#### QUALITY ASSURANCE PROJECT PLAN IRON AND STEEL FOUNDRIES MANUAL EMISSION TESTING CUPOLA WET SCRUBBER IN MICHIGAN

# Prepare for:

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September 1997 p:\s414\s414.002\smco\sectal.qap (wpd)

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# APPROVAL SHEET EPA Contract 68 D20162, Work Assignment No. 4-14 Quality Assurance Project Plan September 11, 1997, Revision No. 0

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#### 1.0 PROJECT ORGANIZATION

Figure 1.1 illustrates the program organization for the iron and steel emissions test program. Each organization and individual shown in Figure 1.1 is described in the following subsections.

#### 1.1 Pacific Environmental Services, Inc. (PES)

As the primary contractor, PES will provide overall project management and supervision for each aspect of the project. PES is a nationwide environmental engineering and industrial hygiene firm. PES is in the fifth year of a five year contract with the U.S. Environmental Protection Agency (USEPA) to provide technical support in the area of air emissions testing.

#### 1.1.1 Project Manager

The project manager has ultimate responsibility to the USEPA for the successful completion of this project. The PM will oversee preparation and planning, on site testing and sample recovery, analysis and reporting. He will be responsible for coordinating activities with the subcontractors, facility personnel, regulatory personnel, and other PES employees. The PM for this project is Daniel Sheffel. Mr. Sheffel has 17 years of experience in the environmental sampling field. Mr. Sheffel has participated in a variety of compliance projects, plant inventories, enforcement actions, technology evaluations, and method development programs.

#### 1.1.2 OA/OC Officer

The QA/QC officer has the responsibility to insure the test program (including design, sampling, and analysis) follows this QAPP and good engineering and laboratory practices. In addition, the QA/QC officer is responsible for insuring proper reporting of QC check results and any anomalies during the test program. The QA/QC Officer for this project is Dr. Wayne Westbrook. Dr. Westbrook has over 11 years' experience executing projects following strict QA/QC requirements. He has been managing environmental measurement, laboratory programs, data analysis, and computer simulation projects for more than 23 years.

#### 1.2 Atlantic Technical Services (ATS)

As a subcontractor, ATS will provide experienced personnel to operate sampling equipment during the testing phase of this project. ATS is an environmental manpower company

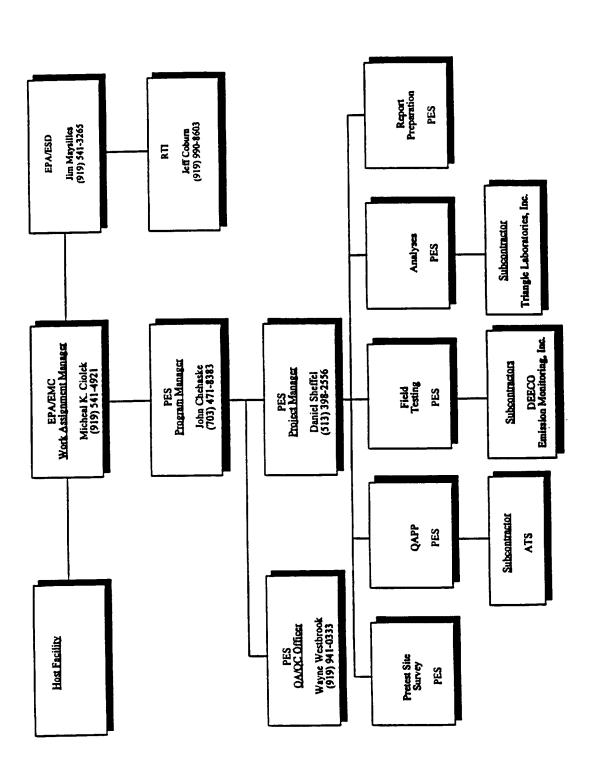


Figure 1.1 Project Organization

providing experienced testing personnel to consulting firms around the nation. ATS testing personnel have a minimum of five years of manual testing experience. The ATS corporate office is located in Chapel Hill, North Carolina.

