PERFORMANCE DEMONSTRATION TEST
QUALITY ASSURANCE PROJECT PLAN
DESOTEC - PARKER FACILITY

PREPARED FOR:

DESOTEC

DESOTEC US LLC
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PARKER, ARIZONA 85344

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REVISION 5
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1.0 QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM AND DISTRIBUTION LIST

Project: DESOTEC – Parker Facility

RCRA Subpart X Performance Demonstration Test

Parker, Arizona

Approved Plan Submittal Date: _____

Scheduled Test Start Date: _____

Key test Personnel Approvals and Distribution

Name/Function/Organization	Signature	Date
Plant Manager DESOTEC US LLC		
Test Manager Focus Environmental, Inc.		
Quality Assurance Officer Focus Environmental, Inc.		
Stack Sampling Coordinator Alliance Technical Group, LLC		
Laboratory Project Mananer Eurofins Environment Testing, Knoxville, TN		

QAPP Distribution List

Project Organization Title	Organization/Name
Plant Manager	DESOTEC US LLC
Test Manager	Focus Environmental, Inc.
Quality Assurance Officer	Focus Environmental, Inc.
Stack Sampling Coordinator	Alliance Technical Group, LLC
Sample Custodian	Alliance Technical Group, LLC and Focus Environmental, Inc.
Laboratory Project Manager	Eurofins Environment Testing, Knoxville, TN
Tribal Representative	Colorado River Indian Tribes (CRIT)
U.S. Environmental Protection Agency	EPA Region IX

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ACRONYMS AND ABBREVIATIONS

ASTM	American Society for Testing Materials
B.P.	Boiling Point
CAR	Corrective action request
CCV	Continuing calibration verification
CEM	Continuous emissions monitor
CEMS	Continuous emissions monitoring system
CF	Calibration factor
CLP	Contract Laboratory Program
CMS	Continuous monitoring system
COC	Chain of Custody
CRIT	Colorado River Indian Tribes
CVAA or CVAAS	Cold vapor atomic adsorption spectroscopy
DI	Deionized (water)
DQO	Data quality objective
DRE	Destruction and removal efficiency
dscf	Dry standard cubic foot
dscfm	Dry standard cubic feet per minute
dscm	Dry standard cubic meter
dscmm	Dry standard cubic meters per minute
ECD	electron capture detectors
EDL	Estimated detection limit
EPA	U.S. Environmental Protection Agency
FID	flame ionization detector
GC/MS	Gas chromatograph/mass spectrometry
g	grams
gr	Grains
HC	Hydrocarbons
HHERA	Human health and ecological risk assessment
HRGC/HRMS	high resolution gas chromatograph/high resolution mass spectrometry
HWC	Hazardous Waste Combustor
ICP or ICAP	Inductively coupled argon plasma spectroscopy
ICP-MS or ICAP-MS	Inductively coupled argon plasma spectroscopy/mass spectroscopy
ICV	Initial calibration verification
kg	Kilograms
L	Liter
lb or lbs	Pounds
LCS	Laboratory control standard
LVM	low-volatile metals
MACT	Maximum Achievable Control Technology
MDL	Method detection limit
mg	Milligrams
μg or ug	Micrograms
MS	Matrix spike
MSD	Matrix spike duplicate
ND	Not Detected
ηg or ng	Nanograms
NO_X	nitrogen oxides
OCP	Organochlorine Pesticide
PAH	Polycyclic Aromatic Hydrocarbon

PCB	Polychlorinated Biphenyl
PCDD	Polychlorinated dibenzo-p-dioxin
PCDF	Polychlorinated dibenzofuran
PDS	Post-digestion spike
PDT	Performance Demonstration Test
PDTP	Performance Demonstration Test Plan
PE	Performance evaluation
PIC	Product of incomplete combustion
POHC	Principal organic hazardous constituent
ppm	Parts per million
ppmv or ppmdv	Parts per million dry volume
QA	Quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality control
RCRA	Resource Conservation and Recovery Act
RFA	Request for analysis
RL	Reporting limit
RPD	Relative percent difference
RSD	Relative Standard Deviation
RF	Response factor of Reactivation Furnace (note difference by context)
RSD	Relative standard deviation
RT	Retention time
SEM	scanning electron microscope
SIM	selected ion monitoring
SO_2	sulfur dioxide
SOP	Standard operating procedure
SVM	semi-volatile metal
TCL	Target Compound List
THC	Total hydrocarbons
TIC	tentatively identified compound
WM	Wide-mouth
VOA	Volatile organic analysis
VOC	Volatile organic compound
VOST	Volatile organic sampling train

3.0 PROJECT DESCRIPTION

3.1 GENERAL

DESOTEC US LLC (DESOTEC) operates a carbon reactivation facility located in the Colorado River Indian Tribes (CRIT) Industrial Park near Parker, Arizona. The facility treats spent activated carbon that has been used by industry, state and federal government agencies, and municipalities for the removal of organic compounds from liquid and vapor phase process waste streams. Once the carbon has been used and is spent, it must be either disposed of or reactivated at a facility such as operated by DESOTEC. A Carbon Reactivation Furnace (RF) is used by DESOTEC to reactivate the spent carbon. Some of the carbon received at the Parker facility is designated as hazardous waste under the Resource Conservation and Recovery Act (RCRA) regulations. DESOTEC has prepared the required RCRA Subpart X Performance Demonstration Test Plan (PDTP) which is designed to demonstrate the capability of RF unit to operate within the applicable emission limitations. DESOTEC will also perform a site-specific multiple pathway human health and ecological risk assessment (HHERA) as required by the RCRA permit. Accordingly, DESOTEC has prepared the PDTP and this Quality Assurance Project Plan (QAPP) to incorporate the gathering of emissions data to demonstrate compliance with the applicable regulatory requirements and for use in the HHERA. Specific guidance issued by EPA (Risk Burn Guidance for Hazardous Waste Combustion Facilities, EPA530-R-01-001, July 2001) has been used, along with the facility's previous Risk Assessment, to preliminarily identify compounds of potential concern for the HHERA and to select appropriate sampling and analytical techniques.

3.2 TEST SCOPE

The DESOTEC Performance Demonstration Test Plan has been prepared to provide comprehensive performance testing of the RF unit to demonstrate compliance with the permit emissions and performance standards that are shown in RCRA Permit Condition V.I. and Table V-1. The objectives of the PDT are to demonstrate compliance with the RCRA Permit operating limits such as Destruction and Removal Efficiency (DRE) and particulate matter emission concentration, while operating at "worst case" (at RCRA Permit limit) conditions processing normal feed materials, which have been augmented with metals, chloride, etc., to confirm the operating conditions in the RCRA Permit. More specifically, the objectives of the Performance Demonstration Test (PDT) are as follows:

1. Demonstrate Compliance with the RCRA Permit and the permit operating limits set forth in RCRA Permit Condition V.I and Table V-1. The operating parameter limits are listed in Column 3 of Table V-1. The Performance Standards are listed in Column 2 of Table V-1.
    - Demonstrate a DRE of greater than or equal to 99.99% for the selected principal organic hazardous constituents (POHCs) chlorobenzene and tetrachloroethene.
    - Demonstrate stack gas carbon monoxide concentration less than or equal to 100 ppmv, dry basis, corrected to 7% oxygen.

- Demonstrate stack gas hydrocarbon concentration of less than or equal to 10 ppmv, as propane, dry basis, corrected to 7% oxygen.
- Demonstrate a stack gas particulate concentration less than or equal to 0.013 gr/dscf corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of hydrogen chloride (HCl) and chlorine (Cl_2) are no greater than 32 ppmv, dry basis, corrected to 7% oxygen, expressed as chloride (Cl^-) equivalents.
- Demonstrate that the stack gas mercury concentration is less than or equal to 130 μg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of semivolatile metals (SVM, cadmium and lead, combined) is less than or equal to 230 μg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of low volatility metals (LVM: arsenic, beryllium, and chromium, combined) is less than or equal to 92 μg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of dioxins and furans does not exceed 0.40 ng/dscm, corrected to 7% oxygen, expressed as toxic equivalents of 2,3,7,8-TCDD (TEQ). This is the applicable standard, shown in Table V-1 Column 2 and 3 of the RCRA Permit, since the gas temperature entering the first particulate matter control device is less than 400°F.
- Demonstrate an emission rate of SO_2 corresponding to an annual emission rate of less than or equal to 30 tons per consecutive 12-month period.
- Demonstrate an emission rate of NOx corresponding to an annual emission rate of less than or equal to 22 tons per consecutive 12-month period, and develop a NOx emission factor in terms of mass of NOx emitted per volume of natural gas consumption.

2. Confirm or Establish Revised RCRA Permit Operating Limits (As referenced in Table V-1, column 3 of the RCRA Permit.)

   Control Parameters that influence DRE:

   - Demonstrate maximum feed rate for spent activated carbon.
   - Demonstrate minimum afterburner gas temperature.
   - Demonstrate maximum combustion gas velocity (or a suitable surrogate indicator).

   Feed rate limits:

   - Demonstrate maximum total chlorine/chloride feed rate.
   - Demonstrate mercury emissions compliance by confirming that the existing Maximum Theoretical Emission Concentration (MTEC)-based limit is acceptable.
   - Demonstrate system removal efficiency (SRE) for semivolatile and low volatility metals so feed rate limits can be confirmed by extrapolation from test results.
   - Confirm/Establish appropriate operating limits for the air pollution control system components.

3. Gather Information for Use in a Site-Specific Human Health and Ecological Risk Assessment (HHERA).

- Measure emissions of an expanded list of metals, including hexavalent chromium, and an expanded list of volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs).
- Measure emissions of hydrogen chloride and chlorine.
- Measure emissions of specific volatile and semivolatile products of incomplete combustion (PICs).
- Measure emissions of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDF).
- Measure emissions of polycyclic aromatic hydrocarbons (PAHs).
- Measure emissions of polychlorinated biphenyls (PCBs).
- Measure emissions of specific organochlorine pesticides (OCPs).
- Measure emissions of total volatile, semivolatile, and nonvolatile organics.
- Measure the stack gas particle size distribution.

3.2.1 Test Protocol Summary

To accomplish the PDT objectives, (i.e., demonstrating that the unit will meet all applicable RCRA Permit performance standards) a single test condition representing "worst case" operations of minimum temperature, maximum combustion gas velocity (minimum residence time), and maximum spent carbon feed rate will be performed. "Worst case" therefore means intentionally operating at or beyond the current RCRA Permit operating limits. The purpose of the fourth test run is an allowance for the following during any test run: 1) possible loss or damage to all or portions of any sample(s) or sample fraction(s), 2) rejection of a specific sample(s) due to sampling or analytical data quality reasons, or 3) deviation/closeness to the system operational targets. Desotec's intent is to select three test runs that are 100% complete for demonstrating compliance. Data from the three selected runs, the first three test runs or any combination of three of the four test runs, will be used to demonstrate compliance with the RCRA permit conditions and risk assessment data collection requirements. Should Desotec elect to exclude a test run for Item 3 above, or should there be data quality issues or incomplete samples with a particular sample data set (Item 1 or Item 2 above), valid data for the additional or "extra" test run may be substituted and used for compliance demonstration and/or risk assessment modeling. In the event that conditions (1), (2), or (3) above invalidate or potentially invalidate a test run, Desotec will substitute the entire data set from the additional test run in place of the invalid test run. EPA's approval will be required prior to substituting any portion of a test run. Compliance with the current associated RCRA permit OPLs, or possible establishment of new OPLs, will be reconciled in accordance with 40 CFR 63.1209(i) as may be necessary.

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

3.2.2 Test Conditions ("Worst-Case" Operations)

The sampling and monitoring protocols that will be utilized during the PDT are summarized as follows:

- Spent Activated Carbon Feed - total chlorine/chloride, elemental (C, H, N, O, and S), moisture, volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn).
- Stack gas particulate, HCl, and Cl_2 using EPA Method 5/26A.
- Stack gas target volatile organic compounds (VOCs) using volatile organic sampling train (VOST), SW-846 Method 0030.
- Stack gas target semivolatile organic compounds (SVOCs) using SW-846 Method 0010.
- Stack gas organochlorine pesticides (OCPs) using a second and separate SW-846 Method 0010 sampling train.
- Stack gas PCDD/PCDFs, PCBs, and polycyclic aromatic hydrocarbons (PAH) using EPA Method 23 (March 2023).
- Stack gas total volatile organics using SW-846 Method 0040.
- Stack gas total semivolatile and nonvolatile organics [a.k.a., total chromatographable organics and gravimetric organics (TCO/Grav)] using SW-846 Method 0010.
- Stack gas metals (Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn) using EPA Method 29.
- Stack gas hexavalent chromium using SW-846 Method 0061.
- Stack gas particle size distribution (PSD) using a second and separate Method 5 sampling train with a smooth surface polycarbonate filter compatible with scanning electron microscopic (SEM) analysis.
- Stack gas CO and O_2 by permanently installed CEM according to the protocols in the Appendix to 40 CFR 63, Subpart EEE; Performance Specification 4B of 40 CFR 60, Appendix B.
- Stack gas total hydrocarbons (as propane) by temporary CEM according to EPA Method 25A and the protocols in the Appendix to 40 CFR 63, Subpart EEE.
- Stack gas Sulfur Dioxide (SO_2) and Nitrogen Oxides (NOx) by temporary CEM according to EPA Methods 6C and 7E, respectively.
- Scrubber blowdown - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)

3.3 QUALITY ASSURANCE PROJECT PLAN SCOPE

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

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

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

4.0 ORGANIZATION OF PERSONNEL, RESPONSIBILITIES, AND QUALIFICATIONS

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

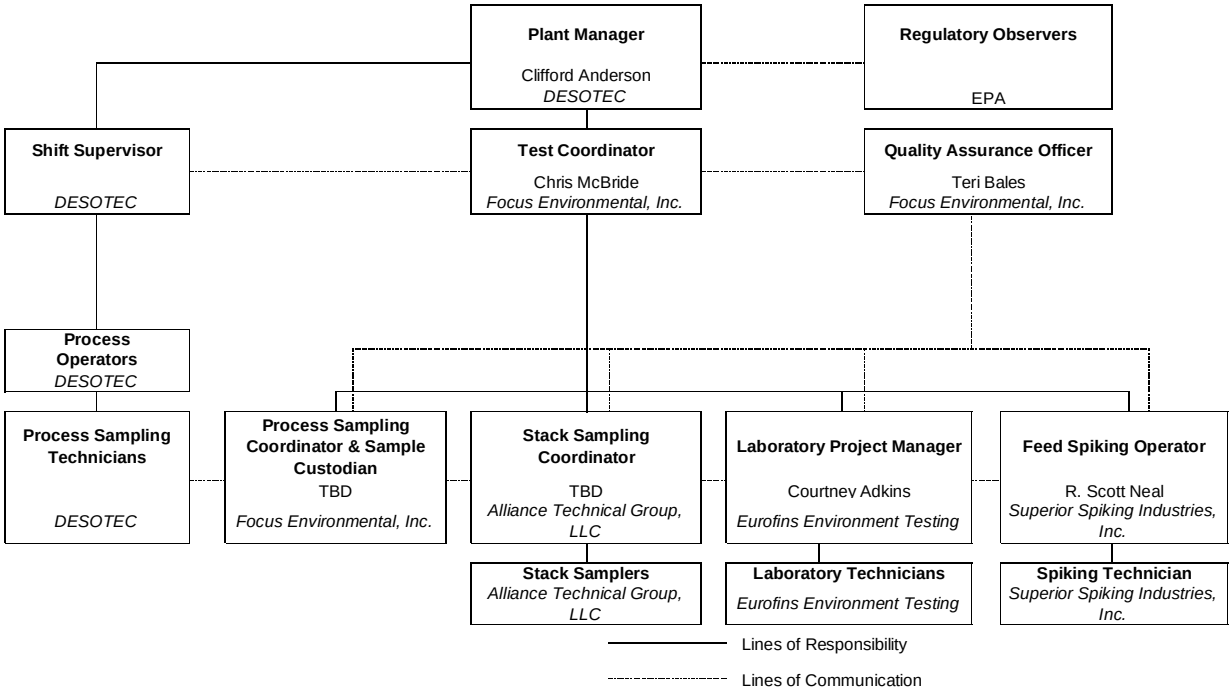

Figure 4-1. Performance Test Project Organization and Responsibilities

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

The contracted project team will implement the test program. The Test Manager will be a consulting contractor who is experienced in the technical coordination and QA/QC associated with the testing of hazardous waste thermal treatment systems. The stack sampling for this project will be conducted by a contractor who is experienced in conducting the stack sampling called for in the PDT plan. Analytical

services will be provided by a contract laboratory(s) experienced and National Environmental Laboratory Accreditation Program (NELAP) accredited in the analysis of the PDT program stack emissions test samples and process samples.

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

- Ensuring compliance with the PDT plan and the QAPP by all project team members during the test
- Documenting testing activities in a field logbook
- Assisting DESOTEC in interfacing with the regulatory observers and/or oversight contractors during the test
- Providing coordination among DESOTEC personnel and the sampling team during the test, especially regarding decisions to start, stop, hold or repeat sampling runs
- Providing field review of process operating logs, sample collection sheets, stack sampling logs, chain of custody forms, and request for analysis forms
- Interfacing with the Laboratory Project Manager while samples are being analyzed
- Interfacing with the Stack and Process Sampling Coordinators while the sampling data is being reduced
- Supervising production of the PDT Report
- Certifying the overall PDT Results and PDT Report.

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

- Reviewing the stack sampling and analytical reports for completeness and accuracy
- Reviewing the sample chain of custody and request for analysis forms for conformance with the sample custody and preservation requirements (refer to Section 7.0)
- Performing an analytical data validation review for conformance with the QAPP analytical data quality objectives (DQOs) (refer to Section 5.0)
- Document the results of the sample custody, sample preservation, and analytical data validation reviews in a written report, included with the PDT report.

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

- Coordinating the preparation and shipment of process sampling equipment, and shipping containers to the test site

- Directing and/or participating in process sampling activities
- Reviewing and approving process sample collection sheets and field data sheets
- Overseeing recovery of samples and preservation of the process samples in the field
- Documenting all the process samples collected
- Preparing a report of the process sampling activities.

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

- Preparing and shipping stack sampling equipment to the test site
- Preparing and calibrating stack sampling equipment
- Directing and/or participating in stack sampling activities
- Recording field test data required by the PDT plan and stack sampling methods
- Reviewing and approving stack sample collection sheets and stack sampling field data sheets
- Overseeing the recovery of the stack samples and preservation of those samples
- Preparing chain of custody (COC) and request for analysis (RFA) forms for the stack gas samples collected
- Reducing the stack sampling data and performing all calculations and QA activities required by the stack sampling methods
- Preparing a report of stack sampling activities.
- Notify the Sample Custodian of all emissions samples taken.

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

- Maintaining the RF systems within specified PDT target operating values
- Maintaining logs of process data as required
- Downloading and providing electronic files in Microsoft Excel format of the RF operating data to the Test Manager
- Collecting selected process samples (if required)
- Notifying the Sample Custodian of all process samples collected.

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

- Providing sample collection and recovery containers to the stack sampling team and process sample technicians
- Assigning and recording sample numbers
- Preparing samples and packaging them for shipment to the laboratory
- Preparing and reviewing COC and RFA forms for all samples
- Coordinate shipping of all samples to the laboratory.

- Monitoring the shipment of samples to the laboratory to ensure that all samples are received on schedule and with all preservation requirements being met (Any discrepancies should be immediately reported to the QAO, Test Manager and DESOTEC).

A Laboratory Project Manager will be appointed for the laboratory that provides analytical services for the project. His/her responsibilities will include:

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

Resumes of key individuals who will be implementing the test are presented in Appendix A. Note that at the time of document preparation, all personnel may not have been identified, so all resumes may not be included in Appendix A at this time. As individuals are identified to fill the designated project roles, their resumes will be added to the project records.

5.0 QUALITY ASSURANCE OBJECTIVES AND QUALITY CONTROL OBJECTIVES

5.1 GENERAL

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

The field and laboratory data obtained during this test will be reviewed by the Quality Assurance Officer, and a complete assessment of the data quality indicators will be included in the final test report. The data quality will be discussed with respect to meeting the respective data quality objectives (DQOs) and the overall project objective. The impacts of exceeding the delineated DQOs relative to the PDT objectives will be assessed on a case-by-case basis as to impacts on the test program objectives in the quality assurance discussion section of the final test report. Specifically, the data will be assessed as to possible biases and the associated impacts on compliance determinations and HHERA data usability.

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

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

Accuracy: The degree of agreement of a measurement (or an average of measurements of the same parameter) X, with an accepted reference or true value, T. Accuracy is usually expressed as the difference between the two values, X – T, or the difference as a percentage of the reference or true value, 100 (X – T)/T, and sometimes expressed as a ratio, X/T. In some cases, accuracy is described as the percentage

recovery of a known quantity of material added to a sample prior to analysis. Accuracy is a measure of the bias in a system.

Completeness: A measure of the amount of valid and usable data obtained compared to the amount expected to be collected under normal conditions. Completeness may be expressed as a percentage *or evaluated on a specific sample basis and its associated impacts on test determinations*.

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

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

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

5.2 PRECISION AND ACCURACY

A number of procedures will be used to meet the precision and accuracy objectives of the analytical program. All sampling and analytical activities will be conducted following referenced procedures. All reference materials used as calibration standards, surrogate compounds, or laboratory control samples will be of the highest purity commercially available. Reagent grade compounds for matrix spikes and surrogates will be used. All spiking levels will be in accordance with the referenced methods. Table 5-1 lists the organic compounds and the applicable control limits of laboratory surrogates and field spikes to be used to spike organic compound samples. The calibration of instruments used during analysis will be verified each day that samples are analyzed as described in later sections of this QAPP. Assessment of data precision and accuracy will be accomplished by evaluating the results from multiple analyses of the same parameter, and analysis of standards, duplicates and spiked samples. Field and laboratory contamination will be assessed through the analysis of reagent, instrument, method, field, and trip blanks.

Precision estimates presented in Table 5-1 represent variability for replicate measurements of the same parameters, expressed in terms of relative percent difference (RPD) for duplicate samples or relative standard deviation (RSD) for three or more measurements, as appropriate. For analyses of samples with detectable concentrations of the target analytes, precision is evaluated by conducting duplicate analyses of unspiked samples and assessing the RPD. In the evaluation of larger data sets (three or more data points), the RSD is assessed. When duplicate analyses are performed, the original analysis result is used in test calculations. If the variance in the duplicate analyses bring into question the analytical precision,

additional analyses, if allowed by the method, will be performed to better determine the actual value or to evaluate the potential reason(s) for the measurement variability.

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

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

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

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

5.2.1 General CEMS Precision and Accuracy

The precision of the installed carbon monoxide and oxygen continuous emissions monitoring system (CEMS) analyzers will be assessed via a Relative Accuracy Test Audit (RATA) performed prior to the test using the recommended calibration gases in accordance with 40 CFR 60, Appendix B, Specification 4B. The precision

of the temporary hydrocarbon, nitrogen oxides, and sulfur dioxide analyzers will be assessed during the test using the recommended calibration gases in accordance with 40 CFR 60, Appendix A, Methods 25A, 7E, and 6C, respectively. Precision will be assessed using the following equation:

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

where:

R_f = Final monitor response at end of the test run

R_i = Initial monitor response at start of the test run

Span = Maximum range of the analyzer.

The installed carbon monoxide and oxygen CEMS include gas conditioning (moisture removal) and therefore measure stack gas concentrations on a dry basis. The oxygen concentrations as measured by the installed oxygen CEMS will be used to continuously correct the installed carbon monoxide CEMS to seven (7) percent oxygen. For other emissions measured via temporary CEMS and the various sampling trains, concurrently measured average oxygen concentration data via Method 3A will be used to oxygen correct emissions concentrations as may be needed or appropriate.

The accuracy of the temporary NO_X and SO_2 CEMS analyzers will be evaluated during the test by the measurement of percent accuracy as defined by the equation below:

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

where:

R_a = Analyzer indicated concentration of the calibration gas

R_c = Certified concentration of the calibration gas

Span = Maximum range of the analyzer.

The accuracy of the Method 25 CEMS analyzer will be evaluated during the test by the measurement of percent accuracy with “Calibration Gas Value” replacing “Span” in the preceding equation. The accuracy of the Method 3A CEMS analyzer will be evaluated during the test by the absolute measurement difference (R_a -R_c) expressed as percent CO_2 or O_2.

The test run average Method 3A CO_2 or O_2 emissions concentration data will be used to calculate stack gas dry molecular weight. The same Method 3A data will be used for all sampling trains from the same test

run. Reduced Method 2 and Method 4 data from the respective isokinetic sampling trains will be used to calculate the emissions concentrations and mass emission rates of the target analytes associated with the respective sampling trains. For non-isokinetic sampling methods that do not include flow measurements (e.g., Method 0030 and Method 0040), stack gas flow rate measured using concurrently operated isokinetic sampling trains (e.g., average of the EPA Method 23 and the three SW-846 Method 0010 variants) will be used to calculate mass emission rates of the target analytes. Similarly, total hydrocarbons as measured by EPA Method 25A are wet and not oxygen corrected. EPA Method 4 moisture data from the concurrently operated isokinetic sampling trains and oxygen data from EPA Method 3A will be used to correct and report total hydrocarbons in parts per million by volume, corrected to seven (7) percent oxygen by volume, dry basis.

5.2.2 Temporary CEMS Data Collection

Temporary CEMS used for CO_2, O_2, THC, SO_2, and NO_X (Methods 3A, 25A, 6C, and 7E) will include a Data Acquisition System with battery backup to record the instrument response in one (1) minute averages. The data will be continuously stored as a *.CSV file in Excel format on the hard drive of a computer.

5.2.3 Temporary CEMS Sampling System and Calibration Gases

The CEMS sampling system for CO_2, O_2, SO_2, and NO_X (Methods 3A, 6C, and 7E) will consist of a heated stainless-steel probe, heated Teflon sample line(s), gas conditioning system and the identified analyzers. The gas conditioning system will be a non-contact condenser used to remove moisture from the source gas.

The CEMS sampling system for THC (Method 25A) will consist of a heated stainless-steel probe, heated Teflon sample line(s), and the identified analyzer. The THC sampling does not include a gas conditioning system to remove moisture from the source gas.

All calibration gases used shall meet EPA Protocol 1 (+/- 2%) standards.

5.2.4 Methods 6C and 7E SO_2 and NO_X CEMS Precision and Accuracy

Method 6C and 7E include the following additional precision and accuracy checks and assessments.

5.2.4.1 Direct Calibration & Calibration Error Test

Low-Level gas will be introduced directly to the analyzer. After adjusting the analyzer to the Low-Level gas concentration and once the analyzer reading is stable, the analyzer value will be recorded. This process will be repeated for the High-Level gas. For the Calibration Error Test, Low-, Mid-, and High-Level calibration gases will be sequentially introduced directly to the analyzer. The Calibration Error for each gas must be within 2.0 percent of the Calibration Span or 0.5 ppmv/% absolute difference.

5.2.4.2 System Bias and Response Time

High or Mid-Level gas (whichever is closer to the stack gas concentration) will be introduced at the probe and the time required for the analyzer reading to reach 95 percent or 0.5 ppm/% (whichever was less restrictive) of the gas concentration will be recorded. The analyzer reading will be observed until it reaches a stable value, and this value will be recorded. Next, Low-Level gas will be introduced at the probe and the time required for the analyzer reading to decrease to a value within 5.0 percent or 0.5 ppm/% (whichever was less restrictive) will be recorded. If the Low-Level gas is zero gas, the acceptable response must be 5.0 percent of the upscale gas concentration or 0.5 ppm/% (whichever was less restrictive). The analyzer reading will be observed until it reaches a stable value, and this value will be recorded. The measurement system response time and initial system bias will be determined from these data. The System Bias for each gas must be within 5.0 percent of the Calibration Span or 0.5 ppmv/% absolute difference.

5.2.4.3 Post Test System Bias Checks

High or Mid-Level gas (whichever is closer to the stack gas concentration) will be introduced at the probe. After the analyzer response is stable, the value will be recorded. Next, Low-Level gas will be introduced at the probe, and the analyzer value will be recorded once it reaches a stable response. The System Bias for each gas must be within 5.0 percent of the Calibration Span or 0.5 ppmv/% absolute difference or the data is invalidated, and the Calibration Error Test and System Bias must be repeated.

5.2.4.4 Post Test Drift Checks

The Drift between pre- and post-run System Bias must be within 3 percent of the Calibration Span or 0.5 ppmv/% absolute difference or the Calibration Error Test and System Bias must be repeated.

5.2.4.5 Interference Check

An interference check will be performed using a calibration gas with 5-15 percent CO_2. As practicable, the CO_2 concentration of the calibration gas used should be the highest expected during testing.

5.2.4.6 Stratification Check

To determine the number of sampling points, a gas stratification check will be conducted prior to initiating testing. The pollutant concentrations will be measured at twelve traverse points (as described in Method 1) or three points (16.7, 50.0 and 83.3 percent of the measurement line). Each traverse point will be sampled for a minimum of twice the system response time.

If the pollutant concentration at each traverse point do not differ more than 5% or 0.5 ppm/0.3% (whichever is less restrictive) of the average pollutant concentration, then single point sampling will be conducted during the test runs. If the pollutant concentration does not meet these specifications but differs less than 10% or 1.0 ppm/0.5% from the average concentration, then three (3) point sampling will be conducted (stacks less than 7.8 feet in diameter - 16.7, 50.0 and 83.3 percent of the measurement line; stacks greater than 7.8 feet in diameter – 0.4, 1.0, and 2.0 meters from the stack wall). If the pollutant concentration differs by more than 10% or 1.0 ppm/0.5% from the average concentration, then sampling will be conducted at a

minimum of twelve (12) traverse points. Copies of stratification check data will be included in the Quality Assurance/Quality Control Appendix of the report.

