	10 μg	1 μg	10 μg	0.62 μg	10 μg	2.5 μg	30 μg	4.12 μg
2-Chloronaphthalene	10 μg	0.5 μg	10 μg	0.5 μg	10 μg	0.53 μg	30 μg	1.53 μg
2-Chlorophenol	10 μg	0.98 μg	10 μg	0.5 μg	10 μg	0.63 μg	30 μg	2.11 μg
4-Chlorophenyl phenyl ether	10 μg	0.51 μg	10 μg	0.5 μg	10 μg	1.19 μg	30 μg	2.2 μg
Di-n-butyl phthalate	10 μg	0.71 μg	20 μg	10 μg	10 μg	0.86 μg	40 μg	11.57 μg
1,2-Dichlorobenzene	10 μg	0.84 μg	10 μg	0.51 μg	10 μg	0.66 μg	30 μg	2.01 μg
1,3-Dichlorobenzene	10 μg	1.2 μg	10 μg	0.57 μg	10 μg	0.54 μg	30 μg	2.31 μg
1,4-Dichlorobenzene	10 μg	1.1 μg	10 μg	0.53 μg	10 μg	0.77 μg	30 μg	2.4 μg
3,3'-Dichlorobenzidine	50 μg	2.7 μg	50 μg	7.4 μg	50 μg	2.96 μg	150 μg	13.06 μg
2,4-Dichlorophenol	10 μg	1.5 μg	10 μg	0.5 μg	10 μg	0.89 μg	30 μg	2.89 μg
2,6-Dichlorophenol	10 μg	0.72 μg	10 μg	0.5 μg	10 μg	1 μg	30 μg	2.22 μg
Diethyl phthalate	10 μg	1.5 μg	10 μg	0.73 μg	10 μg	0.53 μg	30 μg	2.76 μg
2,4-Dimethylphenol	10 μg	2.9 μg	10 μg	6.3 μg	10 μg	0.59 μg	30 μg	9.79 μg
Dimethyl phthalate	10 μg	0.63 μg	10 μg	0.5 μg	10 μg	0.5 μg	30 μg	1.63 μg
4,6-Dinitro-2-methylphenol	50 μg	5 μg	50 μg	8.7 μg	50 μg	0.56 μg	150 μg	14.26 μg
2,4-Dinitrophenol	50 μg	5.9 μg	50 μg	22 μg	50 μg	1.52 μg	150 μg	29.42 μg
2,4-Dinitrotoluene	10 μg	1.6 μg	10 μg	0.5 μg	10 μg	1.03 μg	30 μg	3.13 μg
2,6-Dinitrotoluene	10 μg	1.3 μg	10 μg	0.5 μg	10 μg	0.79 μg	30 μg	2.59 μg
Di-n-octyl phthalate	10 μg	2.1 μg	10 μg	0.56 μg	10 μg	0.96 μg	30 μg	3.62 μg
Hexachlorobenzene	10 μg	0.56 μg	10 μg	0.5 μg	10 μg	1.01 μg	30 μg	2.07 μg
Hexachlorobutadiene	10 μg	1.4 μg	10 μg	0.74 μg	10 μg	0.73 μg	30 μg	2.87 μg
Hexachlorocyclopentadiene	50 μg	10 μg	50 μg	10 μg	50 μg	2.5 μg	150 μg	22.5 μg
Hexachloroethane	10 μg	2.5 μg	10 μg	0.54 μg	10 μg	0.74 μg	30 μg	3.78 μg
Isophorone	10 μg	0.66 μg	10 μg	0.5 μg	10 μg	0.68 μg	30 μg	1.84 μg