#### 1.3 DEECO

As a subcontractor, DEECO will provide experienced personnel and sampling equipment during the testing phase of this project. DEECO is an environmental firm providing air emissions testing services to a variety of source types including: chemical manufacturing, wood processing, cement manufacturing, hazardous waste incineration. DEECO corporate offices are located in Raleigh, North Carolina.

#### 1.4 Triangle Laboratories. Inc. (TLI)

As a subcontractor, TLI will provide reagents and analytical services for the project. Triangle Laboratories, Inc. is an analytical laboratory providing nationwide service to the pharmaceutical industry, the medical community, environmental firms and many others. TLI corporate offices are located in Research Triangle Park, North Carolina.

#### 1.5 U.S. Environmental Protection Agency

The USEPA is funding this study. They will use this data for development of the MACT rule.

#### 1.5.1 Work Assignment Manager (WAM)

The WAM is the USEPA counterpart to the PES project manager. The WAM and the PM will work in coordination to insure all parties are properly prepared and informed. The WAM for this project is Michael K. Ciolek of EPA's Emission Measurement Center.

#### 1.6 Host Facility, Michigan

As the host facility, it will be the foundry's responsibility to provide safe access to the test locations, proper utilities for the test crew and proper operation of the unit to be tested. The foundry will provide records of process monitors during the testing portion of the program for inclusion into the report.

#### 2.0 PROBLEM DEFINITION

A review of emissions information on iron and steel foundries shows that cupolas and pouring, cooling, and shakeout (PCS) lines at sand molding operations are a major source of Hazardous Air Pollutants (HAPs) emissions from these facilities. Primary emissions are typically controlled by either a fabric filter (baghouse) or a wet scrubber. The USEPA Emissions Standards Division (ESD) is therefore investigating both types of control devices as a potential Maximum Achievable Control Technology (MACT) floor technology for cupolas. Two facilities have been selected by ESD as host facilities for the testing program. One uses a baghouse for control of cupola emissions, and the other uses a wet scrubber for the control of cupola emissions. The subject of this Quality Assurance Project Plan (QAPP) is the testing program at the facility which uses a wet scrubber. A separate QAPP will address the testing program at the other facility.

The data acquired will be used to address the following program requirements:

- (1) characterize emissions of HAPs from cupolas that are controlled by wet scrubber;
- (2) characterize uncontrolled emissions of HAPs from pouring, cooling, and shakeout processes;
- (3) determine wet scrubber performance in controlling emissions of HAPs from cupolas;
- (4) identify surrogates for estimating HAP emissions from the subject foundry processes.

The PES test program outlined in this QAPP will incorporate collecting data using manual test methods and direct interface GC/MS for requirements (1) and (3). To this end, emissions testing for HAPs will be conducted at the inlet and outlet of the cupola wet scrubber.

Note: Data gathering for requirements (2) and (4) will be performed by a different EPA contractor using Fourier transform infrared spectroscopy (FTIRS) and continuous emission monitors (CEMs) at the PCS lines. Information on the FTIRS and CEMs test program will be provided by the other contractor and is not a part of this document.

#### 3.0 PROJECT DESCRIPTION

The USEPA's ESD is investigating iron and steel foundries to identify and quantify HAPs emitted from cupolas, electric arc furnaces, and pouring, cooling and shakeout operations of sand mold casting processes. ESD has requested EPA's Emission Measurement Center (EMC) to conduct the required testing. EMC has issued a work assignment to PES to plan and conduct the air emissions test program to gather emissions data to satisfied program requirement (1) and (3) (See Section 2.0). The testing program will be conducted through EPA Contract No. 68D20162, Work Assignment No. 4-14. Table 3.1 outlines the project schedule.