5.2.4.7 NO_x Converter Check

An NO_2 – NO converter check will be performed on the analyzer prior to initiating testing or at the completion of testing. An approximately 50 ppm nitrogen dioxide cylinder gas will be introduced directly to the NOx analyzer and the instrument response will be recorded in an electronic data sheet. The instrument response must be within +/- 10 percent of the cylinder concentration.

5.2.4.8 NO_x Converter Check (Alternate)

An NO_2 – NO converter check will be performed on the analyzer prior to initiating testing or at the completion of testing. Mid-level nitrogen oxide protocol 1 calibration gas will be mixed at a 1:1 ratio with span level protocol 1 oxygen calibration gas in a Tedlar sample bag to form NO_2 gas. The NO_2 gas will be delivered to the nitrogen oxides analyzer directly from a Tedlar sample bag. The response of the analyzer must be stable for the 30-minute duration of the test with the variation less than 2.0% at the end of the test from the maximum value of the test.

5.2.5 Method 3A CO_2 and O_2 CEMS Precision and Accuracy

Method 3A for CO_2 and O_2 include the same system bias, drift, calibration error, and interference checks as noted above for Methods 6C and 7E. If the stratification test provisions in Section 8.1.2 of Method 7E are used to reduce the number of required sampling points, the acceptance criterion for 3-point sampling will be ±0.5 percent CO_2 or O_2, and the alternative acceptance criterion for single-point sampling will be ±0.3 percent CO_2 or O_2.

5.2.6 Method 25A THC CEMS Precision and Accuracy

Method 25A for THC includes the same system response time, drift, and calibration error checks as noted above for Methods 6C and 7E. Stainless steel, or equivalent, three-hole rake type probe will be used. Sample holes shall be 4 mm (0.16-in.) in diameter or smaller and located at 16.7, 50, and 83.3 percent of the equivalent stack diameter. Alternatively, a single opening probe may be used so that a gas sample is collected from the centrally located 10 percent area of the stack cross-section.

The THC emissions standard is 10 ppm. Calibration error span gas values should be nominally 1.5 to 2.5 times the emissions standard, in this case 15 to 25 ppm. Zero and calibration drift tests ensure that bias introduced by monitor drift during the run is no greater than 3 percent of span.

Water vapor in the regeneration furnace stack gas is expected to be 30-35 percent which presents a potential system bias. During the previous PDT, use of a knockout impinger in the stack gas sampling line was necessary to eliminate the moisture interference. The condensate captured during each test run was recovered in a VOA vial and submitted for analysis via Method 8260B. If similar action is necessary for this PDT, this

same protocol will be followed. The condensate analyses will be presented and their impacts evaluated in the PDT report.

5.2.7 Method 0030 VOST Precision and Accuracy

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

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

5.2.8 Method 0010 Semivolatile Organic Sampling Precision and Accuracy

The SW-846 Method 0010 sampling train, sometimes referred to as the Modified Method 5 (MM5) sampling train, will be used to sample the stack gas for target semivolatile organic compounds (SVOC), SVOC PICs and target organochlorine pesticides (OCPs). Two Method 0010 sampling trains will be used, one for SVOCs and another for OCPs. Prior to use in the field, the XAD-2 resin traps for use in the Method 0010 sampling train are spiked with isotopically labeled versions of the semivolatile organics noted in Table 5-1. Prior to their use in the field, two of the prepared XAD-2 resin traps from the batch of traps prepared specifically for this project will be analyzed prior to field sample analysis. The precision assessment for Method 0010 sampling train requires that the RPD associated with each semivolatile analyte be $\leq$50% for the spiked semivolatile and OCP compounds. The variation of surrogate recoveries should be $\leq$40% RSD for actual Method 0010 analyses.

5.2.9 Method 23 PCDD/PCDFs, PCBs, and PAHs Sampling Precision and Accuracy

An EPA Method 23 sampling train will be used to sample the stack gas for polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/PCDFs), polychlorinated biphenyls (PCBs), and polycyclic aromatic hydrocarbons (PAHs). Prior to use in the field, the XAD-2 resin traps for use in the Method 23 sampling train are spiked with isotopically labeled versions of the PCDD/PCDFs, PCBs, and PAHs noted in Table 5-

1. Prior to their use in the field, two of the prepared XAD-2 resin traps from the batch of traps prepared specifically for this project will be analyzed prior to field sample analysis. The precision assessment for Method 23 sampling train requires that the RPD associated with each analyte be ≤30% for the spiked compounds. The variation of surrogate recoveries should be ≤30% RSD for actual Method 23 analyses.

5.2.10 POHC, Metals, and Organic Surrogate Mixture Spiking

POHC, metals, and surrogate spiking accuracy is noted in Table 5-1. Spiking rates are determined via differential weights over time. The scales are upscale and downscale calibration checked before and after testing. This check is performed using certified 50 lb weights. The accuracy standard is ±0.1 lb over the operating range, e.g., 0-500 lbs. Scale calibration records are included in the spiking report documentation. Spiking materials assay or sample analysis concentration data are used to calculate feed rates. Mass flow meter data (if used) are only used as qualitative indicators of feed rates and are not used in the feed rate calculations.

All organic compound, metals, and chlorine spiking will be performed via continuous metering of solvents and solutions. Metering of these materials will be from drums and containers on scales. The feed rates of the respective spiking materials will be determined via loss of mass per unit time. Weights will be recorded manually on log sheets at ten (10) minute intervals during each test run. The accuracy standard for the scales is ±0.1 lb as noted in Table 5-1. Net feed rates of spiked constituents will be calculated using manufacturer's assay for technical grade materials and per run sample analysis of prepared solutions. The constituent feed rates will be within the ranges specified in Table 4-2 of the PDT plan.

5.2.11 Stack Flow Instrument Certification and Corroboration

To confirm the precision and accuracy of the RF stack flowmeter:

- Approximately the week prior to the PDT, Desotec will perform Performance Specification 6 (PS-6) relative accuracy (RA) testing using EPA Method 2 as the reference method (RM) with a RA target of no greater than 20 percent per Equation 2-6 from Section 12.5 of PS-2 and average RM in the denominator.
- Daily Pre-Test Reading: Measure stack flow using EPA Method 2 and compare the calculated flow to the stack flowmeter's average readings during the sampling period. The target difference between the EPA Method 2 and average stack flow instrument readings is ≤10.0%.
- Daily Post-Test Run Data Reduction: After each test run, compare the average of the stack flow instrument HRA values during the test run to the average of the average flows measured by the four (4) concurrently operated semivolatile organic sampling trains (EPA Method 23 and three SW-846 Method 0010-variants). The target difference between the average of the HRA values measured by the stack flow instrument and the average of the average sampling train values is ≤10.0%.

Equation 2-6 from Section 12.5 of PS-2 will be used to calculate the RA, expressed as a percentage, of a set of data as follows:

$$RA = \frac{|d| + |CC|}{}$$

$$RM_{AVG}$$

Where:

|d| = Absolute value of the mean differences

|CC| = Absolute value of the confidence coefficient

RM_{AVG} = Average RM value.

If daily pre-test ≤10% corroboration cannot be achieved, the facility will enact the troubleshooting methods and Corrective Action Request (CAR) process delineated in Section 14.0 of the QAPP. If daily post-testing calculations identify discrepancies >10.0%, Desotec will utilize the least favorable (most conservative) values for relevant data needs to confirm/demonstrate compliance with the permit stack flow rate limit.

5.3 DETECTION LIMITS AND REPORTING

How the detection limits will be used in data reduction and reporting is also described in Section 11.3.3. For inorganic analyses and non-isotope dilution method organic analyses, the laboratory report will provide both the sample specific Method Detection Limit (MDL) and the Reporting Limit (RL). All non-detects for target analytes will be reported at the laboratory-determined MDL. If the analyte is detectable at some value between the MDL and RL, the detected value will be reported and flagged as estimated. For isotope dilution organic analysis methods, the non-detects for the isotope dilution methods will be determined using the method specific SW-846 definition of an estimated detection limit (EDL) without the use of empirical factors or other mathematical manipulations specific to the laboratory.

5.3.1 Process Sample Properties

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

5.3.2 Process Sample Metals

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

5.3.3 Process Sample Organics

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

5.3.4 Stack Gas VOST

Non-detect results for the volatile POHC are not expected for VOST samples due to the relative rates that the volatile POHC will be present in or will be spiked into the furnace along with spent carbon feed. Therefore, performance is not based on the POHC detection limits. Should it be necessary to demonstrate that non-detect values are appropriate, blank spike samples will be performed by the laboratory to demonstrate the recovery and reproducibility of spikes of the POHC applied to the Tenax material of each tube of a VOST tube pair.

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

5.3.5 Stack Gas PCDD/PCDFs, PCBs, and PAHs

Each PCDD/PCDF 2,3,7,8-congener and tetra- through octa- congener group, all 209 PCB congeners, and each target PAH have sample-specific EDLs. When Method 23 stack gas samples for PCDD/PCDFs, PCBs, or PAHs are non-detect for a target analyte, a sample-specific detection limit is calculated for that analyte. This is done by determining the HRGC/HRMS peak height of the noise or interferent in the expected region of the analyte signal. This value is typically multiplied by a factor of 2.5. The resulting EDL signal response value is then used in the sample calculation as if it were a detected value. The result is the estimated sample detection limit.

5.3.6 Stack Gas SVOCs and OCPs

Semivolatile organic compound (SVOC) PICs and organochlorine pesticides (OCP), will be assessed using separate (discrete) Method 0010 sampling trains. The respective sampling train XAD-2 resin traps will be pre-spiked with isotopically labeled surrogate compounds of SVOCs and OCPs. The relative accuracy of the recovery of the surrogate spikes will demonstrate the required accuracy performance.

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

5.3.7 Stack Gas Metals Samples

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

5.3.8 Stack Gas Hexavalent Chromium

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

5.3.9 Stack Gas Chloride and Particulate

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

5.3.10 Stack Gas Method 0040 VOC Samples

SW-846 Method 0040 includes collection and analysis of Tedlar bag samples of the stack gas for C_1 through C_7 hydrocarbons via gas chromatograph/flame ionization detector (GC/FID) analysis. The GC/FID instrument will be set up and calibrated in the field. Bag samples will be analyzed within seventy-two (72) hours of collection for C_1 through C_7 hydrocarbons. The GC/FID will be calibrated in accordance with Method 0040 procedures before and after analysis of the bag samples. Method blank analysis includes nitrogen Tedlar bag samples. The calibration and blank analysis results will demonstrate the required accuracy performance. Condensate samples recovered from the Method 0040 sampling will be collected in 40 ml VOA vials, topped off with deionized water (for zero head space) and submitted to the laboratory for analysis within 14 days of collection.

5.3.11 Stack Gas Method 0010 TCO/Grav Samples

A variation of the SW-846 Method 0010 sampling train [Modified Method 5 (MM5)] sampling train will be used to sample the stack gas for total semivolatile organic compounds (Boiling Points from 100°C to 300°C) and non-volatile organic compounds (Boiling Points greater than 300°C). The XAD-2 resin traps used in this sampling train do not have isotopically labeled surrogates added before their use in the field. The sample extraction process also excludes the use of isotopically labeled surrogates. The extracts of the

pooled components of the sampling train are used to determine the Total Chromatographable Organics (TCO) via GC/FID analysis in the laboratory. The marker compounds are n-heptane and n-heptadecane because their boiling points are 98°C and 302°C, respectively. The non-volatile organics are determined by a gravimetric procedure known as Grav or total gravimetric organics (TGO) from the same pooled extracts of the Method 0010 train components as the semivolatile organic components. Because there are no isotopically labeled surrogates used within the sampling and analysis regime of this method, the only accuracy measurement for this sampling method is the blank train samples prepared and analyzed in the same manner as the actual field samples. The TCO/Grav blank train results will be used to blank correct the test TCO/Grav results.

5.3.12 Particle Size Distribution

Examination of the particulate matter captured in a separate Method 5 sampling train by scanning electron microscopy (SEM), will be used to determine the stack gas for particle size distribution (PSD). Smooth surface polycarbonate filters will be used in this sampling train to facilitate the SEM analysis. SEM analysis includes particle count and relative sizing. The polycarbonate filter reagent blank will be used as the baseline (zero) for performing the SEM analysis.

5.4 COMPLETENESS

Data completeness represents the valid and usable data collected from the total number of valid tests conducted. Completeness may be expressed as a percentage and calculated based on the number of samples reaching the laboratory for analysis. The completeness objective for the test will be met (100% completeness) if three valid test runs are obtained for each test condition. Samples resulting from test runs that are judged invalid based on field performance indicators or aborted runs will not be submitted to the laboratory for analysis. Because the possibility exists that a sample *or portions of a sample* may be lost, broken, *or otherwise unusable* the data from each such individual analytical parameter may not be 100 percent complete for all test runs. The impact of any occurrence of sample loss will be assessed with regard to the objective of obtaining valid runs and will be discussed in the test report. The completeness objective of this test program is to generate sufficient data for the regulatory agencies to assess the performance of the system *relative to compliance*. A partial and/or full retest of affected test runs could result if the aforementioned issue(s) could not be resolved by the troubleshooting and resolution activities outlined in Section 14.0 of the QAPP and also if the issue(s) result in the inability to collect usable data and/or demonstrate compliance (See PDTP Section 1.8.4 and 1.8.5).

5.5 REPRESENTATIVENESS, SENSITIVITY AND COMPARABILITY

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

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

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

Table 5-1 Test Analytical Data Quality Objectives

Parameter	QC type	Precision	Accuracy
Spent Activated Carbon			
Heating value, ash, moisture, total chlorine, total sulfur, and moisture	Duplicates	≤ 10% RPD	---
Heating value, ash, moisture, total chlorine, total sulfur, and moisture	Reference Sample	---	10%
Metals	Duplicates	≤ 35% RPD	---
	Matrix Spikes	≤ 30% RPD	35%
Volatile Organics	Duplicates	≤ 30% RPD	---
	Surrogate spikes	≤ 30% RPD	Note a
Semivolatile Organics	Duplicates	≤ 35% RPD	---
	Surrogate spikes	≤ 35% RPD	Note b
Scrubber Blowdown			
Metals	Duplicates	≤ 35% RPD	---
	Matrix Spikes	≤ 30% RPD	35%
Volatile Organics	Duplicates	≤ 30% RPD	---
	Surrogate spikes	≤ 30% RPD	Note a
Semivolatile Organics	Duplicates	≤ 35% RPD	---
	Surrogate spikes	≤ 35% RPD	Note b
Method 0030 Sampling Train (VOST)			
VOST Tubes	Spiked Resin Blanks	≤ 25% RPD	75-125%
VOST Tubes	Surrogate Spikes	≤ 35% RSD	Note c
VOST Condensate	Surrogate Spikes	≤ 35% RSD	Note c
VOST Breakthrough Analysis	Breakthrough Analysis	---	Note d
Method 23 Sampling Train PCDD/PCDFs, PCBs, and PAHs			
Method 23 PCDD/PCDFs and PAHs	Spiked Resin Blanks	≤ 25% RPD	75-125%
Method 23 PCDD/PCDFs	PCDD/PCDF C^{13} Labeled Sampling Surrogate Spike	≤ 35% RSD	Note e
	PCDD/PCDF C^{13} Isotope Dilution Internal Standard Spikes	---	Note f
	PCDD/PCDF C^{13} Recovery Standard Spikes	---	Note g
Method 23 PAHs	PAH C^{13} Labeled Sampling Surrogate Spike	≤ 35% RSD	Note h
	PAH C^{13} Isotope Dilution Internal Standard Spikes	---	Note i
	PAH C^{13} Recovery Standard Spikes	---	Note j
Method 23 PCBs	PCB C^{13} Labeled Pre-Sampling Surrogate Spike	≤ 35% RSD	Note k
	PCB C^{13} Extraction Isotope Internal Dilution Standard Spikes	---	Note l
	PCB C^{13} Surrogate Standard Spikes	---	Note m
	PCB C^{13} Analysis Internal Standard Spikes	---	Note n
Method 0010 Sampling Train SVOCs			
Method 0010 SVOCs	Spiked Resin Blanks	≤ 25% RPD	75-125%

Table 5-1 Test Analytical Data Quality Objectives

Parameter	QC type	Precision	Accuracy
Method 0010 SVOCs(cont'd)	Semivolatile Sampling Surrogate Spike	≤ 35% RSD	Note o
	Semivolatile Surrogate Spikes	≤ 35% RSD	Note p
Method 0010 Sampling Train OCPs			
Method 0010 OCPs	Spiked Resin Blanks	≤ 25% RPD	75-125%
	OCP Sampling Surrogate Spike	≤ 35% RSD	Note q
	OCP Surrogate Spikes	≤ 35% RSD	Note r
Method 0010 Sampling Train, TCO/Grav			
Method 0010 Chromatographable Organics	Marker Compound Spikes: n-hexane and n-heptadecane	≤ 35% RPD	50-150% (Note s)
Method 0010 Gravimetric	Replicate Weighings	± 0.5 mg	± 0.5 mg
Method 0040 Sampling Train, C_1-C_7 Hydrocarbons			
Tedlar Bag Samples	Duplicates	20%	---
	Field Spikes	30%	80-120%
Condensate Samples	C_8 Surrogate Spikes	30%	50-150%
Method 5 Sampling Train, Particulate			
Particulate Matter	Replicate Weighings	± 0.5 mg	± 0.5 mg
Method 26A Sampling Train, HCl/Cl_2			
Hydrogen Chloride/Chlorine	Duplicates	≤ 35% RPD	---
	Matrix Spikes	≤ 35% RPD	30%
	Reference Sample	---	10%
Method 29 Sampling Train, Multi-Metals			
Method 29 Metals	Duplicates	≤ 35% RPD	---
	Matrix Spike and Post-Digestion Spikes	≤ 35% RPD	---
	Reference Sample	---	70-130%
Method 0061 Sampling Train, Hexavalent Chromium			
Method 0061 Hexavalent Chromium	Field Spikes	≤ 35% RPD	70-130%
	Matrix Spikes	≤ 30% RPD	75-125%
	Duplicates	≤ 35% RPD	---
	Reference Sample	---	70-130%
Method 5 Sampling Train, Particle Size Distribution			
Particle Count/Size	Duplicates	≤ 25% RPD	---
Installed Continuous Emissions Monitors			
Carbon Monoxide	Performance Specification 4B	± 3% of Span	± 5% of Span
Oxygen	Performance Specification 4B	± 0.5% Oxygen	± 0.5% Oxygen
Temporary Continuous Emissions Monitors			
Total Hydrocarbons	EPA Method 25A	± 3% of Span	± 5% of Certified Calibration Gas
Nitrogen Oxides	EPA Method 7E	± 3% of Span	± 2% of Span
Sulfur Dioxide	EPA Method 6C	± 3% of Span	± 2% of Span
Stack Gas Molecular Weight Analyses (Method 3A)			
Carbon Dioxide	Certified Calibration Gas	---	± 0.2% CO_2
Oxygen	Certified Calibration Gas	---	± 0.2% O_2

Table 5-1 Test Analytical Data Quality Objectives

Parameter	QC type	Precision	Accuracy
POHC, Metals, and Surrogate Spiking			
Spiking Materials Composition/Concentration	Preparation Documentation/Certified Composition/Manufacturers' Specifications	1% of expected final concentration/purity	1% of expected final concentration/purity
Scales (for differential weights approach)	Pre- & Post-Test Calibration	± 0.1 lb up- and downscale check	± 0.1 lb
Continuous Mass Flow Metering Systems (if used)	Pre- & Post-Test Calibration	± 0.1 lb	± 0.1 lb

Table 5-1 Notes:

Note a: Volatile Surrogate Compound Spike Recoveries (Method 8260)

Compound	Aqueous Matrices	Solid Matrices
1,2-Dichloroethane-d_4	73 – 136%	70 – 160%
4-Bromofluorobenzene	20 – 120%	57 – 152%
Dibromofluoromethane	76 – 121%	62 – 134%
Toluene-d_8	79 – 120%	71 – 139%

Note b: Semivolatile Surrogate Compound Spike Recoveries (Method 8270)

Compound	Aqueous Matrices	Solid Matrices
2-Fluorophenol	29-90%	35-112%
Phenol-d_5	19-134%	37-122%
Nitrobenzene-d_5	52-109%	31-112%
2-Fluorobiphenyl	56-109%	49-107%
2,4,6-Tribromophenol	40-127%	33-132%
Terphenyl-d_{14}	55-115%	29-138%

Note c: Volatile Surrogate Compound Spike Recoveries (Methods 5041 and 8260)

Compound	VOST Tubes	Condensate
4-Bromofluorobenzene	50-122%	80-120%
Dibromofluoromethane	50-134%	76-121%
1,2-Dichloroethane-d_4	50-124%	70-136%
Toluene-d_8	57-134%	79-120%

Note d: Breakthrough Analysis Criterion.
The front half (Tenax® tube) and back half (Tenax®/charcoal tube) volatile organic sampling train traps are analyzed separately to determine the possible target VOC breakthrough to the Tenax®/charcoal portion of the adsorbents. The analysis of the Tenax®/charcoal trap should show less than 30 percent of each target VOC collected on the two front Tenax® traps. Breakthrough of each target VOC to the Tenax®/charcoal above this level may cause loss of desorption efficiency and would indicate a possible negative bias in the emissions calculations. This criterion does not apply when less than 75 ηg of target VOC is detected on the Tenax®/charcoal trap.

Note e: Method 23 PAH Sampling Surrogate Compound Recoveries (Method 23)

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

Table 5-1 Notes:

Note f: Method 23 PCDD/PCDF Isotope Dilution Internal Standard Compound Recoveries (Method 23)

Compound	Target Recovery
$^{13}C_{12}$-2,3,7,8-TCDD	40 – 135%
$^{13}C_{12}$-1,2,3,7,8-PeCDD	40 – 135%
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	40 – 135%
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	40 – 135%
$^{13}C_{12}$-OCDD	40 – 135%
$^{13}C_{12}$-2,3,7,8-TCDF	40 – 135%
$^{13}C_{12}$-1,2,3,7,8-PeCDF	40 – 135%
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	40 – 135%
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	40 – 135%

Note g: Method 23 PCDD/PCDF Recovery Standard Compound Recoveries (Method 23)

Compound	Target Recovery
$^{13}C_{12}$-1,2,3,4-TCDD	40 – 135%
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	40 – 135%

Note h: Method 23 PAH Sampling Surrogate Compound Recoveries (Method 23)

Compound	Combined Analysis Target Recovery
$^{13}C_6$-Benzo(c)fluorene	70-130%
$^{13}C_{12}$-Benzo(j)fluoranthene	70-130%

Note i: Method 23 PAH Isotope Dilution Internal Standard Compound Recoveries (Method 23)

Compound	C
$^{13}C_6$-Naphthalene	20-130%
$^{13}C_6$-2-Methylnaphthalene	20-130%
$^{13}C_6$-Acenaphthylene	20-130%
$^{13}C_6$-Acenaphthene	20-130%
$^{13}C_6$-Fluorene	20-130%
$^{13}C_6$-Fluoranthene	20-130%
$^{13}C_3$-Pyrene	20-130%
$^{13}C_6$-Benzo[a]anthracene	20-130%
$^{13}C_6$-Chrysene	20-130%
$^{13}C_6$-Benzo[b]fluoranthene	20-130%
$^{13}C_6$-Benzo[k]fluoranthene	20-130%
$^{13}C_4$-Benzo[e]pyrene	20-130%
$^{13}C_4$-Benzo[a]pyrene	20-130%
Perylene-d_{12}	20-130%
$^{13}C_6$-Indeno[1,2,3-cd]pyrene	20-130%
$^{13}C_6$-Dibenz[a,h]anthracene	20-130%
$^{13}C_{12}$-Benzo[g,h,i]perylene	20-130%
$^{13}C_6$-Anthracene	20-130%
$^{13}C_6$-Phenanthrene	20-130%

Table 5-1 Notes:

Note j: Method 23 PAH Recovery Standard Compound Recoveries (Method 23)

Compound	Combined Analysis Target Recovery
d_{10}-Anthracene (Filter Recovery Standard)	70-130%

Note k: Method 23 PCB Pre-Sampling (Field) Surrogate Standard Compound Recoveries (applied to XAD-2 before sampling and front-half components before extraction)

Compound	Target Recovery
$^{13}C_{12}$-2,4'-dichlorobiphenyl	70 – 130%
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	70 – 130%
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	70 – 130%
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	70 – 130%

Note l: Method 23 PCB Pre-Extraction Internal Dilution Standard (IDS) Compound Recoveries (applied to sample fractions before extraction)

Compound	Target Recovery
$^{13}C_{12}$-2,3,3',4,5,5'-HxCB (Filter Recovery Standard)	70 – 130%
$^{13}C_{12}$-2-monochlorobiphenyl	20 – 145%
$^{13}C_{12}$-4-monochlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2'-dichlorobiphenyl	20 – 145%
$^{13}C_{12}$-4,4'-dichlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	20 – 145%
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	20 – 145%
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	20 – 145%
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,3,4,4',5-pentachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	20 – 145%
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	20 – 145%
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	20 – 145%
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	20 – 145%
$^{13}C_{12}$-decachlorobiphenyl	20 – 145%

Table 5-1 Notes:

Note m: Method 23 PCB Surrogate Standard Compound Recoveries (applied to sample fractions before extraction)

Compound	Target Recovery
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	20 – 130%
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	20 – 130%
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	20 – 130%

Note n: Method 23 PCB Pre-Analysis Internal Standard (IS) Compound Recoveries (applied to extracts before analysis)

Compound	Target Recovery
$^{13}C_{12}$-2,5-dichlorobiphenyl	S/N≥10
$^{13}C_{12}$-2,4',5-trichlorobiphenyl	S/N≥10
$^{13}C_{12}$-2,4',6-trichlorobiphenyl	S/N≥10
$^{13}C_{12}$-2,2',5,5'-tetrachlorobiphenyl	S/N≥10
$^{13}C_{12}$-2,2',4,5,5'-pentachlorobiphenyl	S/N≥10
$^{13}C_{12}$-3,3',4,5,5'-pentachlorobiphenyl	S/N≥10
$^{13}C_{12}$-2,2',3,4,4',5'-hexachlorobiphenyl	S/N≥10
$^{13}C_{12}$-2,2',3,4,4',5,5'-heptachlorobiphenyl	S/N≥10
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-octachlorobiphenyl	S/N≥10

Note o: Semivolatile Sampling Surrogate Compound Spike Recoveries (Method 8270)

Compound	Filter/Probe Rinse Target Recovery	Solid, XAD-2 Resin Target Recovery	Aqueous Target Recovery
$^{13}C_6$-Naphthalene	48-110%	48-111%	56-109%

Note q: Semivolatile Surrogate Compound Spike Recoveries (Method 8270)

Compound	Filter/Probe Rinse Target Recovery	Solid, XAD-2 Resin Target Recovery	Aqueous Target Recovery
2-Fluorobiphenyl	48-110%	48-111%	56-109%
2-Fluorophenol	37-105%	42-104%	29-90%
Nitrobenzene-d_5	43-107%	45-110%	52-109%
Phenol-d_5	48-114%	50-118%	19-134%
2,4,6-Tribromophenol	34-121%	51-125%	40-127%

Note r: Organochlorine Pesticide Sampling Surrogate Compound Spike Recoveries (MethodTO4A)

Compound	Combined Analysis Target Recovery
DCB Decachlorobiphenyl	60-120%

Note s: Organochlorine Pesticide Sampling Surrogate Compound Spike Recoveries (Method TO4A)

Compound	Combined Analysis Target Recovery
Tetrachloro-m-xylene	60-120%

Table 5-1 Notes:

Note t: Total Chromatographable Organics Surrogate Compound Spike Recoveries (Method 0010 TCO/Grav)

Compound	Combined Analysis Target Recovery
n-Heptadecane	50-150%
n-Hexane	50-150%

6.0 SAMPLING AND MONITORING PROCEDURES

6.1 GENERAL

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

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

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

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

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

Location [a]	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method [b]
Feed (1)	Spent Activated Carbon (1-Volatiles) (1-Semivolatiles) (1 – Metals) (1 - Properties) (1-Archive)	Conveyor Feed Belt	Teflon scoop 4L glass wide-mouth jar, 250 ml jar (VOA) 1L glass bottles with teflon lined lids	1 scoop per grab; 250 ml volatiles 1L semivolatiles 1L properties 1L metals 1L archive	Collect a grab sample at each 30-minute interval during each test run. Grab samples will be combined in a plastic pail to build run composite. Sample for analysis will be prepared from the homogenized composite sample at the end of the test run.	SW-846, Vol. II, Chapter 9, Section 9.3
Stack (2)	Stack gas Method 29	Port	EPA Method 29 multiple metals sampling train	Minimum 120 minutes[c,d]	Collect integrated sample for target metals and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5, and 29.
Stack (2)	Stack gas Method 0061	Port	SW-846 Method 0061 hexavalent chromium sampling train	Minimum 120 minutes[c,d]	Collect integrated samples for hexavalent chromium and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0061
Stack (2)	Stack gas Method 5/26A	Port	EPA Method 5/26A sampling train	Minimum 120 minutes[c,d]	Collect integrated sample for particulate, hydrogen chloride, and chlorine. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5, and 26A
Stack (2)	Stack gas Method0010-SV	Port	SW-846 Method 0010	Minimum 3 dry standard cubic meters[c,d]	Collect integrated sample for semivolatile organics and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010.