TARGET SEMIVOLATILE ORGANICS

BOSTIK, INC. - MIDDLETON, MA

2008 RCRA TRIAL BURN / MACT CPT

Methods 3542 / 8270C Semivolatiles - Test America

	Filter/Rinse		XAD/Rinse		Condensate		Train Total	
SVOCs (excl. PAHs)	Rept Limit	MDL	Rept Limit	MDL	Rept Limit	MDL	Rept Limit	MDL
2-Methylphenol	10 μg	2.3 μg	10 μg	3 μg	10 μg	0.8 μg	30 μg	6.1 μg
3-Methylphenol & 4-Methylphenol	10 μg	2.3 μg	10 μg	2 μg	10 μg	0.85 μg	30 μg	5.15 μg
2-Nitroaniline	50 μg	0.56 μg	50 μg	0.5 μg	50 μg	1.06 μg	150 μg	2.12 μg
3-Nitroaniline	50 μg	3.8 μg	50 μg	2 μg	50 μg	1.79 μg	150 μg	7.59 μg
4-Nitroaniline	50 μg	2.3 μg	50 μg	2 μg	50 μg	1.46 μg	150 μg	5.76 μg
Nitrobenzene	10 μg	0.73 μg	10 μg	0.57 μg	10 μg	0.63 μg	30 μg	1.93 μg
2-Nitrophenol	10 μg	3.2 μg	10 μg	0.5 μg	10 μg	1.01 μg	30 μg	4.71 μg
4-Nitrophenol	50 μg	3.3 μg	50 μg	3.3 μg	50 μg	1.46 μg	150 μg	8.06 μg
N-Nitrosodiphenylamine	10 μg	0.6 μg	10 μg	0.87 μg	10 μg	0.52 μg	30 μg	1.99 μg
N-Nitrosodi-n-propylamine	10 μg	0.73 μg	10 μg	0.5 μg	10 μg	0.88 μg	30 μg	2.11 μg
Pentachlorophenol	50 μg	25 μg	50 μg	25 μg	50 μg	1.32 μg	150 μg	51.32 μg
Phenol	10 μg	1.1 μg	10 μg	0.9 μg	10 μg	0.82 μg	30 μg	2.82 μg
2,2'-Oxybis(1-Chloropropane)	10 μg	1 μg	10 μg	0.76 μg	10 μg	0.67 μg	30 μg	2.43 μg
1,2,4-Trichlorobenzene	10 μg	0.73 μg	10 μg	0.59 μg	10 μg	0.82 μg	30 μg	2.14 μg
2,4,5-Trichlorophenol	10 μg	2.3 μg	10 μg	1.3 μg	10 μg	0.81 μg	30 μg	4.41 μg
2,4,6-Trichlorophenol	10 μg	1.4 μg	10 μg	0.75 μg	10 μg	0.96 μg	30 μg	3.11 μg

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TARGET VOLATILE ORGANICS
BOSTIK, INC. - MIDDLETON, MA
2008 RCRA TRIAL BURN / MACT CPT

Methods 5041A / 8260B Volatiles - Air Toxics, Ltd.

Target VOCs	Reporting Limit (ng)
Chloromethane	10
Vinyl Chloride	10
Bromomethane	10
Chloroethane	10
Freon 11	10
2-Chloropropane	10
1,1-Dichloroethene	10
Freon 113	10
Carbon Disulfide	10
Acetone	50
3-Chloropropene	10
Methylene Chloride	10
trans-1,2-Dichloroethene	10
Acrylonitrile	10
Hexane	10
1,1-Dichloroethane	10
Vinyl Acetate	50
2,2-Dichloropropane	10
cis-1,2-Dichloroethene	10
Methyl Ethyl Ketone (2-Butanone)	50
Bromochloromethane	10
Chloroform	10
1,1,1-Trichloroethane	10
Carbon Tetrachloride	10
1,1-Dichloropropene	10
Benzene	10
1,2-Dichloroethane	10
Trichloroethene	10
1,2-Dichloropropane	10
Dibromomethane	10
Bromodichloromethane	10
cis-1,3-Dichloropropene	10
trans-1,3-Dichloropropene	10
4-Methyl-2-Pentanone	50
Toluene (POHC)	**10**
1,1,2-Trichloroethane	10
Tetrachloroethene	10
1,3-Dichloropropane	10
2-Hexanone	50
Dibromochloromethane	10

TARGET VOLATILE ORGANICS
BOSTIK, INC. - MIDDLETON, MA
2008 RCRA TRIAL BURN / MACT CPT

Methods 5041A / 8260B Volatiles - Air Toxics, Ltd.