#### 3.1 Objective

The primary objective will be to characterize emissions of HAPs from cupolas, electric arc furnaces, and pouring, cooling and shakeout operations of sand mold casting processes. A secondary objective will be to assess the performance of fabric filters and wet scrubbers in controlling primary emissions of HAPs from cupolas. The data will be used by ESD to set emission limits under the MACT program. This project differs from other test programs in that this is a data gathering effort and there is no pass/fail or major/minor sources determination to be made from the test results. As no data quality objectives have been set by the EPA, PES will compare QA/QC results against criteria set in this document to determine the accuracy and representativeness of the data collected.

#### 3.2 Testing

Testing will be performed at the exit of the cupola scrubber for the following pollutants: dioxins and furans as per EPA Method 23, particulate matter (PM) and HAPs metals as per EPA Method 29, semivolatile organics and polyaromatic hydrocarbons (PAHs) as per SW-846 Method 0010 (MM5), and volatile organics by direct interface GC/MS. These tests are considered critical to the success of the test program.

Testing will also be performed at the inlet of the cupola scrubber for PM and HAPs metals as per EPA Method 29 and volatile organics by direct interface GC/MS. This testing will be simultaneous with the metals testing at the outlet. This test is considered important information by ESD but non-critical to the test program.

The test methods performed in this program will comply with the technical criteria described in the reference methods (See Appendix A) and the quality standards described in this document.

#### 3.3 Training

Personnel involved in this project have been trained in their tasks and have from two to 18 years of experience in the duties they will be performing. GC/MS operators have more than 15 years of experience including method development for direct interface GC/MS. The test crew will be subject to onsite surveillance from PES QA personnel with appropriate corrective action enforced if necessary.

#### 3.4 Records

Records for this project will include a bound field test log, progress reports, and QA reports to management. These records and the final report will be subject to an internal review before submission to the EPA.

TABLE 3.1
PROJECT SCHEDULE

DATE	ACTIVITY				
Friday, 9/12/97	Submit QAPP				
Friday, 9/19/97	Approval of QAPP				
Monday, 9/22/97	Set Up Equipment				
Tuesday, 9/23/97	Begin Test Program				
Friday, 9/26/97	Finish Test Program				
Monday, 9/29/97	Inventory Samples				
Tuesday, 9/30/97*	Transfer Samples to Laboratory				

<sup>\*</sup> Work after 9/30/97 will require a different EPA contract mechanism.

#### 4.0 PROJECT QUALITY OBJECTIVES

The overall objective of this test program is to quantify the controlled and uncontrolled HAPs emissions from the cupola process at an iron and steel foundry during normal operation. To obtain data which will satisfy this objective, the test design must provide enough representative data to be statistically useful and standards for accuracy, precision and completeness of data must be met. Section 8.0 discusses the specific quality control procedures and criteria used to determine compliance with the project quality objectives.

The representative nature of the samples are ensured by their collection at isokinetic conditions. Each sample will be collected at the same velocity, within a 10% margin, as the flue gas. This allows sampling the gas without disturbing the natural flow. In addition, the samples will be taken along the cross section of the duct work to account for any stratification. The samples will be taken over a 240 minute period. One run of each test method will be performed each day. This will account for fluctuations over time.

Each test method will be performed in triplicate. If any tests need to be aborted, a replacement test will be performed to ensure three data points for each parameter. USEPA has established three test runs as the minimum number for statistical purposes.

The QA objectives for accuracy, precision and completeness appear in Table 4.1.

TABLE 4.1

QUALITY ASSURANCE OBJECTIVES

SAMPLE	PARAMETER	PRECISION	ACCURACY	COMPLETENESS
SW-846 M0010	SVOHAPs	<50% RPD	50%-150%	90%
	PAHs	<50% RPD	50%-150%	90%
EPA M23	PCDD/PCDF	<50% RPD	50%-150%	90%
EPA M29	Metals	<35% RPD	70%-130%	90%

Note: Precision refers to the repeatability of a result. This is measured in relative percent difference (% RPD). Accuracy refers to closeness of the result to actual values. Completeness refers to the number of valid results compared to the number required by the sampling program. See Section 15 for a detailed description of how these values are calculated.