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

Location [a]	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method [b]
Stack (2)	Stack gas Method 0010-P	Port	SW-846 Method 0010sampling train	Minimum 3 dry standard cubic meters[c,d]	Collect integrated sample for OCPs and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010.
Stack (2)	Stack gas Method 0010-TOE	Port	SW-846 Method 0010 sampling train	Minimum 3 dry standard cubic meters[c,d]	Collect integrated samples for total semivolatile organics, total nonvolatile organics, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010; EPA TOE Guidance
Stack (2)	Stack gas Method 23	Port	EPA Method 23 sampling train	Minimum 3 hours and 2.5 dry standard cubic meters[c,d]	Collect integrated sample for PCDD/PCDFs, PCBs, and PAHs, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; EPA Method 23.
Stack (2)	Stack gas Method 0030	Port	SW-846 Method 0030 volatile organic sampling train	4 tube pairs per run; 40 minutes per tube pair. Up to 20 liters of stack gas per tube pair	Collect four pairs of sorbent tubes and stack gas condensate for volatile organics during each run.	SW846-0030 (VOST)
Stack (2)	Stack gas Method 0040	Port	SW-846 Method 0040 sampling train	25 – 50 liters	Collect representative sample through a heated sample probe and filter; through a condenser and into a Tedlar bag. Transport dried sample and condensate to GC/FID.	EPA Methods 1 through 5; SW846-0040; EPA TOE Guidance.
Stack (2)	Stack gas Method 5 for PSD analysis	Port	SEM analysis of Method 5 filter and residue	Maximum one minute per traverse point	Separate scanning electron microscopy of smooth surface polycarbonate particulate filter and desiccated/evaporated acetone rinse residue to determine particle size distribution.	EPA Methods 1 through 5; SEM particle count and relative sizing.

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

Location [a]	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method [b]
Stack (3)	Stack gas CEMS	Port	Temporary CEMS THC	Continuous	Continuously monitor stack gas for total hydrocarbons during each run	EPA Method 25A
Stack (3)	Stack Gas CEMS	Port	Temporary CEMS SO_2	Continuous	Continuously monitor stack gas for sulfur dioxide during each run	EPA Method 6C
Stack (3)	Stack Gas CEMS	Port	Temporary CEMS NOx	Continuous	Continuously monitor stack gas for nitrogen oxides during each run	EPA Method 7E
Stack (3)	Stack gas CEMS	Port	Installed CEMS CO	Continuous	Continuously monitor stack gas carbon monoxide during each run.	40 CFR 63 Subpart EEE Appendix; PS 4B
Stack (3)	Stack gas CEMS	Port	Installed CEMS O_2	Continuous	Continuously monitor stack gas oxygen during each run.	40 CFR 63 Subpart EEE Appendix; PS 4B
Scrubber blowdown (4)	Scrubber blowdown (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA ~200 ml per grab; 1L semivolatiles 1L metals 1L archive	Collect one 40 ml VOA vial at each 30 minute interval; each VOA vial will be handled as a discrete sample with syringe compositing to be performed by the analyst in the laboratory for run sample analysis. Collect a ~200 ml grab sample at each 30-minute interval during each test run. Grab samples will be combined in a 4-liter glass jug to build run composite. Samples for analyses other than VOCs will be prepared from the homogenized composite at the end of the test run.	SW-846, Vol. II, Chapter 9, Section 9.2

Notes:

a Refer to Figure 5-1 of the Performance Demonstration Test Plan.

b "SW846" refers to Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates.
"EPA Method" refers to New Source Performance Standards, Test Methods and Procedures, Appendix A, 40 CFR 60.

"PS 4B" refers to Performance Specification 4B, 40 CFR 60.

c The exact volume of gas sampled will depend on the isokinetic sampling rate.

d Isokinetic sampling trains include:

- Collecting one set of bag samples (or using CEM) for oxygen and carbon dioxide analysis to determine stack gas molecular weight (EPA Method 3A)
- Performing stack gas velocity, pressure, and temperature profile measurement for each sampling location (EPA Method 2)
- Determining the moisture content of the stack gas for each sampling train (EPA Method 4)

Table 6-2. Sample Collection Methods, Equipment, and Frequency

Sample Name	Sampling Reference Method	Sample Container	Analysis	Test	Field QC	Total Field Samples
Spent Activated Carbon	ASTM D-4057, E-300-86	1-Liter glass WM jar	SVOCs	4	--	4
		250 mL glass VOA jar	VOCs	4	--	4
		1-Liter glass WM jar	Properties	4	--	4
		1-Liter glass WM jar	Metals	4	--	4
Scrubber Blowdown	ASTM D-4057, E-300-86	1-Liter glass bottle	SVOCs	4	--	4
		40 mL VOA vials	VOCs	4 sets	--	4 sets
		1-Liter glass bottle	Metals	4	--	4
VOST Resin Tubes	SW846 Method 0030	VOST Resin Tube Pairs	VOC PICs	16	--	16
VOST Condensate		40 mL glass VOA vial	VOC PICs	4	--	4
VOST Field Blank Resin Tubes	SW846 Method 0030	VOST Resin Tube Pairs	VOC PICs	4	--	4
VOST Trip Blank Resin Tubes	SW846 Method 0030	VOST Resin Tube Pairs	VOC PICs	1	--	1
VOST Condensate Trip/Field Blank	SW846 Method 0030	40 mL glass VOA	VOC PICs	1	--	1
Method 23 Particulate Filter	EPA Method 23	Petri Dish	PCDD/PCDFs, PCBs, & PAHs	4	1-blank train	5
Method 23Front & Back Half and Impinger Acetone Rinses		500 mL glass sample bottle	PCDD/PCDFs, PCBs, & PAHs	4	1-blank train	5
Method 23Front & Back Half and Impinger Toluene Rinses		500 mL glass sample bottle	PCDD/PCDFs, PCBs, & PAHs	4	1-blank train	5
Method 23 XAD-2 Resin	EPA Method 23	Resin Trap	PCDD/PCDFs, PCBs, & PAHs	4	1-blank train	5
Method 23 Impinger Water		2-liter glass sample bottle	PCDD/PCDFs, PCBs, & PAHs	4	1-blank train	5
Method 23 Acetone Reagent Blank	EPA Method 23	250 mL glass sample bottle	PCDD/PCDFs, PCBs, & PAHs	--	1	1
Method 23 Toluene Reagent Blank	EPA Method 23	250 mL glass sample bottle	PCDD/PCDFs, PCBs, & PAHs	--	1	1
Method 0010 Particulate Filter	SW846 Method 0010 (SVOCs)	Petri Dish	SVOCs	4	1-blank train	5
Method 0010 Front Half Acetone and Methylene Chloride Rinses		250 mL glass sample bottle	SVOCs	4	1-blank train	5
Method 0010 XAD-2 Resin		Resin Trap	SVOCs	4	1-blank train	5

Table 6-2. Sample Collection Methods, Equipment, and Frequency

Sample Name	Sampling Reference Method	Sample Container	Analysis	Test	Field QC	Total Field Samples
Method 0010 Back Half Acetone and Methylene Chloride Rinses	SW846 Method 0010 (SVOCs)	250 mL glass sample bottle	SVOCs	4	1-blank train	5
Method 0010 Condensate		2-liter glass sample bottle	SVOCs	4	1-blank train	5
Method 0010 Acetone Reagent Blank		250 mL glass sample bottle	SVOCs	--	1	1
Method 0010 Methylene Chloride Reagent Blank		250 mL glass sample bottle	SVOCs	--	1	1
Method 0010 Particulate Filter	SW846 Method 0010 (OCPs)	Petri Dish	OCPs	4	1-blank train	5
Method 0010 Front Half Acetone and Methylene Chloride Rinses		250 mL glass sample bottle	OCPs	4	1-blank train	5
Method 0010 XAD-2 Resin		Resin Trap	OCPs	4	1-blank train	5
Method 0010 Back Half Acetone and Methylene Chloride Rinses		250 mL glass sample bottle	OCPs	4	1-blank train	5
Method 0010 Condensate		2-liter glass sample bottle	OCPs	4	1-blank train	5
Method 0010 Acetone Reagent Blank		250 mL glass sample bottle	OCPs	--	1	1
Method 0010 Methylene Chloride Reagent Blank		250 mL glass sample bottle	OCPs	--	1	1
Method 0010 Particulate Filter	SW846 Method 0010 (TOE)	Petri Dish	TCO/Grav	4	1-blank train	5
Method 0010 Front Half Acetone and Methylene Chloride Rinses		250 mL glass sample bottle	TCO/Grav	4	1-blank train	5
Method 0010 XAD-2 Resin		Resin Trap	TCO/Grav	4	1-blank train	5
Method 0010 Back Half Acetone and Methylene Chloride Rinses		250 mL glass sample bottle	TCO/Grav	4	1-blank train	5
Method 0010 Condensate		2-liter glass sample bottle	TCO/Grav	4	1-blank train	5
Method 0010 Acetone Reagent Blank		250 mL glass sample bottle	TCO/Grav	--	1	1
Method 0010 Methylene Chloride Reagent Blank		250 mL glass sample bottle	TCO/Grav	--	1	1
Method 0040 Tedlar Bag Samples	SW846 Method 0040	25-50 L Tedlar gas sample bag	C_1-C_7 Hydrocarbons	8	--	8

Table 6-2. Sample Collection Methods, Equipment, and Frequency

Sample Name	Sampling Reference Method	Sample Container	Analysis	Test	Field QC	Total Field Samples
Method 0040 Condensate		40 mL glass VOA	C_1-C_7 Hydrocarbons	4	--	4
Method 0040 Tedlar Bag Reagent Blank		25-50 L Tedlar gas sample bag with organic free nitrogen	C_1-C_7 Hydrocarbons	4	--	4
Method 0040 Organic Free Water Reagent Blank		40 mL glass VOA	C_1-C_7 Hydrocarbons	1	--	1
Method 29 Particulate Filter	EPA Method 29	Petri Dish	Metals and Mercury	4	1-blank train	5
Method 29 Front Half Nitric Acid Rinses		250 mL glass sample bottle	Metals and Mercury	4	1-blank train	5
Method 29 Acidified Peroxide Impingers and Rinses		2-liter glass sample bottle	Metals and Mercury	4	1-blank train	5
Method 29 Empty Impinger and Rinses		250 mL glass sample bottle	Mercury Only	4	1-blank train	5
Method 29 Acidified Permanganate Impingers and Rinses		1-liter glass sample bottle	Mercury Only	4	1-blank train	5
Method 29 Acidified Permanganate Impingers 8N HCl Rinses		250 mL glass sample bottle	Mercury Only	4	1-blank train	5
Method 29 Particulate Filter Reagent Blank		Petri Dish	Metals and Mercury	--	1	1
Method 29 Nitric Acid Reagent Blank		250 mL glass sample bottle	Metals and Mercury	--	1	1
Method 29 Acidified Peroxide Reagent Blank		250 mL glass sample bottle	Metals and Mercury	--	1	1
Method 29 Acidified Permanganate Reagent Blank		250 mL glass sample bottle	Mercury Only	--	1	1
Method 29 8N HCl Reagent Blank		250 mL glass sample bottle	Mercury Only	--	1	1
Method 5/26A Particulate Filter	EPA Method 5/26A	Petri Dish	Particulate	4	--	4
Method 5/26A Front Half Acetone Rinses		250 mL glass sample bottle	Particulate	4	--	4
Method 5/26A Sulfuric Acid Impingers and Rinses		2-liter glass or HDPE sample bottle	Chloride Ion	4	--	4
Method 5/26A Sodium Hydroxide Impingers and Rinses		1-liter glass or HDPE sample bottle	Chloride Ion	4	--	4
Method 5/26A Particulate Filter Reagent Blank		Petri Dish	Particulate	--	1	1

Table 6-2. Sample Collection Methods, Equipment, and Frequency

Sample Name	Sampling Reference Method	Sample Container	Analysis	Test	Field QC	Total Field Samples
Method 5/26A Acetone Reagent Blank		250 mL glass sample bottle	Particulate	--	1	1
Method 5/26A Deionized Water Reagent Blank		250 mL glass or HDPE sample bottle	Chloride Ion	--	1	1
Method 5/26A Sulfuric Acid Reagent Blank		250 mL glass or HDPE sample bottle	Chloride Ion	--	1	1
Method 5/26A Sodium Hydroxide Reagent Blank		250 mL glass or HDPE sample bottle	Chloride Ion	--	1	1
Method 0061 Potassium Hydroxide Impinger Solution and Rinses	SW846 Method 0061	2-liter HDPE sample bottle	Hexavalent Chromium	3	--	3
Method 0061 Potassium Hydroxide Solution Reagent Blank	SW846 Method 7199	250 mL HDPE sample bottle	Hexavalent Chromium	--	1	1
Method 0061Deionized Water Reagent Blank	SW846 Method 7199	250 mL HDPE sample bottle	Hexavalent Chromium	--	1	1
Method 0061 Potassium Hydroxide Impinger Solution Field Spike	SW846 Method 7199	250 mL HDPE sample bottle	Hexavalent Chromium	--	2	2
Method 0061 Potassium Hydroxide Reagent Field Spike	SW846 Method 7199	250 mL HDPE sample bottle	Hexavalent Chromium	--	1	1
Method 5 Particulate Filter	EPA Method 5 for PSD Analysis	Petri Dish	Particulate	4	--	4
Method 5 Front Half Acetone Rinses		250 mL glass sample bottle	Particulate	4	--	4
Method 5 Particulate Filter Reagent Blank		Petri Dish	Particulate	--	1	1
Method 5 Acetone Reagent Blank		250 mL glass sample bottle	Particulate	--	1	1
Carbon Dioxide and Oxygen	EPA Method 3A	Continuous analyzer or 25-50 L Tedlar gas sample bag	Carbon Dioxide and Oxygen by Analyzer	4	--	4
Carbon Monoxide	Performance Specification 4B	Installed Continuous Emissions Monitor	Carbon Monoxide	--	--	--
Oxygen	Performance Specification 4B	Installed Continuous Emissions Monitor	Oxygen	--	--	--
Total Hydrocarbons	EPA Method 25A	Temporary Continuous Emissions Monitor	Total Hydrocarbons as Propane Equivalents	--	--	--
Nitrogen Oxides	EPA Method 7E	Temporary Continuous Emissions Monitor	Nitrogen Oxides	--	--	--

Table 6-2. Sample Collection Methods, Equipment, and Frequency

Sample Name	Sampling Reference Method	Sample Container	Analysis	Test	Field QC	Total Field Samples
Sulfur Dioxide	EPA Method 7E	Temporary Continuous Emissions Monitor	Sulfur Dioxide	--	--	--
TOTAL FIELD SAMPLES						258

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

- Feed Samples:
  - Spent activated carbon
- Stack Gas Samples
  - SW-846 Method 0030 VOST for Volatile POHCs and PICs
  - SW-846 Method 23 for PCDD/PCDFs, PCBs, and PAHs
  - EPA Method 29 for Metals
  - EPA Method 0061 for Hexavalent Chromium
  - EPA Method 5/26A for Particulate, HCl and Cl_2
  - SW-846 Method 0010 for SVOC PICs
  - SW-846 Method 0010 for OCPs
  - SW-846 Method 0010 for TCO/Grav Organics
  - SW-846 Method 0040 for Total Volatiles
  - EPA Method 5 for scanning electron microscopy (SEM) analysis for Particle Size Distribution (PSD)
  - Temporary CEMS for THC (EPA Method 25A)
  - Temporary CEMS for SO_2 and NOx
  - Installed CEMS for Carbon Monoxide and Oxygen.
- Scrubber Blowdown Samples

Sample tracking is documented using a master sample list (MSL) with unique sample numbering applied to every sample (refer to Figure 7-1), completed sample collection forms, sample labels, completed chain of custody (COC) forms, completed request for analysis (RFAs) forms (Figures 7-2 through 7-5).

6.2 FIELD QUALITY CONTROL SAMPLES

Field QC samples will be collected during the test to provide an indication of quality assurance for the test samples. The field QC samples include: spiked resin blanks for VOST, Method 23 for PDCC/PCDF, PCB, and PAH, Method 0010 for SVOC, and Method 0010 for OCP; field and trip blanks for VOST; reagent blanks for all sampling trains; and blank trains for Method 29, Method 23, Method 0010 for SVOC and PICs, Method 0010 for OCP, and Method 0010 TCO/Grav. Table 6-2 includes the field QC samples that will be collected.

6.2.1 Spiked Resin Blanks

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

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

Two XAD-2 resin traps prepared for the Method 23 PCDD/PCDF, PCB, and PAH sampling train will be spiked with sampling surrogates and internal standards. These samples will be extracted and analyzed for PCDD/CPDFs, PCBs, and PAHs by the XAD-2 trap preparation laboratory to demonstrate the resin is free of background contamination, and to confirm that efficient surrogate recoveries are achievable.

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

6.2.2 Process Samples

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

6.2.3 Stack Gas Samples

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

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

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

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

6.2.3.1 VOST

The VOST will be used to sample stack gas for the selected volatile POHCs, target VOCs, and VOC PICs. The VOST data will be used to assess POHC DRE and volatile PIC emissions. The VOST sampling apparatus will be inspected and leak checked prior to each test run. Four pairs of VOST tubes will be used during each sampling run to sample a nominal 20 liters of stack gas per tube set (40 minutes at 0.5 liters per minute). Three of the four pairs will be analyzed. The fourth set pair will serve as an archive set in the event of sample breakage during shipping and handling. During the analysis, the stack gas Tenax and Tenax/charcoal tube sets will be analyzed separately to assess breakthrough. Field and trip blank Tenax and Tenax/charcoal tube sets may be analyzed separately or as sets.

6.2.3.2 Method 0010 for SVOCs and OCPs

Two Method 0010 sampling trains will be used to sample stack gas: 1) semivolatile organic compound (SVOC) PICs, and 2) organochlorine pesticides (OCP). During each test run, the Method 0010 sampling trains will be assembled, and leak checked. The sampling train will be operated to sample a minimum of three dry standard cubic meters of stack gas during each sampling run. The sampling time to achieve the target sample volume will depend upon the probe nozzle size and sampling rate. However, sampling rate will not exceed one dry standard cubic foot per minute. At the end of each run, the sampling train will be disassembled, and all train samples collected.

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

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

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

The third fraction consists of the condensate impinger catch and rinses. The condensate impinger catch is volumetrically or gravimetrically measured in the field, and this information is added to the stack sampling data sheet to calculate the moisture content of the stack gas. In the laboratory, the condensate impinger sample is placed in a separatory funnel and surrogate compounds for the SVOCs or OCPs are added. The liquid undergoes three separate methylene chloride extractions to assure recovery of acid and base-neutral compounds. The initial extraction is performed with no pH adjustment; the condensate is continuous liquid-liquid extracted using methylene chloride. The methylene chloride extract layer is then removed and fresh methylene chloride added to the sample. The sample is then pH adjusted to acid (pH<2) or basic (pH>11) and is then separatory funnel extracted using methylene chloride. The second methylene chloride extract layer is then removed, and fresh methylene chloride added to the separatory funnel. The methylene chloride/water separatory extraction is then repeated with the pH adjusted to either acid or basic, depending on the original adjustment. The third methylene chloride extract layer is then removed and combined with the earlier neutral and acid or basic methylene chloride extracts.

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

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

GC/MS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.

This test program includes target lists of SVOCs and OCPs for the Method 0010 sampling. Any target SVOC or OCP that is non-detect in all three analysis fractions of the Method 0010 sampling train will be presented in the PDT report at the sample-specific detection limit with an "ND" flag and a less than ("<") sign. For any of the non-target SVOC compounds tentatively identified using a library search for all SW-846 Method 8270 analytes not found during all three runs of the test, the tentatively identified compounds (TICs) will be averaged with zeros for the runs where they are not identified.

6.2.3.3 Method 23 for PCDD/PCDF, PCB, and PAH

A Method 23 sampling train will be used to sample stack gas PCDD/PCDFs, PCBs, and PAHs. During each test run, the Method 23 sampling train will be assembled and leak checked. As required by 40 CFR 63.1208(b)(1)(ii), the Method 23 sampling train will be operated a minimum of 180 minutes (3 hours) to sample a minimum of 2.5 dry standard cubic meters of stack gas during each sampling run. At the end of each run, the sampling train will be disassembled, and all train samples collected. Any PCDD/PCDF congener that is non-detect will be presented in the PDT report at the sample-specific detection limit with an "ND" flag and a less than ("<") sign. Similarly, any PCB or PAH target compound that is non-detect will be presented in the PDT report at the sample-specific detection limit with an "ND" flag and a less than ("<") sign.

The front-half, back half, probe, and impinger acetone rinses, the particulate filter, and XAD-2 resin are combined. Internal standard compounds for the PCDD/PCDFs, PCBs, and PAHs are spiked directly onto the filter before the Soxhlet extraction. The sample is then Soxhlet extracted for using methylene chloride. The methylene chloride extract is recovered. Then the front-half, back half, probe, and impinger toluene rinses are added to the Soxhlet extractor and extracted a second time for using toluene. The toluene extract is then recovered. The methylene chloride and toluene extracts are combined and then blown down. Recovery standards are then added prior to the final concentration for HRGC/HRMS analysis The concentrated extract is then split three ways: one-third for PCDD/PCDF analysis, one-third for PCB analysis, and one-third for PAH analysis.

The Method 23 impinger water (condensate) fraction is required for the PCB and PAH analysis; this fraction is not required for PCDD/PCDF. The impinger water fraction consists of the condensate impinger catch and rinses. The condensate impinger catch is volumetrically or gravimetrically measured in the field, and this information is added to the stack sampling data sheet to calculate the moisture content of the stack gas. In the laboratory, the condensate impinger sample is placed in a continuous liquid-liquid extractor. The liquid undergoes two separate methylene chloride extractions to assure recovery of acid and base-neutral compounds. The sample is pH adjusted to acid (pH<2) and continuous liquid-liquid extracted using

methylene chloride. The methylene chloride extract layer is then removed and fresh methylene chloride added to the sample. The methylene chloride/water extraction is then performed a second time with the pH adjusted to basic (pH>12). The second methylene chloride extract layer is then removed and combined with the other methylene chloride extract. The combined extracts are then blown down and split three ways: 1) one split to combine with the PCB-designated extract from the balance of the sampling train components for PAH analysis, 2) one split to combine with the PAH-designated extract from the balance of the sampling train components for PAH analysis, 3) the third split as an archive fraction.

Three types of spiking materials will be applied to the Method 23 sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the PCDD/PCDFs, PCBs, and PAHs found using the Method 23 sampling method.
- Isotope Dilution Internal Standard Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.
- Recovery Standards- Isotopically labeled compounds applied to the Soxhlet extracts just before GC/MS analysis. These compounds provide the relative response factors which are used to calculate analyte concentrations.

6.2.3.4 Method 29

An EPA Method 29 sampling train will be used to sample stack gas for the project target metals. Samples are subjected to acid digestion using nitric and hydrofluoric acid in either a Parr bomb or microwave pressure relief vessel. Non-mercury metals will be analyzed by SW-846 Method 6010 [inductively coupled argon plasma spectroscopy (ICP or ICAP)] or 6020 [inductively coupled argon plasma spectroscopy/mass spectroscopy (ICP-MS or ICAP-MS)]. Mercury will be analyzed for by SW-846 Method 7470 [cold vapor atomic absorption spectroscopy (CVAA or CVAAS)]. Accuracy and precision are measured though the use of a matrix spike and matrix spike duplicate. One blank train is collected and analyzed, and may be used to blank correct metals emission data per the methodology specified in Method 29. The six Method 29 sampling train fractions will undergo five separate analyses as follows:

- The nitric acid probe rinse and the particulate filter will be combined and digested in the laboratory as the front half composite sample and analyzed for Hg and the non-Hg target metals.
- The condensate knockout impinger (impinger 1) and the HNO_3/H_2O_2 impingers (impingers 2-3) catches will be digested in the laboratory and analyzed for Hg and the non-Hg target metals.
- The empty impinger (impinger 4) catch will be digested in the laboratory and analyzed for Hg.
- The $KMnO_4/H_2SO_4$ impinger (impingers 5-6) catches will be digested in the laboratory and analyzed for Hg.

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

6.2.3.5 Method 0061

A Method 0061 sampling train will be used to sample for hexavalent chromium. In the field, the stack samples are sparged with nitrogen and then filtered. In the laboratory, the samples are preconcentrated and analyzed directly using ion chromatography following SW846 Method 7199. Accuracy and precision are determined through the use of duplicate analysis, laboratory control samples, and matrix spikes.

6.2.3.6 EPA Method 5/26A

An EPA Method 5/26A sampling train will be used to sample for particulate, HCl, and Cl_2. The stack gas is sampled by sparging the gas through impingers containing 0.1N H_2SO_4 (acid) and 0.1N NaOH (basic) solutions in series. In the acid impinger solution, HCl gas is captured. Any Cl_2 passes through to the acid impinger and is captured in the basic impinger solution. The chloride concentrations of the acidic and basic impinger samples are analyzed separately for chloride ion, and are reported as HCl and Cl_2 catches respectively. Precision for these samples are determined through the use of duplicate analyses of calibration standards, QC samples, and field samples. Accuracy is determined by matrix spike/ matrix spike duplicate analyses. Additional matrix specific quality control is provided by separate matrix specific calibrations being analyzed prior to sample analysis.

The stack gas particulate emissions are determined by weighing the tare weighted particulate filter to determine the differential weight of the particulate collected by the Method 5/26A sampling train. Samples are dried/desiccated to a constant weight to the nearest 0.1 mg. Constant weight shall mean a difference between two consecutive weighings of no more than 0.5 mg difference or more than 1 percent of the total weight less the tare weight. This differential weight is added to the weight of the residue remaining after evaporation of the acetone probe and filter holder rinses. The reported particulate catch is the sum of the particulate filter differential weight and the probe rinse residue weight.

6.2.3.7 Method 0010 for TCO/Grav

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

In the laboratory, the TOC/Grav front and back half rinses, particulate filter and XAD-2 resin are combined and Soxhlet extracted with methylene chloride. The final pooled extract volume must be no less than 5

mL. Since the extracts for total organics determinations are analyzed by GC/FID and gravimetric techniques, no sampling surrogate, isotopically-labeled standards, or internal standards associated with GC/MS analysis (Method 8270) are added to the extractors or sample extracts. The combined methylene chloride extracts are split into four portions and used as follows:

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

6.2.3.8 SEM for PSD

Scanning electron microscopy (SEM) examination of the particulate matter captured by a Method 5 train with smooth-surface polycarbonate filter will be used to determine the stack gas for particle size distribution (PSD). Analysis is via scanning electron microscope (SEM). PSD is determined by particle counting by relative particle size. The SEM baseline (zero) is based on reagent blank polycarbonate filter analysis.

6.2.4 Blank Trains and Reagent Blanks

Blank train samples are the samples recovered from sampling trains that have been assembled and charged with all the required chemical reagents and collection media in the same manner as the sampling trains used to sample the stack gases. The sampling trains are leak checked and heated to temperature in a location near the stack. The sampling train remains sealed at the stack location for a period equivalent to the length of time the corresponding sampling train is operated during the test run. The blank train is then recovered in the same way that actual stack gas sampling trains are recovered. The recovered blank train components are labeled as blank train samples and submitted for analysis with the actual stack gas train samples. The results of the blank train samples provide an indication of possible contamination introduced to the samples by reagents, glassware, sampling environment, and sampling recovery. The blank train samples for the stack sampling trains used during this test program will be collected as summarized in Table 6-2. One blank train for each of the Method 29, Method 0010 SVOC, Method 0010 OCP, Method 23 PCDD/PCDF, PCB, and PAH, and Method 0010 TCO/Grav trains will be collected during the testing.

Reagent blanks are samples of the reagent source solvents, solutions, and other media used in stack sampling. Reagent blank samples for the Method 5-PSD, Method 5/26A, Method 29, Method 0061, Method 23 PCDD/PCDF, PCB, and PAH, Method 0010 SVOC, Method 0010 OCP, and Method 0010 TCO/Grav sampling trains used during this test program will be collected as summarized in Table 6-2.

6.2.5 Field Blanks

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

6.2.6 Trip Blanks

Trip blanks are similar to field blanks in that they are used to assess contamination resulting from sample handling and shipment. With each shipment of VOST samples from the test site to the laboratory, a pair of VOST tubes that have remained sealed as shipped from the laboratory to the field to be used as trip blanks. Additionally, a pair of volatile organic analysis (VOA) vials filled with deionized (DI) water is included with the VOST samples shipped from the test site back to the laboratory. The trip blank and DI water analyses demonstrate that the samples are not exposed to contamination during transport from the field to the laboratory. If VOCs are detected in VOST trip blanks above the reporting limit, the impacts on compliance determinations and HHERA data usability will be assessed in the quality assurance discussion section of the final test report.

Trip blanks are not required and generally unnecessary for the other emissions sampling trains, e.g., Methods 0010 and Method 23. Trip blanks are unnecessary for process samples (e.g., spent carbon) expected to contain high concentrations of possible target analytes. Process samples are stored and handled separately from emissions samples to preclude possible contamination of the emissions samples.