Target VOCs	Reporting Limit (ng)
Chlorobenzene	10
1,2-Dibromoethane (EDB)	10
Ethyl Benzene	10
m,p-Xylene	10
o-Xylene	10
Styrene	10
Bromoform	10
1,1,2,2-Tetrachloroethane	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
1,2-Dichlorobenzene	10
Cumene	10
cis-1,4-Dichloro-2-butene	10
Bromobenzene	10
1,2,3-Trichloropropane	10
trans-1,4-Dichloro-2-butene	10
Propylbenzene	10
2-Chlorotoluene	10
4-Chlorotoluene	10
1,3,5-Trimethylbenzene	10
tert-Butylbenzene	10
1,2,4-Trimethylbenzene	10
sec-Butylbenzene	10
p-Cymene	10
1,2-Dibromo-3-Chloropropane	50
1,2,4-Trichlorobenzene	50
Hexachlorobutadiene	50
1,2,3-Trichlorobenzene	50
1,1,1,2-Tetrachloroethane	10
Butylbenzene	10
Iodomethane	10
Freon 12	10
Surrogates	**Method Limits**
Dibromofluoromethane	70 - 130
1,2-Dichloroethane-d4	70 - 130
Toluene-d8	70 - 130
4-Bromofluorobenzene	70 - 130

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TARGET INORGANICS

BOSTIK, INC. - MIDDLETON, MA

2008 RCRA TRIAL BURN / MACT CPT

EPA Methods 29 / 6020 / 7470A
Test America - Sacramento

Metals:
Antimony
Arsenic
Barium
Beryllium
Cadmium
Chromium (Total)
Lead
Mercury
Nickel
Silver
Thallium

EPA Methods 26A / 5
Test America - Knoxville

Inorganics:
Particulate Matter
Hydrogen Chloride
Chlorine

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Appendix B

Continuous Monitoring System Performance Evaluation Test Plan

Continuous Monitoring System (CMS)

Performance Evaluation Test (PET) Plan

CMS PET Plan

Bostik, Inc.
RCRA Trial Burn Plan / MACT CPT Plan

Contents

1.0 Introduction

As part of complying with the requirements of the HWC MACT regulations, facilities must submit a plan along with the CPT Plan and perform an evaluation of their “Continuous Monitoring System” as part of the CPT. EPA defines the CMS in 40 CFR 63.2:

“Continuous Monitoring System (CMS) is a comprehensive term that may include, but is not limited to, continuous emission monitoring systems, continuous opacity monitoring systems, continuous parameter monitoring systems, or other manual or automatic monitoring that is used for demonstrating compliance with an applicable regulation on a continuous basis as defined by the regulation.”

Based on the above definition, the main components of the CMS for the Bostik polyester burner unit includes the following:

- Process instruments that monitor or control key process parameters, including the unit’s continuous emissions monitoring system;
- The data control system (DCS) and data acquisition system (DAS);
- The AWFCO system; and
- The programmable logic that utilizes regulatory set-points to assure compliance.

The CMS Performance Evaluation Test (PET) Plan required by 40 CFR 63.8(e)(4) and 63.1207(b)(1) utilizes a combination of activities to accomplish its objective, which is to verify that the polyester burner is properly controlled and that the equipment and systems that are used are operating properly and are accurate. These activities include instrument audits or calibrations, auditing the function of the AWFCO system and the programmable logic used in the DCS. This plan describes the CMS itself, the procedures and documentation practices that will be used to verify the functionality of the CMS and the Quality Assurance requirements of the evaluation.

2.0 Continuous Monitoring System Description

This section provides an overview of the key components of the CMS. This CMS evaluation includes field instrumentation; the DCS / DAS; the programmable logic and field control (e.g., control and block valves).