#### 5.0 DOCUMENTATION AND RECORDS

#### 5.1 Field Operation Records

Documentation while in the testing phase will use pre-formatted forms. Test personnel will record data on a "Field Data Sheet." An example appears as Figure 5.1. The field data sheet provides for documentation of time, date, location, stack gas parameters, sample equipment parameters, and ambient conditions. Field laboratory personnel will record data on a "Sample Recovery Data Sheet." An example appears as Figure 5.2. The sample recovery data sheet provides for documentation of moisture catch, sample description, and sample labeling. Quality control samples such as field and reagent blanks are also documented on the sample recovery data sheet. Chain of custody will be filled out for each sample component recovered. An example appears as Figure 5.3. If corrective action is required during any segment of the test phase, the reason for the correction and the action taken will be documented on the "Corrective Action Report." An example appears as Figure 5.4. All four of these forms will be written in indelible ink. If correction is required on the form, a single line will be made through the error and the correction will be dated and initialed. Any blank spaces will have a line drawn through to ensure it is not later filled in.

Field personnel will have access to general field procedures in the form of expanded EPA reference methods. If any procedure becomes unclear the test crew can refer to these as a step by step reference.

#### 5.2 Laboratory Records

TLI will provide sample tracking forms, case narratives describing any anomalies and any modifications to procedures, data handling records, and lab notes, for inclusion in the final report.

#### 5.3 Final Report

The final report will include all raw data and records. A summary of any outliers or findings will be presented in the report. The report will undergo an internal review before submittal. After submittal, the report will be filed at PES for a period of no less than three years. The file will also include disk copies of all electronic data used in the development of the report.

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### METHOD 23 CDD/CDF SAMPLE RECOVERY DATA

Plant:					Run No	<b>.:</b>	
Sample Da	te:		Filter No.(s):		Job No	••	
Sample Lo	cation:		·				
Recovery D	Date:		XAD-2 Trap N	o.(s):			
Sample Re							
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Impingers	XAD - 2 Trap	1	2	3 (100 ml H2O) (tipped)	4	Silica gel	
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Silica Gel:		Used -	Color -	%	Spent -		
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Figure 5.2 Sample Recovery Data Sheet

# ANALYTICAL REQUEST AND CHAIN OF CUSTODY

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RECOVERY PERSON:					/						_/				COMMENTS
MAPLE TECHNICIAN				11 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	{/	/			/	/	/			/	(Type of container, apaciel preparation, special handling, etc.)
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Originator:	Date:
Project Number:	Corrective Action Number:
Description of Problem (Give Date and Time Identified)	State Cause of Problem
State Corrective Action Planned (Include Persons Involved in Action)	QA Officer Comments:
Signatures	Project Manager Comments:
QA Officer	
Project Manager	
Originator	

Figure 5.4 Corrective Action

#### 6.0 SAMPLING AND ANALYSIS DESIGN AND PROCEDURES

The test program is designed to characterize HAPs emissions from the selected facility. The methods chosen are appropriate to the source type and can achieve sufficiently low limits of detection. The inlet and the outlet metals testing will be performed simultaneously to properly ascertain scrubber efficiency.

Another contractor for the EPA's ESD will be responsible for recording process and control device operating parameters during testing. The ESD representative will be responsible for monitoring plant processes and control device operations to insure that these processes and operations are being conducted under representative conditions. PES will coordinate with the ESD and their other contractor to ensure that testing is being performed under representative conditions and that all parties are kept informed as to the status of the test program.

#### 6.1 Sample Locations

Source sampling will be conducted at two locations, the inlet and outlet of the cupola scrubber.