7.0 SAMPLE HANDLING, TRACEABILITY, AND HOLDING TIMES

7.1 SAMPLE COLLECTION CHECKLIST

Prior to the test, a master list of the samples required will be compiled by the Test Manager. This list will identify the samples by their assigned unique alphanumeric sample numbers. An example sample master sample list (MSL) is shown in Figure 7-1. As field samples are acquired and routed through the Sample Custodian, the samples will be checked off against the master list to ensure that all the appropriate samples have been taken.

DESOTEC Performance Demonstration Test — **Eurofins Environment Testing, Knoxville, TN**

Parker, AZ — **PDT MASTER SAMPLE LIST** — **31-Jan-25**

Field Sample No.	Test No.	Run No.	RFA/COC No.	Sample Source	Sample Description	Sample Container	Analytical Parameters	Analytical Laboratory	QC Analysis
R- 8001	PDT	1	001	Carbon Feed	Carbon Feed	1-Liter glass jar	Metals & Hg	Eurofins-Knox	DUP/MS/MSD
R- 8002	PDT	1	002	Carbon Feed	Carbon Feed	1-Liter glass jar	Properties	Eurofins-Knox	DUP
R- 8003	PDT	1	003	Carbon Feed	Carbon Feed	1-Liter glass jar	SVOCs	Eurofins-Knox	DUP
R- 8004	PDT	1	004	Carbon Feed	Carbon Feed	250 mL glass VOA jar	VOCs	Eurofins-Knox	DUP
R- 8005	PDT	1	005	Method 29 Train	Particulate Filter	Petri dish	Metals & Hg	Eurofins-Knox	
R- 8006	PDT	1	005	Method 29 Train	0.1 N HNO_3 Probe Rinse	250 mL amber bottle	Metals & Hg	Eurofins-Knox	
R- 8007	PDT	1	005	Method 29 Train	5% HNO_3/10% H_2O_2 Impingers	2-liter amber glass	Metals & Hg	Eurofins-Knox	
R- 8008	PDT	1	005	Method 29 Train	Empty Impinger	250 mL amber bottle	Metals & Hg	Eurofins-Knox	
R- 8009	PDT	1	005	Method 29 Train	4%$KMnO_4$/10%H_2SO_4 Impingers	1-liter amber glass	Metals & Hg	Eurofins-Knox	
R- 8010	PDT	1	005	Method 29 Train	8N HCl Impinger Rinse	250 mL amber bottle	Metals & Hg	Eurofins-Knox	
R- 8011	PDT	1	005	Method 29 Blank Train	Particulate Filter	Petri dish	Metals	Eurofins-Knox	BT
R- 8012	PDT	1	005	Method 29 Blank Train	0.1 N HNO_3 Probe Rinse	250 mL amber bottle	Metals	Eurofins-Knox	BT
R- 8013	PDT	1	005	Method 29 Blank Train	5% HNO_3/10% H_2O_2 Impingers	500 mL amber bottle	Metals	Eurofins-Knox	BT
R- 8014	PDT	1	005	Method 29 Blank Train	Empty Impinger	250 mL amber bottle	Metals	Eurofins-Knox	BT
R- 8015	PDT	1	005	Method 29 Blank Train	4%$KMnO_4$/10%H_2SO_4 Impingers	500 mL amber bottle	Metals	Eurofins-Knox	BT
R- 8016	PDT	1	005	Method 29 Blank Train	8N HCl Impinger Rinse	250 mL amber bottle	Metals	Eurofins-Knox	BT
R- 8017	PDT	1	006	Method 26A Train	Particulate Filter	Petri dish	Particulate	Eurofins-Knox	
R- 8018	PDT	1	006	Method 26A Train	Acetone Probe Rince	500 mL amber bottle	Particulate	Eurofins-Knox	
R- 8019	PDT	1	006	Method 26A Train	0.1N H_2SO_4 Impingers	2-Liter HDPE bottle	HCl	Eurofins-Knox	
R- 8020	PDT	1	006	Method 26A Train	0.5N NaOH Impingers	1-Liter HDPE bottle	Chlorine	Eurofins-Knox	
R- 8021	PDT	1	006	Method 26A Reagent Blank	Particulate Filter Reagent Blank	Petri dish	Particulate	Eurofins-Knox	RB
R- 8022	PDT	1	006	Method 26A Reagent Blank	Acetone Reagent Blank	250 mL amber bottle	Particulate	Eurofins-Knox	RB
R- 8023	PDT	1	006	Method 25A Reagent Blank	0.1N H_2SO_4 Reagent Blank	500 mL HDPE bottle	HCl	Eurofins-Knox	RB
R- 8024	PDT	1	006	Method 25A Reagent Blank	0.5N NaOH Reagent Blank	500 mL HDPE bottle	Chlorine	Eurofins-Knox	RB

Figure 7-1. Example Master Sample List

7.2 PROCESS SAMPLE COLLECTION FORMS

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

PROCESS SAMPLE DATA COLLECTION SHEET

Client:	DESOTEC	**Unit:**	Carbon Reactivation Furnace
Location:	Parker, AZ	**Sheet No.:**	1 of 1
Test Program:	PDT	**Run Start:**	0800
Stream:	Spent Carbon Feed	**Run End:**	1335
Sampler:	CEM	**Sample Frequency:**	30 minutes
Date:	17-Mar-25	**Sample Type:**	Grab/Composite
Test No.:	PDT	**Equip/Container:**	Scoop Grab / 3-gal Pail Composite
Run No.:	1	**Source:**	Carbon Feed Belt

Sample No.	Sample Time	Comment	Sample No.	Sample Time	Comment
1	0800	Run Start 0800	26	:	
2	0830		27	:	
3	0900		28	:	
4	0930		29	:	
5	1000		30	:	
6	1030		31	:	
7	1100		32	:	
8	1130		33	:	
9	1200		34	:	
10	1230		35	:	
11	1300		36	:	
12	1330	Run End 1335	37	:	
13	:		38	:	
14	:		39	:	
15	:		40	:	
16	:		41	:	
17	:		42	:	
18	:		43	:	
19	:		44	:	
20	:		45	:	
21	:		46	:	
22	:		47	:	
23	:		48	:	
24	:		49	:	
25	:		50	:	

Total No. of Grabs: 12
Comments/Notes:
Samples for analysis were prepared at the end of the test run using aliquots of the homogenized composite sample.
The balance of the composite remaining was discarded.

Figure 7-2. Example Field Sampling Record

7.3 SAMPLE LABELING

An example sample label format is presented in Figure 7-3. Each sample container will be labeled to show the source of the sample as DESOTEC; the project identification; sampler's initials; laboratory to which the sample will be shipped; an unique alphanumeric sample number; date and time; sample description; test number; and run number. If a single sample requires multiple containers, the number of the container and the total number of containers will be noted on the label.

DESOTEC Performance Demonstration Test
Parker, AZ

Sample Type:	Method 29 Filter	**Sample No.:**	**R-8005**
Test No. PDT1	**Run No.** 1	**Container(s):**	1 of 1
Analysis Required:	Metals:Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn		
Analysis Laboratory:	Eurofins Environment Testing, Knoxville, TN		
Date:	17-Mar-25	**Initials:**	MJK
Time:	1430	**Preservation:**	NA

Figure 7-3. Example Sample Label Format

Project samples will be tracked via the assigned unique alphanumeric sample numbers. The sample number will appear on the sample label, the RFA and the COC.

7.4 SAMPLE PRESERVATION

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

The location of the VOST sample holding area is of special importance. The sample containers for volatiles will be stored in a clean area separate from the sample preparation area. High concentration volatile organic samples (recovered elements of other sampling trains, spent carbon feed samples, etc.) will be segregated to prevent inadvertent contamination of the VOST samples. The VOST tube sample pairs will be preserved before and after sampling by placing them on ice in a dedicated sample cooler. To preclude contamination from solvents and process samples, VOST samples will be shipped on ice in dedicated shipping containers separate from all other test samples.

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

Parameter	Sample Name/Matrix	Sample Containers	Preservation	Maximum Holding Time
Process Semivolatile Organics	Spent Activated Carbon	Wide mouth amber glass jars with Teflon-lined caps	Chill to 4°C	14 days until extraction, 40 days after extraction
	Scrubber Blowdown	Amber glass Boston round bottles with Teflon-lined caps	Chill to 4°C	14 days until extraction, 40 days after extraction
	POHC Spike	Amber glass Boston round bottles with Teflon-lined caps	Chill to 4°C	14 days until extraction, 40 days after extraction
Process Volatile Organics	Spent Activated Carbon	Glass VOA jars with Teflon-lined caps	Chill to 4°C	14 days
	Scrubber Blowdown	Glass VOA vials with Teflon-lined caps or septum caps	Acidify; Chill to 4°C	14 days
	POHC Spike	Amber glass Boston round bottles with Teflon-lined caps	Chill to 4°C	14 days
Process Physical Properties	Spent Activated Carbon	Wide mouth amber glass jars with Teflon-lined caps	NA	180 days
Chlorine	Spent Activated Carbon	Wide mouth amber glass jars with Teflon-lined caps	NA	30 days
Metals	Spent Activated Carbon	Wide mouth amber glass jars with Teflon-lined caps	NA	180 days/28 days for Hg
Moisture Content	Spent Activated Carbon	Wide mouth amber glass jars with Teflon-lined caps	NA	180 days
Stack Gas volatile organics	Stack Gas VOST Tubes	Glass culture tube in foam cushioned plastic tube	Chill to 4°C	14 days
	Stack Gas VOST Condensate	Glass VOA vials with Teflon-lined lined caps or septum caps	Chill to 4°C	14 days

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

Parameter	Sample Name/Matrix	Sample Containers	Preservation	Maximum Holding Time
Stack Gas PCDD/PCDFs, PCBs, PAHs, SVOCs, & TCO/Grav	Stack Gas Method 0010 & 23 Filters	Glass petri dish	Chill to 4°C	14 days until extraction, 40 days after extraction
	Stack Gas Method 0010 & 23 Sorbent Tubes	Standard cartridge wrapped in aluminum foil and sealed in plastic bag	Chill to 4°C	14 days until extraction, 40 days after extraction
	Stack Gas Method 0010 & 23 Solvents	Amber glass Boston round bottles with Teflon-lined caps	Chill to 4°C	14 days until extraction, 40 days after extraction
	Stack Gas Method 0010 & 23 Condensate (for PCBs, PAHs, SVOCs, & TCO/Grav only)	Amber glass Boston round bottles with Teflon-lined caps	Chill to 4°C	14 days until extraction, 40 days after extraction
Stack Gas Method 0040 Organics	Stack Gas Method 0040 bags	Tedlar bag	Protect from sunlight	72 hours
	Stack Gas Method 0040 Condensate	Amber glass VOA vial, Teflon- lined caps or septum caps	Chill to 4°C	14 days
Stack Gas metals	Method 29 Filter	Glass petri dish	NA	180 days/28 days for Hg
	Method 29 Liquids	Amber glass Boston round bottles with Teflon-lined caps	NA	180 days/28 days for Hg
Stack Gas Particulate	Method 5 Filter	Glass petri dish	NA	180 days
	Method 5 Probe Rinses	Amber glass Boston round bottles with Teflon-lined caps	NA	180 days
Stack Gas Chloride	Method 26A acid impinger liquids	Amber glass Boston round or with Teflon-lined caps or polyethylene bottles	NA	30 days
Stack Gas Hexavalent Chromium	Method 0061 Liquids	Polyethylene bottles	pH>8.5	14 days

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

Parameter	Sample Name/Matrix	Sample Containers	Preservation	Maximum Holding Time
Stack Gas PSD	Method 5 Smooth Carbonate Filters	Glass or plastic petri dish	NA	180 days
	Method 5 Probe Rinses	Amber glass Boston round bottles with Teflon-lined caps	NA	180 days

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

XAD-2 traps for the Method 23 PCDD/PCDF, PCB, and PAH, Method 0010 SVOC, Method 0010 OCP, and Method 0010 TCO/Grav trains will be preserved before and after sampling by placing them on ice in a dedicated sample cooler. The balance of the Method 23 PCDD/PCDF and PAH, Method 0010 SVOC, Method 0010 OCP, and Method 0010 TCO/Grav train sample components will be preserved after sample collection by placing them on ice in a dedicated sample cooler. Other organic analysis samples (e.g., process samples for POHC) will be preserved after sample collection by placing them on triple bagged ice in a dedicated sample cooler.

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

7.5 SAMPLE CUSTODY

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

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

During the test, once the samples are transferred from the sampling technician to the Sample Custodian, sample custody becomes the responsibility of the Sample Custodian until the samples arrive at the analytical laboratory. When overnight couriers are utilized, the air bill will serve to document the transfer of custody from the Sample Custodian to the courier. The courier's air bill becomes part of the chain of custody (COC) record. Upon transfer of the samples from the courier to the analytical laboratory, sample custody will be maintained by the analytical laboratory performing the analyses. Samples for organic analysis (VOST, Method 23, POHC, etc.) will be kept on ice ($4 \pm 2^{o}C$) and shipped to the analytical laboratory in insulated shipping containers (ice chests). Samples not specifically requiring chilling (e.g., particulate, chloride, metals, properties, etc.) may be shipped in insulated shipping containers (ice chests) without ice.

Collected samples will be shipped from the site to the laboratory with request for analysis (RFA) and chain of custody (COC) forms. Example RFA and COC forms are presented as Figures 7-4 and 7-5, respectively. Prior to shipping any samples, the condition of the samples will be inspected and reviewed by the Sample Custodian.

Request for Analysis/Chain of Custody No. 005
DESOTEC Performance Demonstration Test
Parker, AZ

Project Description:	DESOTEC PDT
Eurofins Project No:	XXXXXXX
Client Project Mgr:	Chris McBride 865-694-7517 x3041
Eurofins Project Mgr:	Courtney Adkins 865-291-3019

Laboratory Deliverable Requirements
Analytical Due Date: 21 days from lab receipt

Data Package Due Date: 30 days from lab receipt

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

Laboratory Destination:

Eurofins Environment Testing
5815 Middlebrook Pike
Knoxville, TN 37921
(865)-291-3000

Courier: Hand deliver

Project Deliverables:
Report analytical results on R-02 Reports and in data packages. Include "Field Number", "Sample Type", "test Number", and "Run Number" on all R-02 Reports.

Holding Time Requirements:	
Metals (excluding Mercury)	180 days to analysis
Mercury	28 days to analysis

Field Sample No.		Test No.	Run No.	Sample Collection Date/ Time	Sample Container	Sample Description	Analysis Specifications	Project QC Requirements
R-	8005	PDT	1	17-Mar-25 1415	Petri dish	Method 29 Train Particulate Filter	Combine this sample with the corresponding nitric acid probe rinse, Sample No. R-8006. Perform Method 29 digestion and analyze via Method 6010C or 6020A for Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn, and Method 7470A for Hg.	
R-	8006	PDT	1	17-Mar-25 1415	250 mL amber bottle	Method 29 Train HNO_3 Probe Rinse	Combine this sample with the corresponding filter, Sample No. R-8005. Perform Method 29 digestion and analyze via Method 6010C or 6020A for Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn, and Method 7470A for Hg..	
R-	8007	PDT	1	17-Mar-25 1415	2-liter amber glass	Method 29 Train 5% HNO_3/10% H_2O_2 Impingers	Perform Method 29 digestion and analyze via Method 6010C or 6020A for Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn, and Method 7470A for Hg..	

Figure 7-4. Example Request for Analysis Form

Request for Analysis/Chain of Custody No. 005
DESOTEC Performance Demonstration Test
Parker, AZ

Sample Receipt Log and Condition of the Samples Upon Receipt

Please fill in the following information:		Comments
		(Please write "NONE" if no comment is applicable.)
(1)	Record the identities of any samples that were listed on the Request for Analysis form but were not found in the sample shipment	
(2)	Record the sample shipping cooler temperature of all coolers transporting samples listed on the Request for Analysis form.	
(3)	Record any apparent sample loss or breakage.	
(4)	Record any unidentified samples transported with this shipment of samples.	
(5)	Indicate if all samples were received according to the project's required specifications (i.e, no non-conformances).	

Custody Transfer

	Name	Company	Date/Time
Relinquished by:			
Accepted by:			
Relinquished by:			
Accepted by:			
Relinquished by:			
Accepted by:			

Figure 7-5. Example Chain of Custody Form

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

A labeled temperature blank (labeled bottle or VOA vial with water) will be shipped in every container with samples requiring chilling expressly for the purpose of determining sample temperatures upon delivery to

the laboratory. **The Laboratory Project Manager or designee will, immediately upon opening the sample packaging, open the temperature blank and measure the temperature of the water inside the temperature blank using a thermometer. This temperature will be recorded on the COCs and any applicable laboratory documentation (sample receipt log).** Containers will then be secured in a location accessible only to authorized personnel. Samples for organic analysis shall be secured in refrigerated sample storage. The COC forms are used specifically to track the samples. To provide specific instructions to the analysts, the RFAs will accompany the respective COCs.

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

7.6 REQUEST FOR ANALYSIS/CHAIN OF CUSTODY

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

8.0 SPECIFIC CALIBRATION PROCEDURES AND FREQUENCY

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

The calibration procedures are separated into groups according to the personnel who will perform them. DESOTEC operations personnel will calibrate the process instruments. Stack sampling equipment will be calibrated by the stack sampling contractor and analytical instruments will be calibrated by the contracted laboratory personnel. The calibration procedures for process instrumentation stack gas sampling, and laboratory analytical instruments are described in the following subsections.

8.1 PROCESS INSTRUMENTATION

Prior to the start of testing, the parameter continuous monitoring system (CMS) (thermocouples, flow meters, pressure transducers, etc.) and the continuous emission monitoring system (CEMS) (installed CO and O_2 monitors) will be calibrated in accordance with the facility standard operating procedures.

During testing, the installed carbon monoxide and oxygen CEMS, the monitors will be calibrated daily. The zero and span checks will be considered a verification of the data quality from these monitors.

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

8.2 STACK SAMPLING EQUIPMENT

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

Calibrations are designed to include a minimum of five (5) measurement points evenly spaced over the range. This practice minimizes the probability that false assumptions of calibration linearity will be made. In addition, it is common practice to select, when practical, at least one calibration value approximating the levels anticipated in the actual measurement. Typically, calibration frequency is dictated by the need to demonstrate the stability of the calibration value over the course of measurements. Calibrations are made both pre- and post-test to accomplish the demonstration of stability.

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

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

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

8.2.1 Pitot Tubes

All Type S pitot tubes, whether separate or attached to a sampling probe, are inspected in accordance with the geometry standards contained in EPA Method 2. All Type S pitot tubes >3/8 inches are calibrated over an eight-point range with a wind tunnel. A calibration coefficient is calculated for each pitot tube. Each pitot tube is inspected visually upon return from the field. If a visual inspection indicates damage or raises doubt that the pitot remains in accordance with the EPA geometry standards, the pitot tube is first calibrated, then repaired and recalibrated. The acceptance limits are listed in Table 8-1.

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

Table 8-1. Sampling Equipment Calibration Requirements

Stack Gas Parameter	Quality Parameter	Method of Determination	Frequency	Criteria
Gas Flow	Pitot tube angle & dimensions	Measurements with a vernier micrometer and angle indicator or wind tunnel	Post-test	To specifications in EPA Method 2
	Barometer	Calibrated vs. lab Hg-in-glass barometer	Pre-test	Within 0.2 in. Hg
	Stack gas thermocouple	Calibrated vs. ASTM Hg-in-glass thermometer	Pre-test & Post-test	Within 1.5% as R
Isokinetic Sampling Trains	Dry gas meter	Calibrated against a reference test meter	Pre-test & Post-test	Y within 0.05 of pre-test Y; H@ within 0.15 of pre-test
	Probe nozzle	Measurements with vernier micrometer to 0.001 in.	On-site Pre-test	Maximum difference in any two dimensions within 0.004 in.
	Dry gas meter thermocouples	Calibrated vs. ASTM Hg-in-glass thermometer	Post-test	Within 5 degrees F
	Triple beam balance	Calibrated vs. standard weights	Post-test	Within 0.5g

8.2.2 Differential Pressure Gauges

Some meter consoles are equipped with an inclined-vertical manometers. Fluid manometers do not require calibration other than leak-checks. Manometers are leak-checked in the field prior to each test series and again upon return from the field.

8.2.3 Digital Temperature Indicator

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

8.2.4 Dry Gas Meter and Orifice

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

8.2.4.1 Dry Gas Meter

Each metering system receives a full calibration at the time of purchase and as required by Method 5. Upon request, a post-test calibration can be performed after each field use. If the calibration factor deviates by less than five percent from the initial value, the test data are acceptable. If it deviates by more than 5%,

the meter is recalibrated and the meter coefficient (initial or recalibrated) that yields the lowest sample volume for the test runs is used.

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

8.2.4.2 Orifice

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

8.2.5 Barometer

Each field barometer is adjusted before each test series to agree within ±0.1 inches of a reference aneroid barometer. The reference barometer is checked against the station pressure value (corrected for elevation difference) reported by the National Weather Service.

8.3 LABORATORY ANALYTICAL EQUIPMENT

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

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

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

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

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Semivolatile organics GC/MS (SW846 8270)	Initial calibration	5 standards bracketing expected concentrations. Critical level should be at least 10 times higher than lowest standard	Prior to sample analysis	Variability of average RRF less than or equal to 30% RSD for CCCs. SPCCs greater than or equal to 0.05.	Recalibrate
	Continuing calibration	Midlevel standard	Prior to sample analysis, then every 12 hours or after sample set	RRF for CCCs within 30% of initial calibration average RRF. SPCCs greater than or equal to 0.05.	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check.
	Consistency in chromatography	For MS methods, monitor internal standard retention time and area. For non-MS methods, monitor retention time window for compounds of interest.	Every sample, standard, and blank	Retention time within 30 seconds of last calibration check. Area within -50 to +100% of last calibration check	Perform calibration standard check. Reanalyze sample if possible, or flag data.
	Calibration check	Analysis of independent calibration check standard	In association with each initial calibration	Within 3 std. deviations of historical mean (laboratory specific)	Recalibrate and recheck.
	Method Blank	Analysis of blank	Analyze one with each analytical batch	Results less than method detection limit	Flag data and discuss in the case narrative.
Organochlorine Pesticides (EPA TO4A)	Initial calibration	5 standards bracketing expected concentrations. Critical level should be at least 10 times higher than lowest standard	Prior to sample analysis	Mean CF %RSD $\leq$ 20%	Recalibrate
	Continuing calibration	Midlevel standard	Prior to sample analysis, then every 12 hours or after sample set	CF for each analyte $\leq$ 15%D from the mean CF for the Initial calibration.	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check.

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

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Organochlorine Pesticides (cont'd)	Consistency in chromatography	For MS methods, monitor internal standard retention time and area. For non-MS methods, monitor retention time window for compounds of interest.	Every sample, standard, and blank	Retention time within 30 seconds of last calibration check. Area within -50 to +100% of last calibration check	Perform calibration standard check. Reanalyze sample if possible, or flag data.
	Calibration check	Analysis of independent calibration check standard	In association with each initial calibration	Within 3 std. deviations of historical mean (laboratory specific)	Recalibrate and recheck.
	Method Blank	Analysis of blank	Analyze one with each analytical batch	Results less than method detection limit	Flag data and discuss in the case narrative.
PCDD/PCDF, PCB, and PAH by High Res. GC/MS (EPA Method 23)	Initial Calibration	All five high resolution concentration calibration solutions must be used for the initial calibration	Prior to sample analysis	The %RSD for the mean RRF from the 17 unlabeled standards must not exceed $\pm$ 20%, and those for the 9 labeled reference compounds must not exceed $\pm$ 30%.	Recalibrate
	Continuing Calibration	Midlevel standard	At the beginning and end of each 12-hour shift	RFs must be within $\pm$ 20% of the initial calibration mean RRF for unlabeled standards and $\pm$ 30% for labeled standards	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check.
	Retention time window verification and GC column performance	Monitor retention times	Start of each 12-hour shift	Compliance with Section 8.2.1 of Method 8290	Correct according to method
	Method Blank	Analysis of blanks	Analyze one with each analytical batch	Results less than method detection limit	Flag data as discussed in case narrative

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

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Total Volatiles (Method 0040)	Initial calibration	Minimum of 3 standards bracketing expected concentrations analyzed in duplicate	Prior to sample analysis	Laboratory specific	N/A
	Daily Calibration check	Midlevel standard	At least one per day	± 10% of true value	Reanalyze standard and if necessary, recalibrate system
	Blanks	Tedlar bag blanks and water blanks	One with each sample	Less than 20% of field sample	Evaluate for possible blank correction
TCO (Method 0010)	Initial calibration	Minimum of 3 standards	Prior to sample analysis	Correlation coefficient ≤ 0.97	Recalibrate
	Daily Calibration check	Midlevel standard	At least one per day	± 15% of true value	Reanalyze standard and if necessary, recalibrate system
	Method Blank	Analysis of blanks	One with each batch	< 20% of sample results	Evaluate for possible contamination
Metals by ICP or AAS (SW846 6010 or 6020 ICP and SW846 7470/7471 CVAA)	Initial calibration	Multiple standards (AAS) or 1 standard (ICP) and a calibration blank, bracketing the expected concentrations. Critical level should be at least twice the lowest calibration standard.	Prior to sample analysis	Correlation coefficient of linear plot >0.995 (AAS). Not applicable for ICP.	Recalibrate
	Reagent blank	Analysis of blank	After every 10 samples and at end of analysis	Less than instrument detection limit (IDL)	Reanalyze if greater than the reporting limit and discuss in case narrative if greater than the IDL
	Calibration check	Analysis of independent calibration check standard	Once after initial calibration	90 - 110% of theoretical value	Reanalyze and recalibrate, if necessary
	Serial dilution	Analysis of serial dilution (DF=5)	Once per matrix for high level analytes (ICP only)	90 - 110% of undiluted sample value (ICP samples > 50 times the IDL)	Flag data; discuss in case narrative

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

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Metals by ICP or AAS (cont'd)	Post digestion spike	Analysis of post digest spike, spiked at 2 to 5 times the original sample value	Each sample analyzed by GFAAS One sample per test for ICP	85 - 115% of theoretical value	Flag data; discuss in case narrative
	Calibration accuracy (ICP only)	Reanalysis of high-level standard	After every initial calibration	90 - 110% of theoretical value	Recalibrate and recheck
	Interference check (ICP only)	Analysis of interference sample	After every initial calibration and at the end of each run	80 - 120% of theoretical value	Recalibrate and recheck
	Continuing calibration	Midlevel standard and blank	Beginning and end of each analysis period and after every 10 samples	AAS - Midlevel standard 80 - 120% of theoretical value; blank <50% of lowest calibration standard. ICP - Midlevel standard 90 - 110% of theoretical value; blank <50% of lowest calibration standard or within 3 SD of average blank.	Identify and correct problems; reanalyze samples run since last acceptable continuing calibration check.
Hexavalent chromium by ion chromatography (SW846 7199)	Initial calibration	≥4 standards bracketing expected concentrations	Prior to and following sample analysis	RPD of response of the two standards at each concentration <10% RPD. Also, the average of the standards must yield a relative accuracy of 93 - 107% when calculated versus the linear regression curve.	Reanalyze calibration standards and samples.
	Calibration check or LCS	Analyze independent check standard.	Once per test	90 - 110% of true value	Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze.
	Sample response	Analyze each sample in duplicate	Every sample	<10% RPD of instrument response	Reanalyze samples

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

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Particulate and Gravimetric (EPA Method 5)	Calibration check	Calibration of balance with standardized weights	Prior to analysis, between each group of sample weighings, and at the end of each day.	99 – 101% of theoretical value	Recalibrate and recheck sample weights.
Chloride and Sulfate by Ion Chromatography (SW864 9056 and ASTM D-129)	Initial Calibration	4 standards bracketing expected concentrations Note: Separate calibrations are required for the acid and alkaline samples	Prior to sample analysis	Linear correlation coefficient >0.995	Recalibrate
	Retention time check for ion identification	Determine average retention time for ions of interest or relative retention time of several ions for every calibration curve	Prior to sample analysis	Average Retention Time - Sample identification is positive if results are within retention time window of standards Relative Retention Time – Sample identification is positive if results are within 3 SD of average RRT	Ions of interest are not present if criteria are not met.
Chloride and Sulfate by Ion Chromatography (cont'd)	Control check sample	Midlevel independent standard analyzed in duplicate	Beginning and end of each analysis period and after every 10 samples	90 – 110% of theoretical value	Repeat calibration check. If second check fails criteria, regenerate analytical system and reanalyze all samples since last acceptable calibration check.
	Reagent blank (ICB and CCBs)	Analysis of blanks	Immediately following the ICV and following each CCV.	Less than 1 mg/L	Contamination source must be found and corrected. All samples analyzed since the last acceptable CCB must be reanalyzed.
	Reagent blank (ICB and CCBs)	Analysis of blanks	Immediately following the ICV and following each CCV.	Less than 1 mg/L	Contamination source must be found and corrected. All samples analyzed since the last acceptable CCB must be reanalyzed.