2.1 Field Instrumentation

Tables 4-2 and 4-4 in Section 4.0 of this document provide information pertaining to field instruments that are part of the overall CMS. These instruments monitor and control certain process operations to assure the unit is operating safely and in compliance with applicable environmental requirements. The instruments used for these aspects of process control meet the definition of a "continuous monitor" in 40 CFR 63.1201.

As part of initial instrument specification prior to installation and use in the process, instrument audit and calibration procedures are identified or developed. These procedures specify the frequency of auditing the instrument's function and accuracy and the actual procedure for verification. These procedures specify both the specific steps and the acceptable accuracy requirements that the instrument must meet to "pass". Troubleshooting procedures are typically included to help plant personnel correct any problems and get the instruments operational.

2.2 Continuous Emission Monitoring System

In addition to other field instrumentation, the operation of the polyester burner also relies on its CEMS to monitor stack emissions concentrations. This system is described in Section 4.6.3 of this document with system specifications provided in Table 4-3. When emission levels deviate from allowable limits, the DCS takes appropriate action up to and including initiation of an AWFCO.

2.3 Process Control

The process control systems for the HWC are described in Section 4.6 of this Plan. These systems detect signals from process instruments, perform calculations according to the programmable logic, adjust control equipment and notify operators when key process parameters deviate from acceptable limits. In addition to notifying operating personnel, the AWFCO system described in Section 4.6.4 will automatically shut down the waste feed and the overall process itself in the event of deviations outside acceptable operating limits.

2.4 CMS Operation

All the components of the CMS must be operational for the polyester burner to combust waste. The DCS and overall process control system are designed in such a manner as to continually verify that operation while the unit is running. Field instrumentation (both sensing and control) are connected to the DCS in "control loops" with common wiring, electrical signal transmitters, input / output devices and related programmable logic. All components of each control loop related to the feeding of each material (i.e., waste distillate and the process vent stream) must be operating for the burner to be enabled to burn that stream. The programmable logic is designed in such a way that it can sense and verify that various components of the process (and the overall process) are operating as required. For example, if power is lost to the flow meter on the waste feed line, the programmable logic will sense this signal loss and initiate either an alarm or shutdown. Similarly, if the actual field position of a specific control valve disagrees beyond a certain amount with what the programmable logic calculates it should be, appropriate alarms and / or shutdowns are initiated. (A complete listing of operating limits was provided previously in Section 2.0.)

2.5 Management of Change

A Management of Change (MOC) procedure is implemented at Bostik to ensure that adequate levels of communication exist between all departments when changes are made which can affect the process. A change made in one part of the process or other processes may have unintended effects on other parts of the process because the stationary sources are an integrated system. These proposed changes are therefore appropriately scrutinized before they are made to ensure the changes do not compromise the safety and integrity of the process and avoid adverse effects or worker and public safety and the environment.

The MOC evaluation form includes changes which impact:

- Process chemicals;
- Technology;
- Equipment;
- Procedures; and
- Employees.

Any change to these criteria triggers the use of an MOC form. This procedure does not apply to "replacement in kind" which is defined as replacements that satisfy the design specifications. A form is used to document the MOC procedure when the above changes are planned by facility personnel.

Each type of change requires the appropriate signatures and authorization to proceed with the change. As part of the MOC form, an action item checklist is used to identify tasks as a result of the change and to track their completion. Personnel (e.g., engineering, operations, safety) assess the potential impact of the change on safety and health through a safety checklist for minor changes or a process hazard analysis (PHA) for major changes. The following steps are taken to ensure proper implementation of the change:

- Process safety information is updated as necessary but always prior to startup;
- Operating procedures are updated as necessary in accordance with the written operating procedure program.; and
- Employees (e.g., operations, maintenance, contract) whose job tasks are affected by the change, are trained in accordance with the written training program prior to the startup of the process.

The MOC forms and documentation of completed action items are forwarded to the appropriate personnel for authorization. The MOC records and recommendations are tracked according to the assigned checklist.