#### 6.2 Sampling and Analytical Procedures

Source sampling will be performed at the inlet and outlet of the cupola scrubber to determine the concentrations and emission rates of PM, dioxin/furans, and metal and organic HAPs. For each sampling location, concentrations will be presented in microgram per dry standard cubic feet, nanograms per dry standard cubic meter, parts per million or parts per billion and emission rates will be presented in pounds per hour. A tentative daily test schedule is presented in Table 6.1. For each sampling location, the parameters to be measured, the test method, the number of tests to be performed, and the duration of each test are summarized in Table 6.2. Brief descriptions of the sampling and analysis procedures to be used are presented below. Copies of all the methods to be used are presented in Appendix A.

#### 6.2.1 Location and Measurement Sites and Sample/Velocity Traverse Points

EPA Method 1, "Sample and Velocity Traverses for Stationary Sources," will be used to establish velocity and sample traverse point locations. The process ductwork, and the locations of measurement sites and traverse points are discussed in the Site-Specific Test Plan.

# TABLE 6.1 FIELD TEST SCHEDULE

Date	Activity					
Sunday (pm), 9/21/97	Crew arrives from Cincinnati, OH					
Monday, 9/22/97	Set up Test Equipment					
Tuesday, 9/23/97	Prepare for and conduct one test run of each of the following sample methods:					
	Cupola Scrubber Inlet					
	EPA Method 29 (240 min duration)					
	Cupola Scrubber Outlet					
	SW-846 M0010 (240 min duration)					
	EPA Method 23 (240 min duration)					
	EPA Method 29 (240 min duration)					
Wednesday, 9/24/97	Prepare for and conduct one test run of each of the following sample					
W Canada ay y s = -	methods:					
	Cupola Scrubber Inlet					
	EPA Method 29 (240 min duration)					
	Cupola Scrubber Outlet					
	SW-846 M0010 (240 min duration)					
	EPA Method 23 (240 min duration)					
	EPA Method 29 (240 min duration)					
Thursday, 9/25/97	Prepare for and conduct one test run of each of the following sample methods:					
	Cupola Scrubber Inlet					
	EPA Method 29 (240 min duration)					
	Cupola Scrubber Outlet					
	SW-846 M0010 (240 min duration)					
	EPA Method 23 (240 min duration)					
	EPA Method 29 (240 min duration)					
Friday, 9/26/97	Equipment Pack Up and Travel					

TABLE 6.2

SUMMARY OF SAMPLING LOCATIONS, TEST PARAMETERS, TEST METHODS,
AND NUMBER AND DURATION OF TESTS

Sampling Location	Parameter	EPA Test Methods	Number of Tests	Test Duration, (minutes)
Cupola Scrubber Inlet	Flue Gas Flow Rate	EPA Method 2	3	240
	Percent CO <sub>2</sub> , O <sub>2</sub>	EPA Method 3B	3	240
	Moisture Content	EPA Method 4	3	240
	PM / Metals Concentrations	EPA Method 29	3	240
Cupola Scrubber Outlet	Flue Gas Flow Rate	EPA Method 2	3	240
	Percent CO <sub>2</sub> , O <sub>2</sub>	EPA Method 3B	3	240
	Moisture Content	EPA Method 4	3	240
	SVOHAPs and PAHs Concentration	SW-846 0010	3	240
	Dioxins and Furans Concentrations	EPA Method 23	3	240
	PM / Metals Concentrations	EPA Method 29	3	240

## 6.2.2 Determination of Stack Gas Volumetric Flow Rate

EPA Method 2, "Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)," will be used to determine stack gas velocity. The stack gas volumetric flow rate will be calculated from the results of the velocity traverses, along with the stack gas composition and moisture content data.