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

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Heat Content (Btu) (ASTM D-5865)	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	LCS/LCSD analysis	Once per sample batch (maximum 20 samples)	Accuracy: %Recovery: 98 – 102%, Precision: RPD < 2%	Retest. If the retest fails, correct the cause and retest all samples.
	Duplicate Analyses	Duplicate analysis	Once prior to sample batch analysis (maximum 20 samples), minimum of 1 per set of test samples	Precision: RPD < 10%	(1) Flag data, (2) Discuss in report narrative
Percent Ash (ASTM D-482)	Continuing Calibration Blank (CCB) or Laboratory Method Blank (LMB)	CCB/LMB analysis	Once per sample batch (maximum 20 samples)	Concentrations: Result < Reporting Limit (RL) or < 0.1 sample result	Return all samples to muffle furnace and re-fire them. Reweigh after cooling.
	Laboratory Control Sample (LCS)	LCS analysis	Once per sample batch (maximum 20 samples)	Accuracy: %Recovery: 90 – 110%	Refire all samples and reanalyze if recovery exceeds upper limit.
	Laboratory Control Sample Duplicate (LCSD)	LCSD analysis	Once per sample batch (maximum 20 samples)	Accuracy: %Recovery: 90 – 110%, Precision: RPD ≤ 10%	Refire all samples and reanalyze. If RPD value continues to exceed 10%, reanalyze all samples.
	Duplicate Analyses	Duplicate analysis	Once prior to sample batch analysis (maximum 20 samples), minimum of 1 per set of test samples	Precision: RPD ≤ 10%	(1) Flag data (2) Discuss results in report narrative
Moisture Analysis (ASTM D-3173 or D-4928)	Laboratory Control Sample (LCS)	LCS analysis	Once per sample batch (maximum 20 samples)	Accuracy: %Recovery: 90 – 110%	Refire all samples and reanalyze if recovery exceeds upper limit.

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

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
	Duplicate Analyses	Duplicate analysis	Once per sample batch (maximum 20 samples)	Precision: RPD ≤ 35%	(1) Repeat analysis, (2) Flag data
Ultimate Analysis (ASTM D-3176)	Duplicate Analyses	Duplicate analysis	Once per sample batch (maximum 20 samples)	Precision: RPD ≤ 35%	(1) Repeat analysis, (2) Flag data

The following is a summary of the Eurofins Environment Testing, Knoxville, Tennessee (Eurofins) Laboratory standard operating procedures (SOPs). The revision numbers and dates noted are current as of the publication date of this QAPP. The Eurofins-Knoxville laboratory is National Environmental Laboratory Accreditation Program (NELAP) accredited for all the analyses associated with this test program. The Eurofins SOPs comply with the corresponding EPA, SW846, and ASTM methods' QC checks, frequencies, acceptance criteria, and corrective actions.

Table 8-3. Related Eurofins Analysis Standard Operating Procedures SOPs

Eurofins SOP No.	Revision/Date	Title
KNOX-GC-0010	Rev. 14, 20-Sep-23	Analysis of Extractable Semivolatile and Non-Volatile Organics by Gas Chromatography and Gravimetric Analysis
KNOX-GC-0016	Rev. 6, 13-Dec-22	Analysis of Method 0040 Condensates by Gas Chromatography for Total Unspeciated Mass in the Volatile Range
KNOX-ID-0004	Rev. 23, 19-Jan-23	Analysis of Polychlorinated Dioxins/Furans by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) Based on Methods 8290, 8290A, 1613B, 23, 0023A, and TO-9A
KNOX-ID-0013	Rev. 21, 28-Jun-23	Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HGRMS
KNOX-ID-0016	Rev. 14, 22-Mar-22 Current / under review	Isotope Dilution Analysis of Selected Semivolatile Organic Compounds and Alkylated PAHs by Gas Chromatography/Mass Spectrometry-Selected Ion Monitoring (GC/MS-SIM)
KNOX-IP-0004	Rev. 9 26-Jan-23	Acid Digestion of Soils, SW846 Method 3050B
KNOX-MS-0011	Rev. 15, 07-Dec-23	VOST Analysis
KNOX-MS-0015	Rev. 20, 08-Mar-22 Current / under review	Waste sample VOCs Determination of Volatile Organics By GC/MS Based on Method 8260B
KNOX-MS-0024	Rev. 6, 16-Nov-22	Waste sample SVOCs GC/MS Analysis Based on Method 8270D
KNOX-MT-0006	Rev. 18, 24-Feb-23	Multi-Metals (MMT) Sampling Train Preparation
KNOX-MT-0007	Rev. 18 28-Sep-22	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW-846 Method 6010B and 6010C
KNOX-MT-0009	Rev. 16, 12-Jan-23	Preparation and Analysis of Mercury in Aqueous Samples or Digestates by Cold Vapor Atomic Absorption, SW-846 7470A
KNOX-MT-0010 HG-SOP71019	Rev. 14, 20-Jan-22 22-Apr-24	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, SW-846 7471A, 7471B and EPA Method 245.5
KNOX-OP-0010	Rev. 3, 30-Sep-19	Method 0023A and Method 0010 Sampling Train Pre-Sampling Preparation and Sample Extraction Procedure (Includes TO-09A Sampling Components)
KNOX-OP-0011	Rev. 20, 14-Dec-23	Extraction and Cleanup of Organic Compounds from Waters, Soils, Solids, Sediments, Tissue and Wastes Based on SW-846 3500 and 3600 Methods
KNOX-OP-0016	Rev. 9, 03-Apr-23	Extraction of Method 0010 TCO/GRAV Sampling Trains
KNOX-WC-0003	Rev. 10, 30-Jun-23	Analysis of Hexavalent Chromium Based on SW-846 Methods 0061/7199
KNOX-WC-0005	Rev. 18, 20-Jun-23	Anion Analysis by Ion Chromatography
KNOX-WC-0006	Rev. 14, 08-Sep-22	Sampling Train Particulate Determination
KNOX-WC-0010 WC Phys Test-SOP71021	Rev. 8, 20-Apr-21 Rev. 9, 12-Apr-24	Heat of Combustion (Gross Calorific Value)
KNOX-WC-0013	Rev. 11 07-Jul-20 Current / under review	Ash Content or Loss-on-Ignition (LOI) Determination
KNOX-WC-0016	Rev. 11, 20-Jun-23	Sample Preparation for Total Halogen Determination

9.0 ANALYTICAL PROCEDURES

Analytical procedures and methods are summarized in Table 9-1. Tables 9-2 and 9-3 represent the volatile and semivolatile products of incomplete combustion (PICs) which will be targeted during the analysis of the test samples. These lists represent the target compound list (TCL) to be used by the laboratory. In addition to the TCL compounds, non-TCL peaks greater than 10% of the nearest internal standard will be tentatively identified using a library search for all SW-846 Method 8260 and 8270 analyses. Table 9-4 lists the target metal analytes by SW-846 Methods 6010 or 6020, and 7470/7471. Table 9-5 lists the target PAH analytes. Table 9-6 lists target OCP analytes. Table 9-7 lists the lists the target PCDD/PCDF analytes. Table 9-8 lists the target PCB analytes.

All analyses will be performed by a laboratory qualified in the categories of sample analysis delineated in this section. Laboratory qualifications can be submitted upon request. The following is a list of the analytical reference methods for the procedures presented in Table 9-1:

- Test Methods for Evaluating Solid Waste, SW-846 (SW-846), Third Edition, November 1986 and Updates
- Sampling and Analysis Methods for Hazardous Waste Incineration, EPA 600/8-84-002.
- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series
- Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.
- Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020.
- Performance Specification 4B, Appendix B, 40 CFR 60.

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Spent Activated Carbon	Volatile Organics	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	Solvent extraction (SW846-3550)	GC/MS (SW846-8270)
	Chloride and Sulfur Content	SW846-5050	Ion chromatography (SW846-9056 & ASTM D-129)
	Total metals	Acid digestion (SW846-3050)	ICP (SW846-6010 or 6020) & CVAAS (SW846-7470 for Hg)
	Elemental	NA	ASTM D3176
Stack gas Method 0030	VOCs + TICs (tenax + tenax/charcoal tubes) (Notes 2, 3)	Thermal desorption, trap (SW846-5041A)	GC/MS (SW846-8260)
	VOCs + TICs (condensate) (Note 2)	Purge and trap	GC/MS (SW846-8260)
Stack gas Method 0040	Total VOCs	Purge and trap for condensate Direct injection for gas	GC/FID (Guidance for Total Organics, App. A and E)
Stack gas Method 0010-SV (low res analysis)	Semivolatile Organics & TICs (Note 4)	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Moisture	NA	Gravimetric (EPA Method 4)
	Temperature	NA	Thermocouple (EPA Method 2)
	Velocity	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide (Note 6)	NA	Analyzer or CEM (EPA Method 3A)

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Stack gas Method 0010-P	OCP (Note 7)	Solvent extraction (SW846-3542)	GC/ECD (TO4A)
	Moisture	NA	Gravimetric (EPA Method 4)
	Temperature	NA	Thermocouple (EPA Method 2)
	Velocity	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide (Note 6)	NA	Analyzer or CEM (EPA Method 3A)
Stack gas Method 0010-TOE	Total SVOCs	Solvent extraction (SW846-3542)	TOC GC/FID (Guidance for Total Organics, Appendix C)
	Total NVOCs	Solvent extraction (SW846-3542)	Gravimetric Method (Guidance for Total Organics, Appendix D)
	Moisture	NA	Gravimetric (EPA Method 4)
	Temperature	NA	Thermocouple (EPA Method 2)
	Velocity	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide (Note 6)	NA	Analyzer or CEM (EPA Method 3A)
Stack gas Method 23	PCDD/PDCF, PCB, & PAH (Note 5)	Solvent extraction (EPA Method 23)	HRGC/HRMS (EPA Method 23, and EPA Method 1668C)
	Moisture	NA	Gravimetric (EPA Method 4)
	Temperature	NA	Thermocouple (EPA Method 2)
	Velocity	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide (Note 6)	NA	Analyzer or CEM (EPA Method 3A)
Stack gas Method 29	Metals (Note 8)	Acid digestion (EPA Method 29)	ICP (SW846-6010 or 6020) & CVAAS (SW846-7470 for Hg)
	Moisture	NA	Gravimetric (EPA Method 4)
	Temperature	NA	Thermocouple (EPA Method 2)
	Velocity	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide (Note 6)	NA	Analyzer or CEM (EPA Method 3A)

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Stack gas Method 0061	Hexavalent chromium	NA	Ion chromatography, post-column reactor (SW846-7199)
	Moisture	NA	Gravimetric (EPA Method 4)
	Temperature	NA	Thermocouple (EPA Method 2)
	Velocity	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide (Note 6)	NA	Analyzer or CEM (EPA Method 3A)
Stack gas Method 5/26A	Hydrogen chloride/Chlorine	NA	Ion chromatography (SW846-9056)
	Particulate	NA	Gravimetric (EPA Method 5)
	Moisture	NA	Gravimetric (EPA Method 4)
	Temperature	NA	Thermocouple (EPA Method 2)
	Velocity	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide (Note 6)	NA	Analyzer or CEM (EPA Method 3A)
Stack gas Method5-PSD	Particle size distribution	NA	Scanning electron microscopic evaluation of the M5-PSD filter and acetone rinse residue.
Stack gas temporary CEMS	HC, SO_2, and NOx	NA	Extractive Analyzers, EPA Methods 25A, 6C, and 7E
Stack gas Installed CEMs	Carbon Monoxide (Note 9)	NA	Extractive Analyzers, 40 CFR 63 Appendix
	Oxygen (Note 9)	NA	Extractive Gas Analyzers, 40 CFR 63 Appendix

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Scrubber blowdown	Volatile Organics	Purge & Trap (SW846-5030)	GC/MS (SW846-8260)
	Semivolatile Organics	Liquid-liquid extraction (SW846-3520)	GC/MS (SW846-8270)
	Total metals	Acid digestion (SW846-3050/)	ICP (SW846-6010 or 6020) & CVAAS (SW846-7470 for Hg)

Note 1: "ASTM" refers to American Society for Testing and Materials, Annual Book of ASTM Standards, Annual Series.
"SW846" refers to Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and updates.
"EPA Methods" (Methods 1 through 5 and 23) refer to New Source Performance Standards, Test Methods and Procedures,, App. A, 40CFR 60.
"Guidance for Total Organics" refers to EPA/600/R-96/036, March, 1996.

Note 2: Volatile Target Compounds as listed in this Test Plan, plus tentatively identified compounds.

Note 3: During each sampling run, 4 pairs of VOST tubes (8 samples) will be collected, but only 3 pairs (6 samples) will be analyzed. The extra tube pair provides a contingency in case of breakage or other event that could require analysis of the extra tube pair. Analysis of each tube in each tube pair will be conducted separately.

Note 4: Semivolatile Target Compounds as listed in this Test Plan, plus tentatively identified compounds.

Note 5: Polycyclic Aromatic Hydrocarbon (PAH) and polychlorinated biphenyl (PCB) target compounds as listed in this Plan

Note 6: One set of gas bag samples collected during each stack traverse for analyzer analysis, or CEM (Method 3A).

Note 7: Organochlorinated pesticide (OCP) target compounds as listed in this Test Plan.

Note 8: Metal Target Compounds as listed in this Test Plan.

Note 9: Installed CEMs sampling and analysis is continuous during each run.

Table 9-2. Stack Gas Volatile Organic Target Analytes

Volatiles	CAS Number
Acetone	67-64-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromodichloromethane	75-27-4
Bromochloromethane	74-97-5
Bromoform (Tribromomethane)	75-25-2
Bromomethane (Methyl Bromide)	74-83-9
Butylbenzene, n-	104-51-8
Butylbenzene, sec-	135-98-8
Butylbenzene, tert-	98-06-6
2-Butanone [Methyl Ethyl Ketone (MEK)]	78-93-3
Carbon Disulfide	75-15-0
Carbon Tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chlorodibromomethane	124-48-1
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane (Methyl Chloride)	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Cumene (Isopropylbenzene)	98-82-8
1,2-Dibromoethane [Ethylene dibromide (EDB)]	106-93-4
Dibromomethane	74-95-3
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
1,2-Dichloroethene (cis-)	156-59-2
1,2-Dichloroethene (trans-)	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6
1,3-Dichloropropene	542-75-6

Table 9-2. Stack Gas Volatile Organic Target Analytes

Volatiles	CAS Number
Ethylbenzene	100-41-4
2-Hexanone	591-78-6
Isopropyl toluene, p-	99-87-6
Methyl Isobutyl Ketone (4-methyl-2-pentanone) (MIBK)	108-10-1
Methylene Chloride	75-09-2
Propylbenzene, n-	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane (Freon 11)	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl Chloride	75-01-4
o-Xylene	95-47-6
m- & p- Xylenes	108-38-3 & 106-42-3
Xylenes (total)	1330-02-7

Table 9-3. Stack Gas Semivolatile Organic Target Analytes

Semivolatiles	CAS Number
Acetophenone	98-86-2
Aniline	62-53-3
Benzidine [a]	92-87-5
Benzoic Acid	65-85-0
Benzyl alcohol	100-51-6
Bis(2-chloroethoxy) methane	111-91-1
Bis-(2-chloroethyl) ether	111-44-4
Bis(2-ethylhexyl) phthalate	117-81-7
4-Bromophenyl-phenyl ether	101-55-3
Butylbenzylphthalate	85-68-7
Carbazole	86-74-8
4-Chloroaniline	106-47-8
4-Chloro-3-methylphenol	59-50-7
2-Chloronaphthalene	91-58-7
2-Chlorophenol	95-57-8
4-Chlorophenyl-phenyl ether	7005-72-3
Dibenzofuran	132-64-9
Di-n-butylphthalate	84-74-2
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
3,3'-Dichlorobenzidine	91-94-1
2,4-Dichlorophenol	120-83-2
Diethyl phthalate	84-66-2
2,4-Dimethylphenol	105-67-9
Dimethylphthalate	131-11-3
4,6-Dinitro-2-methylphenol	534-52-1
2,4-Dinitrophenol	51-28-5
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
Di-n-octyl phthalate	117-84-0
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclo-pentadiene	77-47-4

Table 9-3. Stack Gas Semivolatile Organic Target Analytes

Semivolatiles	CAS Number
Hexachloroethane	67-72-1
Isophrone	78-59-1
2-Methylphenol (o-Cresol)	95-48-7
3-/4-Methylphenol (m-/p-Cresol)	108-59-4 & 106-44-5
Cresol (total)	108-59-4, 106-44-5, & 95-48-7
2-Nitroaniline	88-74-4
3-Nitroaniline	99-09-2
4-Nitroaniline	100-01-6
Nitrobenzene	98-95-3
2-Nitrophenol	88-75-5
4-Nitrophenol	100-02-7
N-nitrosodimethylamine	62-75-9
N-Nitrosodiphenylamine	86-30-6
N-Nitroso-di-n-propylamine	621-64-7
2,2'-oxybis (1-Chloropropane)	108-60-1
Pentachlorobenzene	82-62-8
Pentachloronitrobenzene	82-68-8
Pentachlorophenol	87-86-5
Phenol	108-95-2
Pyridine	110-86-1
1,2,4-Trichlorobenzene	120-82-1
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2

Notes:
[a] Benzidine will be reported as a tentatively identified compound (TIC) if positive results are exhibited in the emissions samples.

Table 9-4. Stack Gas Metal Target Analytes

Metal	CAS Number
Aluminum	7429-90-5
Antimony	7440-36-0
Arsenic	7440-38-2
Barium	7440-39-3
Beryllium	7440-41-7
Cadmium	7440-43-9
Chromium	7440-47-3
Cobalt	7440-48-4
Copper	7440-50-8
Lead	7439-92-1
Manganese	7439-96-5
Mercury	7439-97-6
Nickel	7440-02-0
Selenium	7782-49-2
Silver	7440-22-4
Thallium	7440-28-0
Vanadium	7440-62-2
Zinc	7440-66-6

Table 9-5. Stack Gas PAH Target Analytes

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

Table 9-6. Stack Gas OCP Target Analytes

OCP	CAS Number
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Aldrin	309-00-2
α-BHC	319-84-6
β-BHC	319-85-7
γ-BHC (Lindane)	58-89-9
δ-BHC	319-86-8
α-Chlordane (cis-)	5103-71-9
δ-Chlordane (trans-)	5103-74-2
Dieldrin	60-75-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5

Table 9-7. Stack Gas Dioxin/Furan Target Analytes

Dioxin/Furan Compounds	CAS Number
2,3,7,8-TCDD	1746-01-6
Total TCDD	41903-57-5
2,3,7,8-TCDF	51207-31-9
Total TCDF	55722-27-5
1,2,3,7,8-PeCDD	40321-76-4
Total PeCDD	36088-22-9
1,2,3,7,8-PeCDF	57117-41-6
2,3,4,7,8-PeCDF	57117-31-4
Total PeCDF	30402-15-4
1,2,3,6,7,8-HxCDD	57653-85-7
1,2,3,4,7,8-HxCDD	39227-28-6
1,2,3,7,8,9-HxCDD	19408-74-3
Total HxCDD	34465-46-8
1,2,3,6,7,8-HxCDF	57117-44-9
1,2,3,4,7,8-HxCDF	70648-26-9
1,2,3,7,8,9-HxCDF	72918-21-9
2,3,4,6,7,8-HxCDF	60851-34-5
Total HxCDF	55684-94-1
1,2,3,4,6,7,8-HpCDD	35822-39-4
Total HpCDD	37871-00-4
1,2,3,4,6,7,8-HpCDF	67562-394
1,2,3,4,7,8,9-HpCDF	55673-89-7
Total HpCDF	38998-75-3
OCDD	3268-87-9
OCDF	39001-02-0

Table 5-9. Stack Gas Target PCB Target Analytes

PCB No. [1]	BZ/IUPAC No. [2]	PCB Chemical Structure Name [3]	CAS No. [4]
1	1	2-monochlorobiphenyl	2051-60-7
2	2	3-monochlorobiphenyl	2051-61-8
3	3	4-monochlorobiphenyl	2051-62-9
4	4	2,2'-dichlorobiphenyl	13029-08-8
5	5	2,3-dichlorobiphenyl	16605-91-7
6	6	2,3'-dichlorobiphenyl	25569-80-6
7	7	2,4-dichlorobiphenyl	33284-50-3
8	8	2,4'-dichlorobiphenyl	34883-43-7
9	9	2,5-dichlorobiphenyl	34883-39-1
10	10	2,6-dichlorobiphenyl	33146-45-1
11	11	3,3'-dichlorobiphenyl	2050-67-1
12	12	3,4-dichlorobiphenyl	2974-92-7
13	13	3,4'-dichlorobiphenyl	2974-90-5
14	14	3,5-dichlorobiphenyl	34883-41-5
15	15	4,4'-dichlorobiphenyl	2050-68-2
16	16	2,2',3-trichlorobiphenyl	38444-78-9
17	17	2,2',4-trichlorobiphenyl	37680-66-3
18	18	2,2',5-trichlorobiphenyl	37680-65-2
19	19	2,2',6-trichlorobiphenyl	38444-73-4
20	20	2,3,3'-trichlorobiphenyl	38444-84-7
21	21	2,3,4-trichlorobiphenyl	55702-46-0
22	22	2,3,4'-trichlorobiphenyl	38444-85-8
23	23	2,3,5-trichlorobiphenyl	55720-44-0
24	24	2,3,6-trichlorobiphenyl	55702-45-9
25	25	2,3',4-trichlorobiphenyl	55712-37-3
26	26	2,3',5-trichlorobiphenyl	38444-81-4
27	27	2,3',6-trichlorobiphenyl	38444-76-7
28	28	2,4,4'-trichlorobiphenyl	7012-37-5
29	29	2,4,5-trichlorobiphenyl	15862-07-4
30	30	2,4,6-trichlorobiphenyl	35693-92-6
31	31	2,4',5-trichlorobiphenyl	16606-02-3
32	32	2,4',6-trichlorobiphenyl	38444-77-8
33	33	2',3,4-trichlorobiphenyl (2,3',4'-trichlorobiphenyl)	38444-86-9
34	34	2',3,5-trichlorobiphenyl (2,3',5'-trichlorobiphenyl)	37680-68-5
35	35	3,3',4-trichlorobiphenyl	37680-69-6
36	36	3,3',5-trichlorobiphenyl	38444-87-0
37	37	3,4,4'-trichlorobiphenyl	38444-90-5
38	38	3,4,5-trichlorobiphenyl	53555-66-1
39	39	3,4',5-trichlorobiphenyl	38444-88-1
40	40	2,2',3,3'-tetrachlorobiphenyl	38444-93-8
41	41	2,2',3,4-tetrachlorobiphenyl	52663-59-9

Table 5-9. Stack Gas Target PCB Target Analytes

PCB No. [1]	BZ/IUPAC No. [2]	PCB Chemical Structure Name [3]	CAS No. [4]
42	42	2,2',3,4'-tetrachlorobiphenyl	36559-22-5
43	43	2,2',3,5-tetrachlorobiphenyl	70362-46-8
44	44	2,2',3,5'-tetrachlorobiphenyl	41464-39-5
45	45	2,2',3,6-tetrachlorobiphenyl	70362-45-7
46	46	2,2',3,6'-tetrachlorobiphenyl	41464-47-5
47	47	2,2',4,4'-tetrachlorobiphenyl	2437-79-8
48	48	2,2',4,5-tetrachlorobiphenyl	70362-47-9
49	49	2,2',4,5'-tetrachlorobiphenyl	41464-40-8
50	50	2,2',4,6-tetrachlorobiphenyl	62796-65-0
51	51	2,2',4,6'-tetrachlorobiphenyl	68194-04-7
52	52	2,2',5,5'-tetrachlorobiphenyl	35693-99-3
53	53	2,2',5,6'-tetrachlorobiphenyl	41464-41-9
54	54	2,2',6,6'-tetrachlorobiphenyl	15968-05-5
55	55	2,3,3',4-tetrachlorobiphenyl	74338-24-2
56	56	2,3,3',4'-tetrachlorobiphenyl	41464-43-1
57	57	2,3,3',5-tetrachlorobiphenyl	70424-67-8
58	58	2,3,3',5'-tetrachlorobiphenyl	41464-49-7
59	59	2,3,3',6-tetrachlorobiphenyl	74472-33-6
60	60	2,3,4,4'-tetrachlorobiphenyl	33025-41-1
61	61	2,3,4,5-tetrachlorobiphenyl	33284-53-6
62	62	2,3,4,6-tetrachlorobiphenyl	54230-22-7
63	63	2,3,4',5-tetrachlorobiphenyl	74472-34-7
64	64	2,3,4',6-tetrachlorobiphenyl	52663-58-8
65	65	2,3,5,6-tetrachlorobiphenyl	33284-54-7
66	66	2,3',4,4'-tetrachlorobiphenyl	32598-10-0
67	67	2,3',4,5-tetrachlorobiphenyl	73575-53-8
68	68	2,3',4,5'-tetrachlorobiphenyl	73575-52-7
69	69	2,3',4,6-tetrachlorobiphenyl	60233-24-1
70	70	2,3',4',5-tetrachlorobiphenyl	32598-11-1
71	71	2,3',4',6-tetrachlorobiphenyl	41464-46-4
72	72	2,3',5,5'-tetrachlorobiphenyl	41464-42-0
73	73	2,3',5',6-tetrachlorobiphenyl	74338-23-1
74	74	2,4,4',5-tetrachlorobiphenyl	32690-93-0
75	75	2,4,4',6-tetrachlorobiphenyl	32598-12-2
76	76	2',3,4,5-tetrachlorobiphenyl (2,3',4',5'-tetrachlorobiphenyl)	70362-48-0
77	77	3,3',4,4'-tetrachlorobiphenyl	32598-13-3
78	78	3,3',4,5-tetrachlorobiphenyl	70362-49-1
79	79	3,3',4,5'-tetrachlorobiphenyl	41464-48-6
80	80	3,3',5,5'-tetrachlorobiphenyl	33284-52-5
81	81	3,4,4',5-tetrachlorobiphenyl	70362-50-4
82	82	2,2',3,3',4-pentachlorobiphenyl	52663-62-4
83	83	2,2',3,3',5-pentachlorobiphenyl	60145-20-2
84	84	2,2',3,3',6-pentachlorobiphenyl	52663-60-2

Table 5-9. Stack Gas Target PCB Target Analytes

PCB No. [1]	BZ/IUPAC No. [2]	PCB Chemical Structure Name [3]	CAS No. [4]
85	85	2,2',3,4,4'-pentachlorobiphenyl	65510-45-4
86	86	2,2',3,4,5-pentachlorobiphenyl	55312-69-1
87	87	2,2',3,4,5'-pentachlorobiphenyl	38380-02-8
88	88	2,2',3,4,6-pentachlorobiphenyl	55215-17-3
89	89	2,2',3,4,6'-pentachlorobiphenyl	73575-57-2
90	90	2,2',3,4',5-pentachlorobiphenyl	68194-07-0
91	91	2,2',3,4',6-pentachlorobiphenyl	68194-05-8
92	92	2,2',3,5,5'-pentachlorobiphenyl	52663-61-3
93	93	2,2',3,5,6-pentachlorobiphenyl	73575-56-1
94	94	2,2',3,5,6'-pentachlorobiphenyl	73575-55-0
95	95	2,2',3,5',6-pentachlorobiphenyl	38379-99-6
96	96	2,2',3,6,6'-pentachlorobiphenyl	73575-54-9
97	97	2,2',3',4,5-pentachlorobiphenyl (2,2',3,4',5'-pentachlorobiphenyl)	41464-51-1
98	98	2,2',3',4,6-pentachlorobiphenyl (2,2',3,4',6'-pentachlorobiphenyl)	60233-25-2
99	99	2,2',4,4',5-pentachlorobiphenyl	38380-01-7
100	100	2,2',4,4',6-pentachlorobiphenyl	39485-83-1
101	101	2,2',4,5,5'-pentachlorobiphenyl	37680-73-2
102	102	2,2',4,5,6''-pentachlorobiphenyl	68194-06-9
103	103	2,2',4,5',6-pentachlorobiphenyl	60145-21-3
104	104	2,2',4,6,6'-pentachlorobiphenyl	56558-16-8
105	105	2,3,3',4,4'-pentachlorobiphenyl	32598-14-4
106	106	2,3,3',4,5-pentachlorobiphenyl	70424-69-0
107	107/109	2,3,3',4',5-pentachlorobiphenyl	70424-68-9
108	108/107	2,3,3',4,5'-pentachlorobiphenyl	70362-41-3
109	109/108	2,3,3',4,6-pentachlorobiphenyl	74472-35-8
110	110	2,3,3',4',6-pentachlorobiphenyl	38380-03-9
111	111	2,3,3',5,5'-pentachlorobiphenyl	39635-32-0
112	112	2,3,3',5,6-pentachlorobiphenyl	74472-36-9
113	113	2,3,3',5',6-pentachlorobiphenyl	68194-10-5
114	114	2,3,4,4',5-pentachlorobiphenyl	74472-37-0
115	115	2,3,4,4',6-pentachlorobiphenyl	74472-38-1
116	116	2,3,4,5,6-pentachlorobiphenyl	18259-05-7
117	117	2,3,4',5,6-pentachlorobiphenyl	68194-11-6
118	118	2,3',4,4',5-pentachlorobiphenyl	31508-00-6
119	119	2,3',4,4',6-pentachlorobiphenyl	56558-17-9
120	120	2,3',4,5,5'-pentachlorobiphenyl	68194-12-7
121	121	2,3',4,5',6-pentachlorobiphenyl	56558-18-0
122	122	2',3,3',4,5-pentachlorobiphenyl (2,3,3',4',5'-pentachlorobiphenyl)	76842-07-4
123	123	2',3,4,4',5-pentachlorobiphenyl (2,3',4,4',5'-pentachlorobiphenyl)	65510-44-3
124	124	2',3,4,5,5'-pentachlorobiphenyl	70424-70-3