3.0 CMS Performance Evaluation Test Plan

As described in the preceding sections, the CMS Performance Evaluation Test Plan relies on a combination of activities to determine whether the CMS is operating properly. This will include the following:

- Auditing the instrument maintenance and calibration program;
- Calibrating field instruments;
- Auditing the AWFCO Testing Program; and
- Auditing portions of the programmable logic to verify that the AWFCO set-points used assure regulatory limits will be met.

Personnel who are knowledgeable of burner operations, their process control systems and relevant regulatory requirements, will perform these activities.

3.1 Instrument Audit and Calibration

As part of conducting the CMS Performance Evaluation, a two step process will be used to assess the status of the various field instruments identified in Section 4.0. First, audit / calibration records will be reviewed for these instruments to determine when their most recent audit or calibration occurred. From this review, any instruments that are approaching the end of their audit/calibration cycle will be scheduled for audit and/or calibration prior to performing the actual Trial Burn / CPT. For example, if a mass flow meter (annual audit cycle) has been audited / calibrated within several months of the CPT, it will not be re-calibrated prior to the CPT. If however, that same flow meter hasn’t been audited / calibrated within the last nine to ten months, it will be audited and/or calibrated prior to the CPT.

Because certain instruments cannot be audited or calibrated without taking the combustion system offline, these will be scheduled over a period of time prior to the CPT to minimize process interruptions and shutdowns. All instruments requiring pre-CPT audits/calibrations will be tested prior to the CPT.

3.2 AWFCO System Performance Evaluation

Another component of the CMS Performance Evaluation is auditing the AWFCO system and related DCS logic. This will be accomplished by reviewing the last year of AWFCO testing logs to assess whether there are any recurring problems with the AWFCO system. Any incidence of problems with the AWFCO system will be identified for follow-up and correction prior to the CPT.

This evaluation will also include examining the appropriate programmable logic statements to compare the AWFCO set-points with the applicable operating parameter limits to assure that these are appropriate.

3.3 Auditing the CEM System

The CEMS used at Bostik is installed, operated and maintained to comply with the provisions of the applicable performance specifications and/or EPA Reference Methods. In general, this means that the CEMs are calibrated daily (zero/span), quarterly (gas audits) and annually (Relative Accuracy Test Audits). Thus, the evaluation of the performance of this system will be done as part of meeting those requirements and a separate evaluation will not be conducted under this plan.

3.4 Schedule

The Performance Evaluation itself will be conducted from one to three months prior to a CPT or Confirmatory Test as required by the HWC MACT regulations. All CMS Performance Evaluation activities will be completed with all components meeting their respective accuracy requirements prior to performing the CPT.

3.5 Reporting and Documentation

The results of the CMS Performance Evaluation will be included as part of the Final Trial Burn Report / Notification of Compliance as required by 40 CFR 63.9(h)(2). This will include the following information:

- Description of the CMS components;
- Description of the CMS Performance Evaluation Plan;
- Listing of all field instruments that are part of the CMS and their audit/calibration status;
- Listing of field instruments that have been specifically audited/calibrated as part of the CMS Performance Evaluation;
- Copies of the most recent audit/calibration results for CMS instruments;
- AWFCO system evaluation results;
- CEM system evaluation results; and
- Copies of relevant programmable logic statements showing where calculations and regulatory alarms and set-points are used in the coding to assure compliance.

4.0 Quality Assurance

The quality assurance requirements for this Performance Evaluation are specified in the table below. The QA requirements for CMS equipment components are established by other criteria outside this Performance Evaluation.

Table 4-1 Quality Assurance for CMS Performance Evaluation

CMS Component	Basis for QA Requirement	QA Specification
Field Instruments	Manufacturer recommendations	Audit/calibration meets recommended specifications for all affected instruments
AWFCO System Evaluation	RCRA permit and MACT requirements	No failures of the AWFCO system
CEM System	40 CFR 60, Appendices A, B and F and Appendix to Subpart EEE	Meets those specifications
Programmable Logic	RCRA Permit and MACT requirements	All set points programmed correctly