Table 5-9. Stack Gas Target PCB Target Analytes

PCB No. [1]	BZ/IUPAC No. [2]	PCB Chemical Structure Name [3]	CAS No. [4]
		(2,3',4',5',5-pentachlorobiphenyl)	
125	125	2',3,4,5,6'-pentachlorobiphenyl (2,3',4',5',6-pentachlorobiphenyl)	74472-39-2
126	126	3,3',4,4',5-pentachlorobiphenyl	57465-28-8
127	127	3,3',4,5,5'-pentachlorobiphenyl	39635-33-1
128	128	2,2',3,3',4,4'-hexachlorobiphenyl	38380-07-3
129	129	2,2',3,3',4,5-hexachlorobiphenyl	55215-18-4
130	130	2,2',3,3',4,5'-hexachlorobiphenyl	52663-66-8
131	131	2,2',3,3',4,6-hexachlorobiphenyl	61798-70-7
132	132	2,2',3,3',4,6'-hexachlorobiphenyl	38380-05-1
133	133	2,2',3,3',5,5'-hexachlorobiphenyl	35694-04-3
134	134	2,2',3,3',5,6-hexachlorobiphenyl	52704-70-8
135	135	2,2',3,3',5,6'-hexachlorobiphenyl	52744-13-5
136	136	2,2',3,3',6,6'-hexachlorobiphenyl	38411-22-2
137	137	2,2',3,4,4',5-hexachlorobiphenyl	35694-06-5
138	138	2,2',3,4,4',5'-hexachlorobiphenyl	35065-28-2
139	139	2,2',3,4,4',6-hexachlorobiphenyl	56030-56-9
140	140	2,2',3,4,4',6'-hexachlorobiphenyl	59291-64-4
141	141	2,2',3,4,5,5'-hexachlorobiphenyl	52712-04-6
142	142	2,2',3,4,5,6-hexachlorobiphenyl	41411-61-4
143	143	2,2',3,4,5,6'-hexachlorobiphenyl	68194-15-0
144	144	2,2',3,4,5',6-hexachlorobiphenyl	68194-14-9
145	145	2,2',3,4,6,6'-hexachlorobiphenyl	74472-40-5
146	146	2,2',3,4',5,5'-hexachlorobiphenyl	51908-16-8
147	147	2,2',3,4',5,6-hexachlorobiphenyl	68194-13-8
148	148	2,2',3,4',5,6'-hexachlorobiphenyl	74472-41-6
149	149	2,2',3,4',5',6-hexachlorobiphenyl	38380-04-0
150	150	2,2',3,4',6,6'-hexachlorobiphenyl	68194-08-1
151	151	2,2',3,5,5',6-hexachlorobiphenyl	52663-63-5
152	152	2,2',3,5,6,6'-hexachlorobiphenyl	68194-09-2
153	153	2,2',4,4',5,5'-hexachlorobiphenyl	35065-27-1
154	154	2,2',4,4',5,6'-hexachlorobiphenyl	60145-22-4
155	155	2,2',4,4',6,6'-hexachlorobiphenyl	33979-03-2
156	156	2,3,3',4,4',5-hexachlorobiphenyl	38380-08-4
157	157	2,3,3',4,4',5'-hexachlorobiphenyl	69782-90-7
158	158	2,3,3',4,4',6-hexachlorobiphenyl	74472-42-7
159	159	2,3,3',4,5,5'-hexachlorobiphenyl	39635-35-3
160	160	2,3,3',4,5,6-hexachlorobiphenyl	41411-62-5
161	161	2,3,3',4,5',6-hexachlorobiphenyl	74472-43-8
162	162	2,3,3',4',5,5'-hexachlorobiphenyl	39635-34-2
163	163	2,3,3',4',5,6-hexachlorobiphenyl	74472-44-9
164	164	2,3,3',4',5',6-hexachlorobiphenyl	74472-45-0
165	165	2,3,3',5,5',6-hexachlorobiphenyl	74472-46-1
166	166	2,3,4,4',5,6-hexachlorobiphenyl	41411-63-6

Table 5-9. Stack Gas Target PCB Target Analytes

PCB No. [1]	BZ/IUPAC No. [2]	PCB Chemical Structure Name [3]	CAS No. [4]
167	167	2,3',4,4',5,5'-hexachlorobiphenyl	52663-72-6
168	168	2,3',4,4',5',6-hexachlorobiphenyl	59291-65-5
169	169	3,3',4,4',5,5'-hexachlorobiphenyl	32774-16-6
170	170	2,2',3,3',4,4',5-heptachlorobiphenyl	35065-30-6
171	171	2,2',3,3',4,4',6-heptachlorobiphenyl	52663-71-5
172	172	2,2',3,3',4,5,5'-heptachlorobiphenyl	52663-74-8
173	173	2,2',3,3',4,5,6-heptachlorobiphenyl	68194-16-1
174	174	2,2',3,3',4,5,6'-heptachlorobiphenyl	38411-25-5
175	175	2,2',3,3',4,5',6-heptachlorobiphenyl	40186-70-7
176	176	2,2',3,3',4,6,6'-heptachlorobiphenyl	52663-65-7
177	177	2,2',3,3',4',5,6-heptachlorobiphenyl (2,2',3,3',4,5',6'-heptachlorobiphenyl)	52663-70-4
178	178	2,2',3,3',5,5',6-heptachlorobiphenyl	52663-67-9
179	179	2,2',3,3',5,6,6'-heptachlorobiphenyl	52663-64-6
180	180	2,2',3,4,4',5,5'-heptachlorobiphenyl	35065-29-3
181	181	2,2',3,4,4',5,6-heptachlorobiphenyl	74472-47-2
182	182	2,2',3,4,4',5,6'-heptachlorobiphenyl	60145-23-5
183	183	2,2',3,4,4',5',6-heptachlorobiphenyl	52663-69-1
184	184	2,2',3,4,4',6,6'-heptachlorobiphenyl	74472-48-3
185	185	2,2',3,4,5,5',6-heptachlorobiphenyl	52712-05-7
186	186	2,2',3,4,5,6,6'-heptachlorobiphenyl	74472-49-4
187	187	2,2',3,4',5,5',6-heptachlorobiphenyl	52663-68-0
188	188	2,2',3,4',5,6,6'-heptachlorobiphenyl	74487-85-7
189	189	2,3,3',4,4',5,5'-heptachlorobiphenyl	39635-31-9
190	190	2,3,3',4,4',5,6-heptachlorobiphenyl	41411-64-7
191	191	2,3,3',4,4',5',6-heptachlorobiphenyl	74472-50-7
192	192	2,3,3',4,5,5',6-heptachlorobiphenyl	74472-51-8
193	193	2,3,3',4',5,5',6-heptachlorobiphenyl	69782-91-8
194	194	2,2',3,3',4,4',5,5'-octachlorobiphenyl	35694-08-7
195	195	2,2',3,3',4,4',5,6-octachlorobiphenyl	52663-78-2
196	196	2,2',3,3',4,4',5,6'-octachlorobiphenyl	42740-50-1
197	197	2,2',3,3',4,4',6,6'-octachlorobiphenyl	33091-17-7
198	198	2,2',3,3',4,5,5',6-octachlorobiphenyl	68194-17-2
199	201/199	2,2',3,3',4,5,5',6'-octachlorobiphenyl	52663-75-9
200	199/200	2,2',3,3',4,5,6,6'-octachlorobiphenyl	52663-73-7
201	200/201	2,2',3,3',4,5',6,6'-octachlorobiphenyl	40186-71-8
202	202	2,2',3,3',5,5',6,6'-octachlorobiphenyl	2136-99-4
203	203	2,2',3,4,4',5,5',6-octachlorobiphenyl	52663-76-0
204	204	2,2',3,4,4',5,6,6'-octachlorobiphenyl	74472-52-9
205	205	2,3,3',4,4',5,5',6-octachlorobiphenyl	74472-53-0
206	206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	40186-72-9
207	207	2,2',3,3',4,4',5,6,6'-nonachlorobiphenyl	52663-79-3
208	208	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	52663-77-1

Table 5-9. Stack Gas Target PCB Target Analytes

PCB No. [1]	BZ/IUPAC No. [2]	PCB Chemical Structure Name [3]	CAS No. [4]
209	209	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	2051-24-3

Notes:

1. The PCB congener number is from Method 1668C and Chemical Abstract Services.

2. The BZ number is from Ballschmiter and Zell (1980). The IUPAC number, when different from the BZ, follows the recommended changes to the BZ number per Schulte and Malisch (1983) and Guitart et al. (1993).

3. The chemical structure names are from Ballschmiter and Zell (1980). IUPAC nomenclature structure names are listed in parenthesis when different from the BZ name (source CAS Registry).

4. Chemical Abstract Service Registry number (source CAS Registry and 1668A Table 1).

10.0 SPECIFIC INTERNAL QUALITY CONTROL CHECKS

10.1 DEFINITIONS

The various types of QA/QC checks that may be performed as part of the test, both for sampling and analysis, are defined below. One or more of these QA/QC checks are associated with each measurement system in order to assess the compliance of the data to the DQOs established in Section 5.0. Table 10-1 (at the end of this Section) is a summary of all the sample analyses and their associated internal quality control checks associated with this test program.

Audit Sample An audit sample is a field or alternate laboratory prepared blank spike submitted to the test laboratory to assess accuracy or potential sample degradation.

Blank, Field A field blank is a sampling train or sampling component that is set-up in the field but is not used for test sampling. The field blank is used to assess background contamination that may affect the representativeness of the field samples.

Blank, Media A sample of unused sampling media analyzed to ensure the media are uncontaminated. This type of sample may also be referred to as a “reagent blank” (see below).

Blank, Method A method blank is a sample of unused media that is prepared and analyzed in the test laboratory to assess background contamination that may exist in the laboratory, on glassware, or in the analytical system.

Blank, Reagent A sample of unused reagent(s) used to demonstrate the absence of contamination in the reagents.

Blank, Spike A blank spike is a laboratory prepared sample of blank media that is spiked with a known amount of target analyte(s) used to assess the accuracy of the analytical method.

Blank, System An aliquot of uncontaminated reagent used to clean out the analytical system after high level samples have been analyzed or before analysis begins.

Blank, Trip A trip blank is an unused sample component that is shipped to the field along with the sampling equipment/media and/or returned to the laboratory without having been exposed to field conditions. If contamination is encountered in the field blank(s), the trip blank is analyzed to assess whether or not the

contamination originates in the field, is inherent in the equipment/media, or results from exposure during shipping and handling.

Breakthrough Check The result of the analysis of a secondary component (i.e., sorbent tube) in a sampling train is compared to the result of the primary component to assess whether or not the primary component has successfully captured the target analytes. If the result of the secondary component analysis is high compared to the primary component analysis, the possibility exists that the analytical results may be artificially low.

Calibration Check A standard solution from a source other than the calibration standards used to verify the integrity of an instrument's calibration.

Calibration Standards High purity compounds or mixtures of compounds used to adjust the response of an analytical instrument. The laboratory will use traceable standards and submit standard preparation logs as part of the deliverables package.

Contingency Sample An archived portion of a field sample from the same location as other field samples that is collected and held in case of breakage or QA/QC failure during the handling or analysis of the primary sample. This type of sample is sometimes referred to as an “archive sample.”

Continuing Calibration Verification A mid-point standard, from the same Calibration source as the initial calibration solution analyzed periodically to verify that calibration conditions have not drifted from the initial calibration.

Duplicate Analysis A duplicate is a sample that is split in the laboratory and prepared and analyzed twice. The results of the two analyses are compared as a measure of precision.

Duplicate Injection A second analysis of a single sample preparation. This QC test may be used to assess analytical QC failures, matrix interferences, or as a measure of analytical system precision.

Initial Calibration A series of analyses of solutions, that have known concentrations, used to establish the correspondence between the amount of an analyte present in the solution and the instrument's response across the expected analytical range of the samples. Initial calibrations also establish retention time windows for identification purposes in chromatographic methods.

Interference Check An interference check sample is analyzed, for ICP analysis only, to assess the possible error in analytical results arising from the interaction of various metals in the sample under the conditions of analysis.

Internal Standard Recovery Internal standards are non-target spikes added to samples for quantitation purposes. The percent recovery of the internal standards is checked to assess whether or not significant matrix interferences may affect the accuracy and precision of analytical results.

Performance Evaluation (PE) Sample See Audit Sample.

Proficiency Test A series of blank spikes analyzed in the test laboratory to demonstrate an analyst's ability to successfully perform the method with acceptable precision and accuracy.

Replicate One of a series of identical samples or splits of a single sample used to assess precision.

Serial Dilution The result of the analysis of a highly contaminated sample, run undiluted, is compared to the results for the same sample after serial dilution. The two results are expected to match to within method specified criteria. This test is a measure of the linearity of ICP calibration and the analysis technique.

Spike, Field See Audit Sample.

Spike, Matrix Spike of the known or controlled amount of an actual target analyte to an actual sample matrix that is then analyzed for that analyte. The percent recovery of the spiked analyte provides a measure of the matrix bias.

Surrogates Non-target or isotopically labeled analytes spiked into field samples as a measure of method efficiency and accuracy.

10.2 SPECIFIC QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

A variety of QC checks are required both in the field and in the laboratory to ensure the collection of samples that accurately represent the field conditions under study, to assess compliance with the Data Quality Objectives (DQOs), and to assess biases in the measurement system.

10.2.1 Field Activities

In order to ensure the representativeness of samples collected during the test, and to ensure integrity of field measurements, a variety of QC checks and controls will be implemented throughout the sampling program. These checks and controls will include:

- Standard forms and/or standard field notebooks will be used to document field activities and for data collection. The data collection forms and field notebooks will be reviewed routinely by senior staff for accuracy, completeness, and internal consistency.
- The strict adherence to detailed operating procedures as documented in the various project controlling documents and related SOPs will be enforced by experienced senior technical staff.
- Project personnel will be selected based on appropriate levels of training and experience and will receive project specific training prior to working on-site. Training will include health and safety requirements; security requirements; briefings on overall project goals, objectives, and schedules; and, specific technical training related to their assigned tasks. Training will be documented in the project files.
- Routine calibration will be performed on measurement systems and sampling equipment including metering systems, thermocouples, barometers, rotameters, and pitot tubes. Guidance related to equipment calibration is provided in Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods and Quality Assurance/Quality Control Procedures for Hazardous Waste Incinerators, Appendix A. The detailed specifications, acceptance criteria, and corrective action requirements are presented in Section 8.0 of this QAPP. All calibrations will be documented, and the documentation maintained in the project files.
- Leak checks will be performed according to method specifications before and after sampling.
- Field QC samples will be routinely submitted including audit (PE) samples, field blanks, media blanks, reagent blanks, trip blanks, and contingency samples. The frequency of submittal for these field QC samples and other field samples are provided in Tables 5-1 and 8-2.
- Field audits/surveillance will be performed periodically to assess conformance to specifications. If nonconforming conditions are noted, the corrective action provisions of the QA plan will be invoked.

10.2.2 Laboratory Activities

Standard laboratory QA procedures, required of each laboratory, provide discussions related to QA/QC checks and controls within the laboratory. Specific data quality objectives, calibration requirements, acceptance criteria, and corrective action requirements for this test program are presented in Table 5-1 and Table 8-2 of this plan.

In addition to the requirements referenced above the laboratory will provide for quality control of sampling media and sample collection equipment. Sorbents used in the organic sampling trains will be prepared according to method specifications. Samples of the prepared media will be tested according to the intended method of use and analysis prior to shipping media to the field. The results of these tests will be retained in the laboratory's files for future reference.

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Ash	Spent Activated Carbon	4	--	Gravimetric before muffle furnace combustion (ASTM Method D-482)	Gravimetric of residue after muffle furnace combustion (ASTM Method D-482)	Duplicate	One per test	1	5
Heat Content				NA	Bomb calorimeter (ASTM Method D-5865)	Duplicate	One per test	1	5
Total Chloride				Oxygen bomb combustion (SW-846 Method 5050)	Ion chromatography of residue (SW-846 Method 9056A)	Duplicate	One per test	1	5
Total Sulfur				Oxygen bomb combustion (ASTM-D129)	Ion chromatography of residue (ASTM D-129)	Duplicate	One per test	1	5
Ultimate (C,H,O,N and Moisture)				ASTM D-3176	ASTM D-3176	Duplicate	One per test	1	5
Volatile Organics	Spent Activated Carbon	4	--	Puge and Trap (SW-846 Method 5030B)	GC/MS (SW-846 Method 8260B)	Surrogate spikes	Every analysis [c]	4	5
						Duplicate	One per test	1	
	Scrubber Blowdown	4	--	NA	GC/MS (SW-846 Method 8260B)	Surrogate spikes	Every analysis [c]	4	5
						Duplicate	One per test	1	
	Analytical system QC	NA	NA	NA	GC/MS (SW-846 Method 8260B)	LCS	One per batch/ matrix specific	2 or more	2
						Method Blank	One per batch/ matrix specific	2 or more	2

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Semivolatile Organics	Spent Activated Carbon	4	--	Ultrasonic Extraction & Dilution (SW-846 Method 3550C)	GC/MS (SW-846 Method 8270D)	Surrogate spikes incl. duplicates [c]	Every analysis [c]	4	5
						Duplicate	One per test	1	
	Scrubber Blowdown	4	--	NA	GC/MS (SW-846 Method 8270D)	Surrogate spikes	Every sample	4	5
						Duplicate	One per test	1	
	Analytical system QC	NA	NA	NA	GC/MS (SW-846 Method 8270D)	LCS	One per batch/ matrix specific	2 or more	2
						Method Blank	One per batch/ matrix specific	2 or more	2
Metals by ICP	Spent Activated Carbon	4	--	Digestion (SW-846 Method 3051B)	ICP (SW-846 Method 6010C/6020A)	MS/MSD [d]	One per test	2	6
	Scrubber Blowdown	4	--	Digestion (SW-846 Method 3010A)	ICP (SW-846 Method 6010C/6020A)	MS/MSD [d]	One per test	2	6
	Analytical system QC	NA	NA	Digestion (SW-846 Method 3010A/3051B)	ICP (SW-846 Method 6010C/6020A)	LCS	One per batch/ matrix specific	2 or more	2
						Serial dilution	One per batch/ matrix specific	2 or more	2
						Method Blank	One per batch/ matrix specific	2 or more	2

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Method 0030 VOST for Volatile PICs	VOST stack sample tube pairs	16	--	NA (Note: Only 3 of the 4 tube pairs from each test run will be analyzed; the fourth pair is a back-up in case of tube breakage.	Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); Each tube in each tube pair is analyzed separately.	Surrogate spikes[c]	Every analysis [c]	27	27
	VOST Condensate	4	--	NA	Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B)	Surrogate spikes[c]	Every analysis [c]	4	4
	VOST field blank tube pairs	4	--	NA	Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); Tube pair is analyzed as a single sample.	Surrogate spikes[c]	Every analysis [c]	4	4
	VOST trip blank tube pairs	1	--	NA	Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); Tube pair is analyzed as a single sample.	Surrogate spikes[c]	Every analysis [c]	1	1
	VOST Cond. Trip Blanks	1	--	NA	Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B)	Surrogate spikes[c]	Every analysis [c]	1	1
	Spiked resin blank tube pairs	--	8	NA	Purge and trap, GC/MS (SW-846 Methods 5041A, 8260B); Tube pair is analyzed as a single sample.	Surrogate spikes[c]	Every analysis [c]	8	8
	Analytical system QC	NA	NA	NA	Purge and trap, GC/MS (SW-846 Method 8260B)	LCS	1 per condensate batch	2 or more	3
						Method blank	1 per analytical run	2 or more	2

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
PCDD/PCDFs, PCBs, and PAHs by EPA Method 23	Method 23 solvent rinses, filter, and XAD resin composite	4	1	Soxhlet extraction (EPA Method 23)	HRGC/HRMS for PCDD/PDDFs (EPA Method 23)	PCDD/PCDF pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	5	5
						PCDD/PCDF isotope dilution internal standard spike	Every analysis [c]	5	
						PCDD/PCDF recovery standard spike	Every analysis [c]	5	
	Method 23 solvent rinses, filter, XAD resin, and condensate composite	4	1	Soxhlet extraction and separatory funnel extraction (EPA Method 23)	HRGC/HRMS for PAHs (EPA Method 23)	PAH pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	5	5
						PAH isotope dilution internal standard spike	Every analysis [c]	5	
						PAH recovery standard spike	Every analysis [c]	5	
	Method 23 solvent rinses, filter, XAD resin, and condensate composite	4	1	Soxhlet extraction and separatory funnel extraction (EPA Method 23)	HRGC/HRMS for PCBs (EPA Method 23)	PCB pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	5	5
						PCB extraction isotope dilution internal standard spike	Every analysis [c]	5	
						PCB surrogate standard spike	Every analysis [c]	5	
						PCB recovery standard spike	Every analysis [c]	5	

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
PCDD/PCDFs, PCBs, and PAHs by EPA Method 23	Method 23 acetone reagent blank	--	1	NA	HRGC/HRMS for PCDD/PDDFs (EPA Method 23)	PCDD/PCDF isotope dilution internal standard spike	Every analysis [c]	1	1
						PCDD/PCDF recovery standard spike	Every analysis [c]	1	
	Method 23 toluene reagent blank	--	1	NA	HRGC/HRMS for PCDD/PDDFs (EPA Method 23)	PCDD/PCDF isotope dilution internal standard spike	Every analysis [c]	1	1
						PCDD/PCDF recovery standard spike	Every analysis [c]	1	
	Method 23 acetone reagent blank	--	1	NA	HRGC/HRMS for PAHs (EPA Method 23)	PAH isotope dilution internal standard spike	Every analysis [c]	1	1
						PAH recovery standard spike	Every analysis [c]	1	
	Method 23 toluene reagent blank	--	1	NA	HRGC/HRMS for PAHs (EPA Method 23)	PAH isotope dilution internal standard spike	Every analysis [c]	1	1
						PAH recovery standard spike	Every analysis [c]	1	

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
PCDD/PCDFs, PCBs, and PAHs by EPA Method 23	Method 23 acetone reagent blank	--	1	NA	HRGC/HRMS for PCBs (EPA Method 23)	PAH isotope dilution internal standard spike	Every analysis [c]	1	1
						PAH recovery standard spike	Every analysis [c]	1	
	Method 23 toluene reagent blank	--	1	NA	HRGC/HRMS for PCBs (EPA Method 23)	PAH isotope dilution internal standard spike	Every analysis [c]	1	1
						PCB surrogate standard spike	Every analysis [c]	1	
						PAH recovery standard spike	Every analysis [c]	1	
	Method 23 spiked XAD-2 resin blank	--	2	Soxhlet extraction (EPA Method 23)	HRGC/HRMS for PCDD/PDDFs (EPA Method 23)	PCDD/PCDF pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	1	2
						PCDD/PCDF isotope dilution internal standard spike	Every analysis [c]	1	
						PCDD/PCDF recovery standard spike	Every analysis [c]	1	
	Analytical system QC	NA	NA	NA	HRGC/HRMS for PCDD/PDDFs (EPA Method 23)	Method blank	1 per analytical batch	1 or more	1
						Blank spike	2 per analytical batch	2	2

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
PCDD/PCDFs, PCBs, and PAHs by EPA Method 23	Method 23 spiked XAD-2 resin blank	--	2	Soxhlet extraction (EPA Method 23)	HRGC/HRMS for PCBs (EPA Method 23	PCB pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	2	2
						PCB extraction isotope dilution internal standard spike	Every analysis [c]	2	
						PCB surrogate standard spike	Every analysis [c]	2	
						PCB recovery standard spike	Every analysis [c]	2	
	Analytical system QC	NA	NA	NA	HRGC/HRMS for PCBs (EPA Method 23	Method blank	1 per analytical batch	1 or more	1
						Blank spike	2 per analytical batch	2	2
	Method 23 spiked XAD-2 resin blank	--	2	Soxhlet extraction (EPA Method 23)	HRGC/HRMS for PAHs (EPA Method 23	PAH pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	2	2
						PAH isotope dilution internal standard spike	Every analysis [c]	2	
						PAH recovery standard spike	Every analysis [c]	2	
	Analytical system QC	NA	NA	NA	HRGC/HRMS for PAHs (EPA Method 23	Method blank	1 per analytical batch	1 or more	1
						Blank spike	2 per analytical batch	2	2

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
SVOCs by SW846 Method 0010	Method 0010 front half composite; filter, and front half of filter holder and probe rinses (cont'd)	4	1	Soxhlet extraction (SW-846 Method 3542A)	GC/MS for SVOCs (SW-846 Method 8270D)	Semivolatile surrogate spikes	Every analysis [c]	5	5
						Semivolatile internal standard surrogate spikes	Every analysis [c]	5	
					GC/MS with SIM for PDCB (SW-846 Method 8270D)	Semivolatile surrogate spikes	Every analysis [c]	5	5
						Semivolatile internal standard surrogate spikes	Every analysis [c]	5	

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
SVOCs by SW846 Method 0010 (cont'd)	Method 0010 back half composite; XAD-2 resin, and back half of filter holder and condenser rinses.	4	1	Soxhlet extraction (SW-846 Method 3542A)	GC/MS for SVOCs (SW-846 Method 8270D)	Pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	5	5
						Semivolatile surrogate spikes	Every analysis [c]	5	
						Semivolatile internal standard surrogate spikes	Every analysis [c]	5	
					GC/MS with SIM for PDCB (SW-846 Method 8270D)	Pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	5	5
						Semivolatile surrogate spikes	Every analysis [c]	5	
						Semivolatile internal standard surrogate spikes	Every analysis [c]	5	

Table 10-1. Summary of Test Program Analyses (cont'd)

Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]	Total Analyses [b]
SVOCs by SW846 Method 0010 (cont'd)	Method 0010 Condensate Impinger	4	1	Separatory funnel extraction (SW-846 Method 3542A)	GC/MS for SVOCs (SW-846 Method 8270D)	Semivolatile surrogate spikes	Every analysis [c]	5	5
						Semivolatile internal standard surrogate spikes	Every analysis [c]	5	
					GC/MS with SIM for PDCB (SW-846 Method 8270D)	Semivolatile surrogate spikes	Every analysis [c]	5	5
						Semivolatile internal standard surrogate spikes	Every analysis [c]	5	
	Method 0010 acetone reagent blank	--	1	NA	GC/MS for SVOCs (SW-846 Method 8270D)	Semivolatile surrogate spikes	Every analysis [c]	1	1
						Semivolatile internal standard surrogate spikes	Every analysis [c]	1	
					GC/MS with SIM for PDCB (SW-846 Method 8270D)	Semivolatile surrogate spikes	Every analysis [c]	1	1
						Semivolatile internal standard surrogate spikes	Every analysis [c]	1	

Table 10-1. Summary of Test Program Analyses (cont'd)

Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]	Total Analyses [b]
SVOCs by SW846 Method 0010 (cont'd)	Method 0010 methylene chloride reagent blank	--	1	NA	GC/MS (SW-846 Method 8270D)	Semivolatile surrogate spikes	Every analysis [c]	1	1
						Semivolatile internal standard surrogate spikes	Every analysis [c]	1	
					GC/MS with SIM for PDCB (SW-846 Method 8270D)	Semivolatile surrogate spikes	Every analysis [c]	1	1
						Semivolatile internal standard surrogate spikes	Every analysis [c]	1	
	Method 0010 spiked XAD-2 resin blank	--	2	Soxhlet extraction (SW-846 Method 3542A)	GC/MS for SVOCs (SW-846 Method 8270D)	Pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	2	2
						Semivolatile surrogate spikes	Every analysis [c]	2	
						Semivolatile internal standard surrogate spikes	Every analysis [c]	2	

Table 10-1. Summary of Test Program Analyses (cont'd)

Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]	Total Analyses [b]
SVOCs by SW846 Method 0010 (cont'd)					GC/MS with SIM for PDCB (SW-846 Method 8270D)	Pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling [c]	2	2
						Semivolatile surrogate spikes	Every analysis [c]	2	
						Semivolatile internal standard surrogate spikes	Every analysis [c]	2	
	Analytical system QC	NA	NA	NA	GC/MS for SVOCs (SW-846 Method 8270D)	Method blank	1 per analytical batch	1 or more	1
						Blank spike	2 per analytical batch	2	2
					GC/MS with SIM for PDCB (SW-846 Method 8270D)	Method blank	1 per analytical batch	1 or more	1
						Blank spike	2 per analytical batch	2	2

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
OCPs by SW846 Method 0010	Method 0010 solvent rinses, filter, XAD resin, and condensate composite	4	1	Soxhlet extraction and separatory funnel extraction (SW-846 Method 3542A)	GC/ECD for OCPs (EPA TO4A)	OCP surrogate spikes	Every analysis [c]	5	5
						OCP internal standard surrogate spikes	Every analysis [c]	5	
	Method 0010 acetone reagent blank	--	1	NA	GC/ECD for OCPs (EPA TO4A)	OCP surrogate spikes	Every analysis [c]	1	1
						OCP internal standard surrogate spikes	Every analysis [c]	1	
	Method 0010 methylene chloride reagent blank	--	1	NA	GC/ECD for OCPs (EPA TO4A)	OCP surrogate spikes	Every analysis [c]	1	1
						OCP internal standard surrogate spikes	Every analysis [c]	1	
	Method 0010 spiked XAD-2 resin blank	--	2	Soxhlet extraction (EPA Method 23)	GC/ECD for OCPs (EPA TO4A)	Pre-sampling OCP surrogate spikes	Every XAD-2 resin tube before sampling [c]	2	2
						OCP surrogate spikes	Every analysis [c]	2	
						OCP internal standard surrogate spikes	Every analysis [c]	2	

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
OCPs by SW846 Method 0010 (cont'd)	Analytical system QC	NA	NA	NA	GC/ECD for OCPs (EPA TO4A)	Method blank	1 per analytical batch	1 or more	1
						Blank spike	2 per analytical batch	2	2
Total SVOC, and NVOC by Method 0010 EPA TOE Guidance	Semivolatile front half composite: filter and solvent rinses	4	1	Soxhlet extraction (SW-846 Method 3540C)	GC/FID analysis of one-half of extract (SW-846 Method 8015B)	Marker compounds added to TCO fraction for GC/FID analysis	Every analysis	5	5
					Gravimetric (Grav) analysis of one-half of extract (TOE Guidance)	Replicate weighing of Grav fraction to constant weight	Every analysis	5	5
	Semivolatile back half composite: XAD-2 resin and condenser solvent rinses	4	1	Soxhlet extraction (SW-846 Method 3540C)	GC/FID analysis of one-half of extract (SW-846 Method 8015B)	Marker compounds added to TCO fraction for GC/FID analysis	Every analysis	5	5
					Gravimetric (Grav) analysis of one-half of extract (TOE Guidance)	Replicate weighing of Grav fraction to constant weight	Every analysis	5	5

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Total SVOC, and NVOC by Method 0010 EPA TOE Guidance (cont'd)	Semivolatile impinger liquid and rinses	4	1	Separatory funnel extraction (SW-846 Methods 3540C)	GC/FID analysis of one-half of extract (SW-846 Method 8015B)	Marker compounds added to TCO fraction for GC/FID analysis	Every analysis	5	5
					Gravimetric (Grav) analysis of one-half of extract (TOE Guidance)	Replicate weighing of Grav fraction to constant weight	Every analysis	5	5
	Acetone reagent blank	--	1	NA	GC/FID analysis of one-half of extract (SW-846 Method 8015B)	Marker compounds added to TCO fraction for GC/FID analysis	Every analysis	1	1
					Gravimetric (Grav) analysis of one-half of extract (TOE Guidance)	Replicate weighing of Grav fraction to constant weight	Every analysis	1	1
	Methylene chloride reagent blank	--	1	NA	GC/FID analysis of one-half of extract (SW-846 Method 8015B)	Marker compounds added to TCO fraction for GC/FID analysis	Every analysis	1	1
					Gravimetric (Grav) analysis of one-half of extract (TOE Guidance)	Replicate weighing of Grav fraction to constant weight	Every analysis	1	1

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Total volatile organics by Method 0040	Tedlar Bags	8	--	NA	GC/FID (SW-846 Method 0040/8015B)	Duplicate	Every sample	8	24
	Field Blank Bag	4	--	NA	GC/FID (SW-846 Method 0040/8015B)	Field blank	One per run	4	
	Condensate	4	--	NA	Purge and trap GC/FID (SW-846 Method 0040/8015B)	Duplicate	One per run	2	9
	Condensate blank	1	--	NA	Purge and trap GC/FID (SW-846 Method 0040/8015B)	Field blank	One per test	2	
	Analytical system QC	NA	NA	NA	GC/FID (SW-846 Method 0040/8015B)	Zero gas	One per run	4	12
						Known gas	Two per run	8	
					Purge and trap GC/FID (SW-846 Methods 0040 and 5030)	Method blank (water)	One per analytical batch	1 or more	1
Metals by Method 29	Method 29 front half: filter, and nitric acid probe and front half filter holder rinses	4	1	Method 29	ICP (SW-846 Method 6010C/6020A)	PDS [d]	One per test	1	6
					CVAA (SW-846 Method 7470A/7471B)	PDS [d]	One per test	1	6

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Metals by Method 29 (cont'd)	Method 29 10%HNO_3/5%H_2O_2 impinger contents and rinses	4	1	Method 29	ICP (SW-846 Method 6010C/6020A)	PDS [d]	One per test	1	6
					CVAA (SW-846 Method 7470A/7471B)	PDS [d]	One per test	1	6
	Method 29 initially empty contents and rinses	4	1	Method 29	CVAA (SW-846 Method 7470A/7471B)	PDS [d]	One per test	1	6
	Method 29 4%$KMnO_4$/10%H_2SO_4 impinger contents and rinses	4	1	Method 29	CVAA (SW-846 Method 7470A/7471B)	PDS [d]	One per test	1	6
	Method 29 4%KMnO4/10%H2SO4 8N HCl rinses	4	1	Method 29	CVAA (SW-846 Method 7470A/7471B)	PDS [d]	One per test	1	6
	Method 29 filter reagent blank	--	1	Method 29	ICP (SW-846 Method 6010C/6020A)	Reagent Blank	One for test program	1	1
					CVAA (SW-846 Method 7470A/7471B)	Reagent Blank	One for test program	1	1

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Metals by Method 29 (cont'd)	Method 29 HNO_3 reagent blank	--	1	Method 29	ICP (SW-846 Method 6010C/6020A)	Reagent Blank	One for test program	1	1
					CVAA (SW-846 Method 7470A/7471B)	Reagent Blank	One for test program	1	1
	Method 29 10%HNO_3/5%H_2O_2 reagent blank	--	1	Method 29	ICP (SW-846 Method 6010C/6020A)	Reagent Blank	One for test program	1	1
					CVAA (SW-846 Method 7470A/7471B)	Reagent Blank	One for test program	1	1
	Method 29 4%$KMnO_4$/10%H_2SO_4 reagent blank	--	1	Method 29	CVAA (SW-846 Method 7470A/7471B)	Reagent Blank	One for test program	1	1
	Method 29 8N HCl reagent blank	--	1	Method 29	CVAA (SW-846 Method 7470A/7471B)	Reagent Blank	One for test program	1	1
	Analytical system QC	NA	NA	NA	ICP (SW-846 Methods 3010A, 6010C/6020A)	LCS	One per batch/ matrix specific	1	1
						Serial dilution	One per batch/ matrix specific	1	1
						Method blank	One per batch/ matrix specific	1	1

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Metals by Method 29 (cont'd)	Analytical system QC	NA	NA	NA	CVAA (SW-846 Method 7470A/7471B)	LCS	One per batch/ matrix specific	1	1
						Serial dilution	One per batch/ matrix specific	1	1
						Method blank	One per batch/ matrix specific	1	1
Hexavalent Chromium by Method 0061	Method 0061 Impinger Solution and Rinses	6	--	NA	Ion chromatography post-column reactor (SW-846 Method 7199)	Duplicate	One per test	1	5
	Method 0061 Potassium Hydroxide Reagent Blank	--	1	NA	Ion chromatography post-column reactor (SW-846 Method 7199)	Reagent Blank	One for test program	1	1
	Method 0061 Deionized Water Reagent Blank	--	1	NA	Ion chromatography post-column reactor (SW-846 Method 7199)	Reagent Blank	One for test program	1	1
	Method 0061 Field Spiked to Impinger Solution Aliquot	--	2	NA	Ion chromatography post-column reactor (SW-846 Method 7199)	Field spike	One set for test program	2	2

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Hexavalent Chromium by Method 0061 (cont'd)	Method 0061 Field Spiked Potassium Hydroxide Solution	--	1	NA	Ion chromatography post-column reactor (SW-846 Method 7199)	Field spike	One for test program	1	1
	Analytical system QC	NA	NA	NA	Ion chromatography post-column reactor (SW-846 Method 7199)	LCS/LCSD	1 per batch following initial calibration (separate calibration for each matrix)	1	2
						Method Blank	One per batch/ matrix specific - analyzed in duplicate	1	
Particulate by Method 5/26A	Method 5/2A particulate filter	4	--	Desiccate to constant mass	Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	4	4
	Method 5/26A probe and filter holder acetone rinses	4	--	Evaporate/Desiccate to constant mass	Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	4	4

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Particulate by Method 5/26A (cont'd)	Method 5/26A filter reagent blank	--	1	Desiccate to constant mass	Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	1	1
	Method 5/26A acetone reagent blank	--	1	Evaporate/Desiccate to constant mass	Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	1	1
HCl by Method 5/26A	Method 5/26A H2SO4 impingers	4	--	NA	Ion chromatography (EPA Method 26A)	Duplicate	One per test	1	7
						MS/MSD analyzed in duplicate [d]	1 per batch (assuming all samples batched together)	2	
	Method 5/26A H2SO4 reagent blank	--	1	NA	Ion chromatography (EPA Method 26A)	Reagent Blank	One for test program	1	1
Cl_2 by Method 5/26A	Method 5/26A NaOH impingers	4	--	NA	Ion chromatography (EPA Method 26A)	Duplicate	One per test	1	7
						MS/MSD analyzed in duplicate [d]	1 per batch (assuming all samples batched together)	2	

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Cl^- ion by Method 5/26A (cont'd)	Method 5/26A NaOH reagent blank	--	1	NA	Ion chromatography (EPA Method 26A)	Reagent Blank	One for test program	1	1
	Method 5/26A deionized water reagent blank	--	1	NA	Ion chromatography (EPA Method 26A)	Reagent Blank	One for test program	1	1
Cl^- ion chromatography	Analytical system QC	NA	NA	NA	Ion chromatography (EPA Method 26A)	LCS/LCSD	1 per batch following initial calibration (separate calibration for each matrix)	1	2
						Method Blank	One per batch/ matrix specific - analyzed in duplicate	1	
Method 5 PSD	Method 5-PSD particulate filter	4	--	NA	Scanning Electron Microscope (SEM)	NA	NA	NA	4
	Method 5-PSD probe and filter holder acetone rinses	4	--	NA	Scanning Electron Microscope (SEM)	NA	NA	NA	4

Table 10-1. Summary of Test Program Analyses (cont'd)

Analysis	Sample Matrix	Test	Field QC	Reference Preparation Method	Reference Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Method 5 PSD (cont'd)	Method 5-PSD filter reagent blank	--	1	NA	Scanning Electron Microscope (SEM)	NA	NA	NA	1
	Method 5-PSD acetone reagent blank	--	1	NA	Scanning Electron Microscope (SEM)	NA	NA	NA	1
TOTAL									**400**

Notes:
[a] Each test condition is comprised of four replicate sampling runs.
[b] Total laboratory analyses includes field sample analyses and laboratory QC analyses
[c] Surrogate spikes are applied to all samples. Refer to Table 5-1 notes for the surrogate compounds.
[d] MS = Matrix spike
MSD = Matrix spike duplicate
PDS = Post-digestion spike
[e] Refer to Table 5-1 notes for the matrix spike compounds.

11.0 DATA REDUCTION, DATA VALIDATION, AND DATA REPORTING

11.1 DATA REDUCTION

11.1.1 General Principles

11.1.1.1 Field

Data reduction will occur for the field measurements at the point of sampling. At the point of sampling, the data as measured by the field instrument will be reported in the field notebooks and/or on any forms required for the project.

11.1.1.2 Office

After the field event, the data may be further reduced to data tables, trend analysis tables, or graphs. At any point where manual transcriptions of data take place, an editing function will be invoked to ensure accuracy of the transcriptions.

Upon the return of the analytical results from the laboratory and after data validation, the data will be further reduced to data tables. The data tables will contain the following information:

- Information identifying exactly the samples represented on the tables (e.g. sample location, matrix, etc.),
- The compounds for which the samples were tested,
- The results for each compound, and
- The data flags as applied by the laboratory or by data validators (if used).

Emissions concentrations and mass emissions of the stack gas target analytes will be calculated using the laboratory analyses and the sampled volume, stack flow, moisture, temperature, and pressure data from the respective sampling trains used to sample for the specific target constituents. Example calculations will be included in the emissions sampling report and example calculations appendices to the PDT report.

Total hydrocarbons as measured by EPA Method 25A are wet and not oxygen corrected. EPA Method 4 moisture data from concurrently operated isokinetic sampling trains and oxygen data from EPA Method 3A will be used to correct and report total hydrocarbons in parts per million by volume, corrected to 7 percent oxygen by volume, dry basis.

Method 0030 and Method 0040 VOC sampling do not include measurement of stack flow. EPA Methods 2, 3A, and 4 flow, gas molecular weight, and moisture data from concurrently operated isokinetic sampling trains will be used to report mass emissions for these sampling methods.

11.1.1.3 Laboratory

Data reduction in the laboratory is covered in the Laboratory's QA Manual and SOPs. The laboratory's data reduction process will include at a minimum the following.

- Transcription of data results from raw data printouts to data report forms. This will include any calculations required to report the data in the required units.
- Transcription of QA/QC data onto summary forms to provide the required information for evaluation of the validity of the data. The requirement for each type of data is included in section 11.3.

11.1.2 Specific Data Reduction Requirements

11.1.2.1 GC and GC/MS Techniques

Organic analyses will all be conducted using gas chromatography (GC) techniques. The VOC, SVOC, OCP, PCDD/PCDF, PCB, and PAH analyses will employ mass spectral (MS) detectors. Although the principles of operation and specific methods of calibration differ according to the analyte specific methods, the general data reduction scheme is the same for all of these tests. The individual methods should be consulted for details. A summary of the data reduction scheme is presented below.

Depending on the specific method, analytical instrumentation is calibrated using a minimum of five (5) points covering the expected analytical range. The gas chromatograph/flame ionization detector (GC/FID) or gas chromatograph/electron capture detector (GC/ECD) methods generally employ an external standard calibration technique while the GC/MS methods employ an internal standard technique. For GC/FID and GC/ECD methods, a calibration factor (CF) is calculated using the following formula:

$$CF = \frac{R}{M}$$

Where: CF = Calibration Factor

R = Response or Area of the GC Peak

M = Mass Injected (in nanograms)

The calibration factors must agree to within method specified criteria for the percent relative standard deviation (%RSD). The formula for %RSD is given below.

$$\%\,\mathrm{RSD} = \frac{\sigma}{\mathrm{avg\,CF}} \times 100$$

Where: $\%RSD$ = Percent Relative Standard Deviation

σ = Standard Deviation of the Calibration Factors

$avg\ CF$ = Average Calibration Factor

For GC/MS calibrations a response factor (RF) is used rather than the CF. The formula for the RF is:

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}$$

Where: RF = Response Factor

A_s = Response for the Analyte

A_{is} = Response for the Internal Standard

C_s = Concentration of the Analyte

C_{is} = Concentration of the Internal Standard

The RFs must also pass a test of the %RSD in order for the calibration to be considered valid.

When samples are analyzed, the area of the peak produced by a given analyte is compared to the CF or RF to arrive at an analytical result according to the following formula:

$$M_x = A_x \times CF$$

Where:

M_x = The Mass of Analyte in the Sample

A_x = The Response of the Analyte in the Sample

CF = The Calibration (or Response) Factor

Where possible, samples containing more of an analyte than the instrument is calibrated for, will have a dilution performed, if such a dilution is practical given the sample preparation method. For most analyses where the initial analysis value exceeds the calibrated range (“E” flagged values), an aliquot of the prepared sample is available that can be serial diluted and re-analyzed to bring the exceeding analyte to within the calibrated range for quantification. In that case M_x is multiplied by the dilution factor to arrive at the final result. If, under the circumstances of the method, a dilution is not possible, the analytical result must be considered estimated.

To arrive at a concentration in the gas sample the mass of any sub-samples must be added together and then compared to the volume of gas sampled according to the following formula:

$$C_x = \frac{(M_1 + M_2 + ...M_n)}{V}$$

Where: C_x = The Concentration of the Analyte in the Gas Sample

M_n = The Result (Mass) for Each Component in the Sampling Train

V = The Volume of Gas Sampled

11.1.2.2 Analysis of Metals by ICP and Atomic Absorption

The analysis of metals also begins with an instrument calibration check at 2 to 6 points, depending on the specific analytical method. For inductively coupled argon plasma spectroscopy (ICP) for non-mercury (non-Hg) metals analyses and cold vapor atomic adsorption spectroscopy (CVAA) for mercury (Hg) analyses, instruments are profiled and calibrated according to the instrument manufacturer's instructions. A calibration blank and a QC check standard are then analyzed to ensure appropriate instrument response. A percent recovery (%R) is calculated according to the following formula:

$$\%R = \frac{Found}{True} \times 100$$

Where: $\%R$ = Percent Recovery

$Found$ = The Result of the Analysis

$True$ = The Expected Result

The %R is expected to be within method specifications before analysis can begin. The calibration is verified periodically according to method specifications using the same technique.

Atomic absorption instruments are calibrated at a minimum of five (5) points. A linear regression is performed, and a correlation coefficient is calculated to assess linearity of the curve. It is beyond the scope of this document to provide a detailed explanation of the statistics supporting linear regression and the calculation of the correlation coefficient. Reference can be made to any standard statistics text for additional information. Calibration checks are performed as above and periodically verified.

Analytical results for metals are read directly from the instrument in terms of concentration. Dilution factors must be used as discussed above if applicable. In order to combine the results of the sub-samples of a metals sampling train, the concentration is converted back to mass using the following formula:

$$M_{xs} = C_{xs} \times V_{xs}$$

Where: M_{xs} = Mass in the Sub-sample
C_{xs} = Concentration in the Sub-sample
V_{xs} = Volume of the Sub-sample

The concentration in the gas sample can then be calculated.

11.1.2.3 Ion Chromatography

Anions, such as chloride, are separated on the ion chromatograph using a system comprising separator columns, guard columns, and eluents. The system is calibrated at a minimum of five (5) points and the calibration is verified with a mid-range standard. Samples are quantitated in the same manner as given above.

11.1.2.4 Direct Reading Instruments

Gravimetric, temperature, pressure, flow, and CEMS data are directly read from the measurement instrumentation. The instrumentation will be calibration checked prior to the test, and routinely prior to reading measurements, however, no data reduction beyond formatting into tables is expected.

11.2 DATA VALIDATION

The results of all sample analysis and all QA/QC sample analysis (100% of the laboratory data) will be compared, step by step, by the QAO or his/her designee, to the specifications given in Tables 5-1 and 8-2. The data validation criteria outlined in: Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis, (1994) and Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis, (1994); prepared by USEPA Data Review Work Group will be followed as applicable to the individual methods used. Any sample data associated with a QC check that fail to meet the target criteria established in these tables will be flagged in the final report, and an assessment of the impact, if any, of missing the target data quality objective will be provided. Additional guidance will be found in the analytical methods and EPA/625/6-89/023, Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration.

Each laboratory providing analysis will be required to provide the results based on the method detection limit (MDL) and reporting limit (RL) for all non-isotope dilution method compounds. Non-detects for the isotope dilution methods will be determined using the method specific SW-846 definition of an estimated detection limit (EDL) without the use of empirical factors or other mathematical manipulations specific to the laboratory. Results reported between the MDL or EDL and the RL will be flagged as estimated.

Particular attention will be paid to the results of blank data. Analytical data will not be routinely corrected for contamination. They are however evaluated on a case-by-case basis for possible blank correction. A "B" flag will be applied to the samples associated with contaminated blanks such that this information may be assessed in the final report. Data for HHERA use will be evaluated against all associated blanks to determine if sample results are less than five times (ten times for common laboratory contaminants) the concentration reported in the blanks. Sample results reported at concentrations less than 5X/10X the blank concentration will be considered not present in the sample and will be qualified as not detected (ND or U).

The output from the data validation process will be a summary comparison of the QA/QC results to the specified data quality objectives, a review and discussion of any deficiencies identified in the data assessments of laboratory performance, and, overall precision and accuracy, representativeness and completeness of the data set.

Detailed procedures for the internal review of data in the laboratory are found in the laboratories QA Manuals and related standard operating procedures (SOPs).

11.3 DATA REPORTING

11.3.1 Experimental Data

Experimental data that will be reported as part of the final test report will include:

- All relevant field measurements in raw and tabular form. This will include, but not necessarily be limited to, calibration data for field instruments, velocity and gas flow measurements, and temperature and pressure measurements.
- Process monitoring data
- All CEMS data to include CO, O_2, CO_2, THC, NOx, and SO_2
- Analytical laboratory data for all laboratory measurements.

The laboratory deliverable package is expected to include the following elements:

The following forms for all organic analyses using Gas Chromatography/Mass Spectroscopy methods:

- Case narrative and sample identification cross reference

- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Organic analysis data sheet for samples, blanks, and QC analysis (CLP Form 1 or equivalent)
- System monitoring compound/surrogate recoveries summary (CLP Form 2 or equivalent)
- Duplicate analysis summary (CLP Form 3 or equivalent if MS/MSD)
- QC Check Sample summary
- Method blank summary (CLP Form 4 or equivalent) and results
- Instrument performance check summary – tuning reports (CLP Form 5 or equivalent)
- Initial calibration summary (CLP Form 6 or equivalent)
- Continuing calibration check (CLP Form 7 or equivalent)
- Internal standard area and RT summary (CLP Form 8 or equivalent)
- Internal standard recovery summary (PCDD/PCDFs, PCBs, and PAHs)
- DDT/Endrin breakdown standards
- Raw data: run logs, mass spectra, quantitation reports, manual integration, and chromatograms for samples, tunes, calibrations, and QC samples, sample preparation logs, and run logs.
- Standards preparation logs and certificate is required
- Sample receipt information including temperature and pH information if preservation is required
- Documentation of all nonconformances and the actions taken
- Examples of all calculations performed
- Detection limits including method detection limits and sample quantitation limits
- Any performance evaluation samples provided
- Percent solids or percent moisture for soil samples

The following forms for all organic analyses using Gas Chromatography:

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Organic analysis data sheet for samples, blanks, and QC samples including retention times of required for the analysis (CLP Form 1 or equivalent)
- System monitoring compound/surrogate recoveries summary (CLP Form 2 or equivalent)
- Duplicate analysis summary (CLP Form 3 or equivalent if MS/MSD)
- QC Check Sample summary
- Method blank summary and results (CLP Form 4 or equivalent)
- Initial calibration summary (CLP Form 6 or equivalent)

- Calibration verification summary (CLP Form 7 or equivalent)
- Graphic Representation of Curve Fit, with Correlation Coefficient
- Analytical Sequence (run logs)
- Raw data: quantitation reports, manual integrations, and chromatograms, and retention times for each column in all field and QC samples, sample preparation logs, and run logs.
- Standards preparation logs and certificates (if applicable)
- Sample receipt information including temperature and pH information if preservation is required
- Documentation of all nonconformances and the actions taken
- Examples of all calculations performed
- Compound confirmation (if required for the analysis)
- Peak Resolution Summary (if required for analysis)
- Retention time window determination
- Detection limits including method detection limits and sample quantitation limits
- Any performance evaluation samples provided
- Percent solids or percent moisture for soil samples

The following forms for all metals analyses:

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Inorganic analysis data sheet (CLP Form 1 or equivalent)
- Initial and continuing calibration verification (CLP Form 2 or equivalent)
- Blanks summary (CLP Form 3 or equivalent)
- Spike sample recovery/Post digest spike sample recovery (CLP Form 5 or equivalent)
- Interference checks sample results (CLP Form 4 or equivalent)
- Serial dilution results (CLP Form 9 or equivalent)
- Duplicate results (CLP Form 6 or equivalent)
- Laboratory control sample results (CLP Form 7 or equivalent)
- Raw data for samples, blanks, QC samples, calibrations, and instrument checks, sample preparation logs and, instrument run logs.
- Documentation of all nonconformances and the actions taken
- Sample receipt information including temperature and pH information if preservation is required
- Examples of all calculations performed
- Detection limits including method detection limits, instrument detection limits, and quantitation limits

- Method of standard additions data
- ICP-AES inter-element correction (IEC) factors
- ICP-MS tunes
- ICP-MS internal standards relative intensity summary
- Any performance evaluation samples provided
- Standards preparation logs and certificates (if applicable)
- Percent solids or percent moisture for soil samples
- AA – wavelengths used for analysis

The following forms for all inorganic non-metals analyses: (as appropriate for analysis)

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references
- Inorganic analysis data sheet
- Calibration summaries
- Method blank results summary
- Sample spike recovery
- Duplicate sample results
- Laboratory Control Sample.
- Raw data, chromatograms, area printouts, sample preparation logs and, instrument run logs
- Examples of all calculations performed
- Documentation of all nonconformances and the actions taken
- Sample receipt information including temperature and pH information if preservation is required
- Standards preparation logs and certificates (if applicable)
- Percent solids or percent moisture for soil samples
- Logbook pages (gravimetric)
- Scale calibrations (gravimetric)

11.3.2 Reporting of Tentatively Identified Compounds

In addition to the target analytes identified for volatile and semivolatile organic stack gas analysis, there are generally a number of non-target components observed in the chromatogram. Attempts to identify and quantify these unknown chromatographic peaks can improve the percentage of identified organic compounds and reduce overall uncertainty. However, because the instrument is not calibrated for these unknown compounds, the identification and quantitative analysis is tentative until the identification is

confirmed by the analysis of a standard. EPA HHERA guidance recommends that TICs be considered "identified" compounds for purposes of site-specific risk assessments to ensure that appropriate credit is given to defensible efforts to identify the maximum number of organic compounds.

To identify non-target TICs, the mass spectrum can be searched against a computerized library of reference mass spectra. A forward library search selects the largest mass spectral peaks from the unknown mass spectrum and looks for reference spectra in the library that contain the peaks of the unknown. A reverse library search looks for the peaks in the reference spectrum that occur in the unknown mass spectrum. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library matches should the analyst assign a tentative identification. Any components that are identified are referred to as TICs, since no reference standard was analyzed at the same time as the unknown. Without calibration of the instrument with the actual compound, TICs are quantified using the nearest-eluting internal standard with a relative instrument response factor of 1.00. The resulting concentration is considered "estimated," because the response factor is not compound-specific. An unknown level of error in the quantitation is introduced using the response factor of 1.00; this level of error will vary from compound to compound.

Methods 8260/8270 present guidelines for identification of TICs, and these guidelines are summarized below:

- Relative intensities of major ions in the reference mass spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample mass spectrum.
- The relative intensities of the major ions should agree within ±20 percent. Example: for an ion with an abundance of 50 percent in the standard spectrum, the corresponding sample ion abundance should lie between 30 and 70 percent.
- Molecular ions present in the reference mass spectrum should be present in the sample mass spectrum.
- Ions present in the sample mass spectrum but not in the reference mass spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference mass spectrum but not in the sample mass spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library programs can sometimes create these discrepancies.

If, in the judgment of the experienced mass spectral interpreter, no valid tentative identification can be made, the compound should be reported as "unknown." The mass spectral interpreter should give additional classification of the unknown compound, if possible (i.e., unknown aromatic compound, unknown hydrocarbon, unknown chlorinated compound). If a probable molecular weight can be distinguished, this molecular weight should also be reported. The experienced interpreter should apply this experience and

judgment to the mass spectral interpretations supplied by the computerized library search. For example, if a hydrocarbon occurring 40 minutes into the chromatographic analysis is identified by the computer as "octane," analytical judgment dictates that this identification is scientifically illogical, and the compound should be reported as "unknown hydrocarbon." By no means should the computer identifications be accepted uncritically.

11.3.3 Project Reporting Format

The format for the PDT Report final report is outlined in Figure 11-1.

11.3.4 Detection Limits and Data Reduction

Detection limits for organic compounds will be derived as recommended by the U.S. EPA Office of Solid Waste (OSW) in the "Human Health Risk Assessment Protocol" published in July 1998. This protocol recommends that non-isotope dilution methods quantify non-detects using the MDL and RL. The MDL is defined in 40 CFR Part 136 Appendix A. RL is the lowest calibration standard. Each laboratory will be required to provide MDLs for each non-isotope dilution target compound along with the RLs. Isotope dilution methods quantify non-detects using EDLs as defined in SW-846 Method 8290.

Detection limits for inorganic compound compliance data will be derived according to the referenced analytical method and the laboratory's standard operating procedures. Each laboratory will be required to provide detection limits for each inorganic analyte.

Analytical results that are reported as "Not Detected" (ND) in compliance demonstration samples will be handled in the following manner. The analytical result will be reported as "ND" and the appropriate detection limit as discussed above will be shown. The detection limit will be used as the assumed sample concentration in all subsequent calculations. For analyses of single component samples (e.g., feed or residue samples) that are reported as "ND", the results of all subsequent calculations using those values will be accompanied by a "less than" ("<") sign. For analyses of multi-component samples (e.g., VOST, Method 0010), where the analytical results must be combined for use in subsequent calculations, the detection limit will be used for each component reported as "ND". Results of calculations utilizing values derived from multi-component analyses where the analyte of concern was non-detect in one or more components but not in all of the components, the result will be accompanied by a "<" sign. If the analyte of concern in a multi-component sample is non- detect in all the components of a particular sample, the result will be shown with a "<" sign, and will also be marked as "ND". Where destruction and removal efficiency or similar performance measurements are calculated using emissions rates reported with a "<" value, the resulting performance measurement will be accompanied by a "greater than" (">") sign.

For the purpose of the HHERA, if all results for a compound are found to be non-detect, one-half of the detection limit will be used.

11.3.5 Final Case Files

At a minimum, the following documents will be retained upon the completion of the project in the final case file, which must be maintained at the DESOTEC facility for a period at least three years:

- All legal documents and orders,
- All field documents including those used for preliminary field activities,
- Copies of all analytical data,
- Copies of the final report and background documents, and
- All correspondence relating to the project as well as corrective action requests.

Figure 11-1. Example Performance Test Report Outline

EXECUTIVE SUMMARY

TEST PROGRAM SUMMARY

- Engineering Description
  - General Description
  - Residence Time Determination
  - Burner Description
  - Spent Carbon Feed Systems
  - Auxiliary Fuel System
  - Air Pollution Control System
  - Process Monitoring System (CMS)
  - Continuous Emissions Monitoring System (CEMS)
  - Automatic Waste Feed Cutoff System
- Summary of Test Plan and Objectives
- Test Implementation Summary
  - Deviations from the Test Plan

PROCESS OPERATIONS

- Process Operating Conditions
- Feed Material Characteristics
- Feed Material Spiking
- Effluent Characteristics

COMPLIANCE RESULTS

- POHC Destruction and Removal Efficiency
- Particulate Emissions
- Hydrogen Chloride and Chlorine Emissions
- Metals Emissions
- Stack Gas Oxygen, Carbon Monoxide, and Total Hydrocarbons
- Dioxin and Furan Emissions

QUALITY ASSURANCE/QUALITY CONTROL RESULTS

- QA/QC Activities and Implementation
  - QA Surveillance
  - Sample Collection
  - Sample Analysis
  - Process Instrumentation
  - Stack Sampling Equipment
  - Laboratory Analytical Instrumentation
- Audits and Data Validation
- Calculations
- Conclusions

Figure 11-1. Example Performance Test Report Outline

ANTICIPATED PERMIT OPERATING CONDITIONS

- Confirmation of RCRA Permit Operating Limits
- Recommended Specific Control Parameter Changes

RECOMMENDED EMISSIONS DATA FOR USE IN HHERA

- Metals
- Hydrogen Chloride and Chlorine
- Particle Size Distribution
- Speciated Volatile Organics
- Total Volatile Organics
- Speciated Semivolatile Organics
- Total Semivolatile and Nonvolatile Organics
- Dioxins and Furans
- Speciated PCBs
- Speciated PAHs
- Speciated Organochlorine Pesticides

APPENDICES

A. Process Operating Data
B. Test Manager's Log
C. Spiking Report and Certificate of Analysis for Spiking Material
D. Process Instrument Calibration Data
E. Continuous Emissions Monitoring Data
F. Sampling Report
G. List of Samples
H. Analytical Report
I. Calculations
J. Documentation to Support Metals Extrapolation
K. Data Validation Report
L. Corrective Action Requests

12.0 ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES

12.1 SAMPLING EQUIPMENT

All equipment used in emission testing measuring systems must be maintained in good operating order. To achieve this objective, a routine preventive maintenance program is necessary. Procedures used in this program follow those outlined in Maintenance Calibration and Operation of Isokinetic Source Sampling Equipment, Publication No. APTD-05-76 and Volume III of the Quality Assurance Handbook for Air Pollution Measurement Systems.

The potential impact of equipment malfunction on data completeness is minimized through two complementary approaches. First, an equipment maintenance program is part of routine operations. The maintenance program's strengths include:

- Trained technicians experienced in the details of equipment maintenance and fabrication,
- Adequate spare parts inventory, and
- The availability of tools and specialized equipment.

The second approach is based upon equipment redundancy. Backup equipment, spare parts and tools are included on the materials transported to the field for each sampling task. This approach allows the sampling team to respond to equipment breakage or malfunction in a timely fashion, minimizing the quantity of lost data.

For field equipment, preventive maintenance schedules are based on the results of routine inspections and on accumulated experience. At a minimum, equipment will be inspected prior to the beginning of and at the conclusion of each test. A record of each inspection (Figure 12-1) will be kept as part of the final case file. Maintenance schedules for continuous emissions monitors follow manufacturer's recommendations.

Each item of field test equipment is assigned a unique, permanent identification number. An effective preventive maintenance program is necessary to ensure data quality. Each item of equipment returning from the field is inspected before it is returned to storage. During the course of these inspections, items are cleaned, repaired, reconditioned and recalibrated where necessary. Each item of equipment transported to the field for this test program is inspected again before being packed to detect equipment problems that may originate during periods of storage. This minimizes lost time on the job site due to equipment failure. Occasional equipment failure in the field is unavoidable despite the most rigorous inspection and maintenance procedures. For this reason, adequate spare parts are kept in a central location so the sampling contractor can quickly respond to the job site with replacement equipment for all critical sampling train components.

12.2 LABORATORY INSTRUMENTS

The laboratories perform regular maintenance on all analytical instruments. An inventory of replacement parts is kept to prevent downtime. Manufacturers' service representatives are also contracted, as required, for major instrument repairs.

Preventive and routine maintenance is covered in each of the laboratories' QA Manuals and SOPs or in accordance with manufacturer's recommendations (i.e., instrument manuals). Daily maintenance (such as replacement of injector septa, etc.) is covered in instrument SOPs. Inoperative equipment is tagged as non-usable until repairs are performed. Logbooks are maintained for each instrument to record usage, maintenance, and repairs.

12.3 PROCESS INSTRUMENTS

On-site personnel perform regular maintenance on all process instrumentation. Routine and preventive maintenance procedures are documented and updated as required. Where appropriate, manufacturers' recommendations for maintenance of process instruments are followed. Operators conduct daily reviews of process instrumentation by noting suspicious or inconsistent readings. Maintenance logs are used to record the frequency and type of repairs necessary for process instruments. Process instruments used to demonstrate compliance with operating limits will be calibrated prior to the test. Records of these calibrations will be included in the final test report.

Figure 12-1. Example Equipment Inspection Record Form

Client:_____ **Project No.**_____

Date/Time of inspection: _____

Equipment Inspected:

1) _____

Condition: Good See Problems Section Below

2) _____

Condition: Good See Problems Section Below

3) _____

Condition: Good See Problems Section Below

4) _____

Condition: Good See Problems Section Below

5) _____

Condition: Good See Problems Section Below

Problems Noted:

_____

_____

_____

_____

Action Taken

_____

_____

_____

Inspector's Signature: _____

13.0 ASSESSMENT PROCEDURES FOR ACCURACY, PRECISION, & COMPLETENESS

The QA activities implemented in this study will provide a basis for assessing the accuracy and precision of the analytical measurements. Section 5.0 discusses the QA activities that will generate the accuracy and precision data for each sample type. The generalized forms of the equations that will be used to calculate accuracy and precision are presented below.

13.1 ACCURACY

When a reference standard material is used in the analysis, percent Accuracy (A) will be calculated as follows:

$$A = \frac{\text{Found concentration}}{\text{True concentration}} \times 100$$

Percent analyte Recovery (R) will be calculated as follows:

$$R = \frac{X - N}{S} \times 100$$

Where X is the experimentally determined value, N is the amount of native material in the sample, and S is the amount of spiked material of the species being measured. Recoveries are used to determine accuracy when standards are not available, or are not appropriate for a given matrix.

13.2 PRECISION

When less than three analyses of the same parameter are available, precision will be calculated as a Relative Percent Difference (RPD) from the average of replicate measurements according to:

$$\text{RPD} = \frac{\left(X_1 - X_2\right)}{\text{Average } X} \times 100$$

Where X_1 and X_2 are the highest and lowest results of replicate measurements.

Where three or more analyses of the same parameter are available, the precision will be determined as the Relative Standard Deviation (RSD) according to:

$$\text{RSD} = \frac{\text{Standard deviation}}{\text{Average X}} \times 100$$

13.3 COMPLETENESS

Completeness of data generated from a test program is usually calculated as follows:

$$\%\,\text{Completeness} = \frac{\text{Valid data}}{\text{Expected data}} \times 100$$

Data completeness is defined in Section 5.0 of this QAPP as the percentage of valid data collected from the total number of valid tests conducted.

The purpose of the fourth test run is an allowance for the following during any test run: 1) possible loss or damage to all or portions of any sample(s) or sample fraction(s), 2) rejection of a specific sample(s) due to sampling or analytical data quality reasons, or 3) deviation/closeness to the system operational targets. Desotec's intent is to select three test runs that are 100% complete for demonstrating compliance. Data from the three selected runs, the first three test runs or any combination of three of the four test runs, will be used to demonstrate compliance with the RCRA permit conditions and risk assessment data collection requirements. Should Desotec elect to exclude a test run for Item 3 above, or should there be data quality issues or incomplete samples with a particular sample data set (Item 1 or Item 2 above), valid data for the additional or "extra" test run may be substituted and used for compliance demonstration and/or risk assessment modeling. In the event that conditions (1), (2), or (3) above invalidate or potentially invalidate a test run, Desotec will substitute the entire data set from the additional test run in place of the invalid test run. EPA's approval will be required prior to substituting any portion of a test run. Compliance with the current associated RCRA permit OPLs, or possible establishment of new OPLs, will be reconciled in accordance with 40 CFR 63.1209(i) as may be necessary.

Also refer to Sections 3.2.1 and 5.4 of this QAPP.

14.0 AUDIT PROCEDURES, CORRECTIVE ACTION, AND QA REPORTING

14.1 PERFORMANCE AND SYSTEM AUDITS

This section presents information related to the procedures used by the QA staff to assess conformance of the project staff to the specifications contained in the relevant project controlling documents. Further, auditing may be employed to assess the ability of subcontractors to successfully perform the work.

14.1.1 Field Audits

The Test Manager review the operations at the site to ensure that work is being performed in accordance with the various project controlling documents and associated standard operating procedures. The audit will cover, but not necessarily be limited to, such areas as:

- Conformance to EPA and SW-846 methodologies
- Completeness and accuracy of the sampling documentation
- Chain of custody procedures
- Request for analysis documentation
- Compliance with Health and Safety requirements.

14.1.2 Performance Evaluations

The Regulatory Agencies may provide Performance Evaluation (PE) samples (referred to elsewhere as "Audit Samples") to the laboratory. Any agency-provided PE samples will be submitted for analysis to demonstrate analytical performance on an as required basis.

No Stationary Source Audit Sample Program (SSASP) PE samples are expected to be analyzed during this test program. At the time of this test plan submittal, one of the two previously EPA-approved providers has withdrawn from the program. In accordance with the regulations, EPA has suspended the SSASP until such time a second vendor is qualified and audit samples from at least two vendors are made available (Federal Register, Volume 84, No. 176, Page 47882, September 11, 2019).

14.1.3 Office Audits

The QAO may also conduct audits of the case files. These audits will assess the completeness of the files and verify that all the appropriate information is included in the files.

14.1.4 Laboratory Audits

DESOTEC or its appointed representative may choose to audit the laboratories at any time during the course of the project on an as-required basis to assess the laboratory's ability to successfully perform the work and to ensure mutual agreement between DESOTEC and the laboratory with regard to the scope of work, QA/QC requirements, and deliverable requirements. Reasonable notice will be provided prior to any on-site inspection of the laboratory.

14.2 CORRECTIVE ACTION

The following procedures have been established to ensure that nonconforming conditions, such as malfunctions, deficiencies, deviations and errors are promptly investigated, documented via the Corrective Action Request Form, evaluated and corrected. Every person employed in the test is expected to function as a QC inspector to ensure the quality of the final product. Quality, as it relates to this project, is defined as "performing the work according to the agreed upon specifications contained in the PDT plan and relevant SOPs or causing the specification to be changed *in a controlled manner*." Each individual is encouraged to identify any condition adverse to the successful completion of the work or any modification to the specifications that might result in a better end product. These improvements might be framed in terms of higher quality, greater safety, greater efficiency, and/or lower cost. However, it cannot be stressed strongly enough, that only documented and approved changes to the specifications are allowable.

14.2.1 Field

When a nonconforming condition or an opportunity for improvement is noted at the site or contractor location, the corrective action provisions of this plan will be invoked to identify the condition and recommend corrective action, utilizing the Corrective Action Request form. Condition identification, cause, reference documents and the corrective action planned to be taken will be documented and reported at a minimum to the employee's immediate supervisor.

A Corrective Action Request (CAR), as shown in Figure 14-1, should be used to identify the adverse condition or opportunity for improvement, reference document(s) and recommended corrective action(s). The CAR is directed to the Test Manager. The Test Manager affixes his signature and the date to the corrective action block that states the cause of the condition(s) and corrective action(s) to be taken. The Test Manager is responsible for first notifying the regulatory agency representative of any problems or deviations from the QAPP, or PDT plan identified in the CAR. The Test Manager then forwards the requested response to the QAO for follow-up and filing. The QAO maintains the log for status control of CARs and responses confirms the adequacy of the intended corrective action(s) and verifies its implementation. The QAO will issue and distribute copies of completed CARs to the originator, Test

Manager, DESOTEC Test Project Manager, CRIT representatives, and the involved contractor(s) if any. CARs are transmitted to the project file for future reference, and are incorporated into the final test report.

Testing activities may be impacted by a number of factors, including process interruptions, operating conditions which are outside of specifications, inclement weather, or sampling train difficulties. A set of field troubleshooting guidelines has been developed and presented in Table 14-1 to assist in recognizing and resolving these issues in the field.
In all cases in the QAPP, “Test Manager” will be used consistently (to match the PDT Plan), including Figure 4-1, Table 14-1, and the balance of the QAPP. The following text is proposed to replace Section 14.3 of the QAPP:

Every person active in the PDT program is expected to function as a quality control (QC) inspector to ensure the quality of the final product. This includes every person noted in Figure 4-1 of this QAPP, whether that be DESOTEC or its contractors, CRIT, or EPA and its representatives and contractors. Quality, as it relates to this project, is defined as *"performing the work according to the agreed upon specifications contained in the PDT plan and relevant SOPs or causing the specification to be changed in a controlled manner."* Each individual is encouraged to identify any condition adverse to the successful completion of the work or any modification to the specifications that might result in a better end product. These improvements might be framed in terms of higher quality, greater safety, greater efficiency, and/or lower cost. However, it cannot be stressed strongly enough, that only documented and approved changes to the specifications are allowable. The Corrective Action Request (CAR) procedure is established to ensure that nonconforming conditions, such as malfunctions, deficiencies, deviations and errors are promptly investigated, documented, evaluated and corrected. The CAR form shown as Figure 14-1 provides for the official documentation during the course of testing/sampling, analysis, and test data reduction. The following is the general procedure for executing a CAR:

1. Part I: Any person noted in Figure 4-1 may initiate a CAR. The CAR (for which Part I is completed), shall be presented to the responsible test party (e.g., likely the DESOTEC Plant Manager or Test Manager) to correct the situation.
2. Each CAR shall be concurrently presented by the originator and discussed with the DESOTEC Plant Manager, Test Manager, EPA Observer Team Leader, and CRIT. The test team parties shall discuss the CAR and agree upon the corrective action.
3. Part II: Once the corrective action is agreed upon, the DESOTEC Plant Manager or Test Manager shall complete the *RESOLUTION/RESPONSE* section to document the agreed upon action(s).
4. The CAR Recipient, DESOTEC Plant Manager, EPA Observer Team Leader, and CRIT Representative shall sign in Part II acknowledging agreement as to the corrective action.

5. Final responsibility for ensuring implementation of the corrective action shall reside with the DESOTEC Plant Manager and Test Manager.
6. Part III: Once the corrective action has been resolved, the Recipient shall communicate and/or present evidence to the DESOTEC Plant Manager or Test Manager that the action(s) as required by the CAR have been implemented. The Recipient shall certify such by signing Part III of the form, and returning the form to the DESOTEC Plant Manager or Test Manager.
7. Part IV: The DESOTEC Plant Manager or Test Manager shall present the CAR (via hard and/or electronic copies) to the DESOTEC Plant Manager, EPA Observer Team Leader, and CRIT. The Test Manager shall provide verbal or additional written explanation to these parties as may be necessary.
8. Once all parties are satisfied with the CAR correction/resolution, the designated representative of each party shall sign the Part IV acknowledgement section of the CAR.
9. Copies (hard and/or electronic) of the completed form shall be provided to the DESOTEC Plant Manager, Test Manager or Quality Assurance Officer (QAO), EPA Observer Team Leader, and CRIT.
10. A copy of each completed form(s) shall be included with the final PDT Report as part of the test documentation.

14.2.2 Laboratory

The laboratories' QA Manuals and the related SOPs, contain detailed discussions of corrective actions to be taken if established criteria fail during laboratory analysis. The laboratory has the responsibility to immediately notify the Test Manager and/or QAO when any analytical QC nonconformance occurs, so a mutually acceptable course of action can be pursued.

14.3 QA REPORTS TO MANAGEMENT

The QAO will provide a written report to the Test Manager. This report will address:

- Overview of activities and significant events related to QA/QC
- Summary of audit results
- Review of corrective action request status
- Laboratory QA/QC reports
- Data validation reports
- Summary of significant changes in procedures or QA/QC programs
- Recommendations.

Upon project completion, a Final QA Report will be issued, assessing the overall degree of project conformance to specifications and the impact of any nonconforming conditions on data quality that may affect management decisions. This report will be incorporated into the final test report.

The nature of the laboratories' Quality Assurance reports is provided in their respective Laboratory Quality Assurance Manuals and SOPs. Where no other specifications exist, the laboratory must conform to the provisions given in this section.

Figure 14-1. Example Corrective Action Request Form

CORRECTIVE ACTION REQUEST

Number: _____
Date: _____
Project: DESOTEC – Parker Facility Performance Demonstration Test (PDT)

Part I: *CORRECTIVE ACTION ORIGINATION*

To: _____
_____
_____

You are hereby requested/directed to take the corrective or alternative action indicated below or as otherwise determined and directed by the test team representatives to correct/resolve the noted condition.

Condition:

Reference Documents:

Recommended Corrective Actions:

_____ _____ _____
Corrective Action Originator Date Signature
Printed Name
PDT Organization/Role

Part II: *RESPONSE/RESOLUTION CONCURRENCE*

Cause of Condition:

Agreed Upon Corrective Action Resolution:

Affected Activities/Documents:

The undersigned concur as to the situation and proposed corrective action as documented in Part II.
(include full printed names and signatures)

_____ _____ _____
Recipient Signature Date Signature
Printed Name
PDT Organization/Role

_____ _____ _____
DESOTEC Plant Manager Date Signature

_____ _____ _____
Test Manager or QAO Date Signature

_____ _____ _____
EPA Region 9 Representative Date Signature
(on behalf of the Region 9 Administrator)

_____ _____ _____
CRIT Representative Date Signature

Figure 14-1. Example Corrective Action Request Form (cont'd)

CORRECTIVE ACTION REQUEST (CONTINUED)

Part III: CERTIFYING THE CORRECTIVE ACTION IS IMPLEMENTED AS PER PART II

The undersigned certifies the corrective action has been implemented as directed in Part II except as may be noted below.

Detail of the Corrective Action(s) and Exception(s) (if any):

(include full printed name and signature)

_____	_____	_____
Recipient Signature	Date	Signature
Printed Name		
PDT Organization/Role		

Part IV: CONFIRMATORY SIGNATURES ACKNOWLEDGING AND VERIFYING RESOLUTION

The undersigned concur that the corrective action(s) have been implemented as per Part II above and resolved the situation to the satisfaction of the respective test team parties.

(include full printed names and signatures)

_____	_____	_____
DESOTEC Plant Manager	Date	Signature
_____	_____	_____
Test Manager or QAO	Date	Signature
_____	_____	_____
EPA Region 9 Representative	Date	Signature
(on behalf of the Region 9 Administrator)		
_____	_____	_____
CRIT Representative	Date	Signature

Table 14-1. Performance Test Troubleshooting Guidelines

Condition	Resolution Guidance
Process conditions cannot be maintained within the specifications of the test plan.	Document which parameters need to be modified. If modified conditions will be more conservative (e.g., minimum temperature higher than originally planned, spent carbon feed rates less than originally planned) these modifications should be acceptable. The operator must understand, however, that certain permit limits normally based on actual test conditions may later be changed accordingly. If modified conditions will be less conservative than specified in the approved test plan, specific regulatory agency approval will be needed. In all cases, the on-site regulatory observers should be notified of process operating changes. (Note: This condition can usually be avoided by conducting limited scope miniburns/pretests prior to the formal compliance test.)
Run start/re-start	Prior to a run start, ensure that the process has attained the desired operating conditions and has remained at those conditions (within reasonable bounds of normal process variability) for at least 60 minutes. The 60-minute time allows rolling averages to be reset to the test condition values at the beginning of the run. If spiking is performed, allow spiking systems to operate at test conditions for at least 30 minutes or for the time required to achieve “steady-state” for the process (system specific, usually equal to the solid feed residence), whichever is longer. If a run is stopped due to process deviations, allow the process to operate at desired conditions for at least 30 minutes before re-starting the run. In most cases spiking systems should be operated throughout process upsets. If spiking has been interrupted, allow spiking systems to operate at test conditions for at least 30 minutes or for the time required to again achieve “steady-state” prior to re-starting the run.
Process conditions drift within or between runs	A certain amount of process variability is normal, and will occur due to variability in the feeds, ambient conditions, and process control tolerances. The process should be operated as closely as possible to the target test conditions for each run, and all runs within a given test condition should be conducted at similar conditions so the results of each run within a test conditionare comparable. Normal and expected process variability within a run or between runs is noted in Table 4-2 of the PDT Plan. Wider variability may be acceptable for certain processes, and should be discussed on a case-by-case basis relative to performance or emissions compliance and as noted in the first condition in table 14-1.
Minor process upsets	If the process experiences short-term, minor process upsets, the sampling should continue through the event, and a notation should be made in the test manager’s log. If the process conditions drift by more than the acceptable amounts within a run, and cannot be corrected within a reasonable amout of time, or if conditions between runs do not match within acceptable limits, the testing should be halted until appropriate process conditions are established. An on-going run can normally be resumed after process conditions are corrected (see below for additional guidance on resuming a run). At the discretion of the facility operator, the run may be aborted and repeated at any time.

Table 14-1. Performance Test Troubleshooting Guidelines

Condition	Resolution Guidance
Major process upsets	If the process significantly deviates from the desired target conditions, all testing activities will be halted. Testing may be resumed after the process is stabilized at the test conditions (see below for additional guidance on resuming a run). If two or more major process upsets occur within a run, consideration will be given to aborting and repeating the run. At the discretion of the facility operator, a run may be aborted and repeated at any time. In cases where the major upset or equipment malfunction occurs that requires the testing to be curtialed, samples and data collected from previously completed test runs shall still be considered valid. The additional testing necessary to complete the test program will be perfromed at a later date.
Minor sampling system problems	If a minor problem occurs in an individual sampling system (e.g., filter changeout, full impinger, power problem, temperature problem) the affected sampling system will be temporarily halted until the problem is corrected. All other sampling systems will continue as normal. The run may need to be extended and/or repeated to allow for collection of the appropriate sample volume only in the affected sampling system. (Note: It is not critical that all sampling is conducted concurrently as long as process operating conditions are consistent during the entire run).
Major sampling system problems	If a major problem occurs in an individual sampling system (e.g., broken glassware, failure of control module) or if a problem impacts several sampling systems (e.g., power outage) all sampling will be halted until the problem is diagnosed and an appropriate course of action is determined. If it appears that the problem can be corrected within approximately 60 minutes, all sampling will be suspended until the problem is corrected, and then sampling will resume. If it appears that more than 60 minutes will be needed to correct the problem, the entire sampling program may be suspended until the problem is corrected or the unaffected systems may resume sampling, and the affected system will be resumed after the problem is corrected. The run may need to be extended to allow for collection of the appropriate sample volume in the affected sampling system(s). (Note: It is not critical that all sampling is conducted concurrently as long as process operating conditions are consistent during the entire run).
Temporary halt of sampling	If a temporary halt of sampling is required, the test manager will communicate to all parties that sampling is to be placed “on hold”. Each sampler will note the event and the time on the sample collection sheet, and the test manager will note the event and the time in the test log. Stack sampling trains will be stopped, but will be kept in place unless the halt exceeds (or is expected to exceed) about 15 minutes. If the temporary halt exceeds about 15 minutes, stack sampling train probes will be removed from the stack and the nozzle openings will be capped. Sampling system heating and cooling (as appropriate to each train) will be continued. Process samples will be secured at the sampling location until the test is resumed.

Table 14-1. Performance Test Troubleshooting Guidelines

Condition	Resolution Guidance
Resumption of testing after a temporary halt	Once the condition that caused a temporary halt has been resolved, the test manager (in consultation with the facility operator and the regulatory observers) may either abort the run or may resume it using the following guidance and site-specific considerations. In all cases, the decision to abort or resume will be documented in the test log. If sampling is resumed, each sampler will note the time of sample resumption on the sample collection sheets and the test manager will note the time in the test log. 1. In general, a run can be resumed if it can be completed within the same test day. The samples will not be held overnight and resumed the following day. 2. If the process downtime during a run exceeds 3 hours the run should be aborted unless sound technical justification is provided, and all parties agree. 3. If the reason for the sample interruption was a major process upset, or if the temporary halt exceeds 1 hour, the volatile organic sample resin tube set that was being collected at the time of the test interruption should be discarded, and the test resumed with a fresh tube set. Note that this does not apply to the resin in a semivolatile organic [Modified Method 5 (SW846 Method 0010 and its variants for PCDD/PCDF, etc.] sampling trains.
Disposition of samples from aborted runs	A sample run is aborted because it is deemed in the field that the samples will not be suitable for their intended purpose. In general, samples from aborted runs are discarded, and the run is repeated in its entirety, using the original test run identification. There may be rare circumstances where the samples are recovered and archived for possible analysis. In this case, the samples from the aborted run are assigned their original sample identifications, and the repeat run is assigned a new run identification.
Sample lost during recovery operations	If, after completing a run, a sample is broken, spilled, contaminated, compromised, or otherwise lost, the test manager, facility operator, and regulatory observer must decide on a corrective action to replace the sample. In most cases, and additional test run will be needed to collect a replacement sample, but the entire set of samples collected from the run does not need to be repeated, as long as process conditions are reasonably consistent with those experienced during the original run. For example, if a test run included stack gas sampling for organics, particulate, and metals, and the metals train is dropped and broken during removal from the stack, a repeat run will be needed, but only the metals train needs to be operated during the repeat run. (Note: It is not critical that all sampling is conducted concurrently as long as process operating conditions are consistent during the entire run).
Sample cannot be collected as specified in the approved test plan	This situation can arise due to unique features and circumstances of the process. Appropriate modifications must be discussed and agreed between the test manager, facility operator, and regulatory observer on a case-by-case basis. The test manager will describe the situation and resolution in the test log. The Test Manager, Facility Manager, and Regulatory Observer will approve and sign the test log.

Table 14-1. Performance Test Troubleshooting Guidelines

Condition	Resolution Guidance
Sampling train fails leak check at port change or at test completion (isokinetic samples), or at tube set completion (volatile organic samples)	Leak checks of the isokinetic sampling trains shall be conducted before the run and at run completion as required by the sampling methods. Allowable leakage rates for isokinetic sampling trains is 0.02 cfm at a vacuum equivalent to or higher than the sampling condition. The volatile organic sampling trains (VOST) is leak checked before and after each tube set. Allowable leakage rate for Method 0030 is 2.5 mm Hg per minute. Allowable leakage rate for Method 0031 is 0.02 liters per minute for sampling rates of 1 liter per minute, and 0.01 liters per minute for sampling at lower rates. Leaks found before sampling will be corrected before sampling begins. Isokinetic sampling trains must be moved from one port to the next to complete the required sampling traverses. The handling of the sampling train moving from one port to the next can loosen probe, filter holder, and impinger connections. Additional leak checks of isokinetic sampling trains may be performed pre-port change and post-port change. Pre-port change is after the probe is withdrawn from the port at the end of a traverse and before the samplng train is moved to the next port. Post-port change is after the sampling train is moved to the next port to start the next traverse and before the probe is re-inserted to the port. If the post-port change leak check fails but a pre-port leak check was perfromed and was passing, such will not be considered a “leak”. In such cases, the sampling team may take actions to isolate and remediate the leak. Once the cause of the leak is adressed and the samplng train passes leak check, sampling may be resumed If an isokinetic sampling train fails a leak check, the sampling team should isolate the train sections and determine where the leak occurred. In some cases it may also be possible to determine or know when the leak problem occurred. If it can be determined that the leak occurred during the port change and can be remediated as described above, the sampling train will still be considered valid. If the leak occurred after the sampling was completed, or a leak check can only be perfromed in a limited fashion, e.g., due to the glass nozzle or glass probe liner being broken or the probe inadvertantly disconnecting from the sampling train as the probe was withdrawn from the port, the test will still be considered valid In cases where the glass nozzle or glass probe liner was broken or the probe inadvertantly disconnected from the sampling train, provided a leak check from the probe connection through the impingers is passing, the sampling train will still be considered valid.

Table 14-1. Performance Test Troubleshooting Guidelines

Condition	Resolution Guidance
Sampling train fails leak check at port change or at test completion (isokinetic samples), or at tube set completion (volatile organic samples) (continued)	In other instances, if the leak is small (less than 2x the allowable leakage rate) and occurred at a location inside the stack (probe nozzle assembly) it may be justified to keep the sample, pending concurrence from the regulatory observer. In-stack leakage can be confirmed by checking the collected moisture content from the train in question compared to other trains from the same run or from other runs within the same test condition. If it is decided to keep the sample, the portion of the run during which the leak occurred should be corrected for the leak (See procedure in EPA Method 5) even though the leak appeared to have been inside the stack, and the isokinetic sampling rate should be confirmed with the sample volume correction. This procedure is conservative since it yields a smaller sample volume than was actually collected. Leaks larger than 0.02 cfm which are found to have been outside the stack will result in rejection of the sample unless sound technical justification is provided and all parties agree. If an isokinetic samplng train fails the leak check at the completion of a test run, the sampling train will be voided. The sampling train may be replaced by: 1. Setting up a new sampling train and extending the test run time to allow for collection of the a replacement sample, or 2. Performing an additional test run at a later time (e.g., after Run 3) with the same process operating conditions and with the sampling limited to the previously voided sampling train(s) If a volatile organic sample fails the leak check at the completion of a tube set, the tube set will be discarded and an additional set will be collected during the run as a replacement. The run time may need to be extended to allow for collection of the appropriate sample volume and number of tube sets in the affected sampling system(s).