A Type S pitot tube, constructed according to Method 2 criteria and having an assigned coefficient of 0.84, is connected to an inclined-vertical manometer. The pitot tube is inserted into the stack and the velocity pressure ( $\Delta p$ ), is recorded at each traverse point. The effluent gas temperature is also recorded at each traverse point using a Type-K thermocouple. The average stack gas velocity is calculated from the average square roots of the velocity pressure, average stack gas temperature, stack gas molecular weight, and absolute stack pressure. The volumetric flow rate is the product of velocity and the stack cross-sectional area of the duct at the sampling location.

### 6.2.3 Determination of Stack Gas Dry Molecular Weight

EPA Method 3B, "Gas Analysis for the Determination of Emissions Rate Correction Factor or Excess Air," will be used to determine carbon dioxide  $(CO_2)$  and oxygen  $(O_2)$  content of the flue gases. Gas samples will be extracted from each duct simultaneous with one of the isokinetic sample trains using the multi-point, integrated bag sampling technique.

### 6.2.4 Determination of Stack Gas Moisture Content

EPA Method 4, "Determination of Moisture Content in Stack Gases," will be used to determine the flue gas moisture content. EPA Method 4 will be performed in conjunction with each of the following: SW-846 0010, EPA Method 23, and EPA Method 29. Integrated, multipoint, isokinetic sampling will be performed. Condensed moisture will be determined by recording pre-test and post-test weights of the impingers, reagents, and silica gel. The XAD® -2 sorbent trap from the SW-846 0010 and EPA Method 23 sample trains will also be weighed. At each location, there is a possibility of water droplets. Therefore, the moisture content will also be calculated based upon saturation at stack gas temperature and pressure. The lower of the two moisture values will be used.

# 6.2.5 <u>Determination of Semi-volatile organic hazardous air pollutants (SVOHAPs) and Polvaromatic Hydrocarbons (PAHs)</u>

Method SW-846 0010 "Modified Method 5 Sampling Train (MM5)," will be used to collect semi-volatile organic hazardous air pollutants (SVOHAPs) at the cupola scrubber outlet. A multi-point, integrated sample will be extracted isokinetically from the 24 traverse points. At each traverse point, sampling will be performed for 10 minutes for a total run time of 240 minutes per test. Readings will be taken every 5 minutes. The Method SW-846 0010 samples will be extracted through a glass nozzle, a heated glass lined probe, a treated glass mat filter, a water cooled condenser coil and a sorbent trap containing approximately 40 g of XAD®-2 sorbent resin. A schematic of the SW-846 M0010 sampling train is shown in Figure 6.2.

The MM5 samples will be extracted following the procedure of Method 3542, dated January 1995. The MM5 samples will be analyzed in accordance with the guidelines of Method 8270 by High Resolution Gas Chromatography / Low Resolution Mass Spectrometry. The three sample fractions for each test run, i.e., front half extract, the back half extract (XAD<sup>©</sup>-2 and back half rinse), and the condensate extract will be combined for one analysis per test run. The samples will be analyzed for the presence of the analytes shown in Table 6.3. The detection limit for almost all of the analytes is 10 ug per sample or 3.3 ug/m³ for a three cubic meter sample. Pentachlorophenol and 4-Nitrophenol have a detection limit of 25 ug per sample or 8.3 ug/m³ for a three cubic meter sample.

As stated previously, this data is critical to the success of the test program.

#### 6.2.6 Determination of Dioxins and Furans

EPA Method 23, "Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources" will be used to collect dioxins and furans at the outlet of the cupola scrubber. A multi-point integrated sample will be extracted isokinetically from the 24 traverse points. At each traverse point, sampling will be performed for 10 minutes for a total run time of 240 minutes per test. Readings will be taken every 5 minutes. The EPA Method 23 samples will be extracted through a glass nozzle, a heated glass lined probe, a treated glass mat filter, a water cooled condenser coil and a sorbent trap containing approximately 40 g of XAD®-2 sorbent resin. A schematic of the EPA Method 23 sampling train is shown in Figure 6.2. As stated previously, this data is critical to the sucess of the test program.

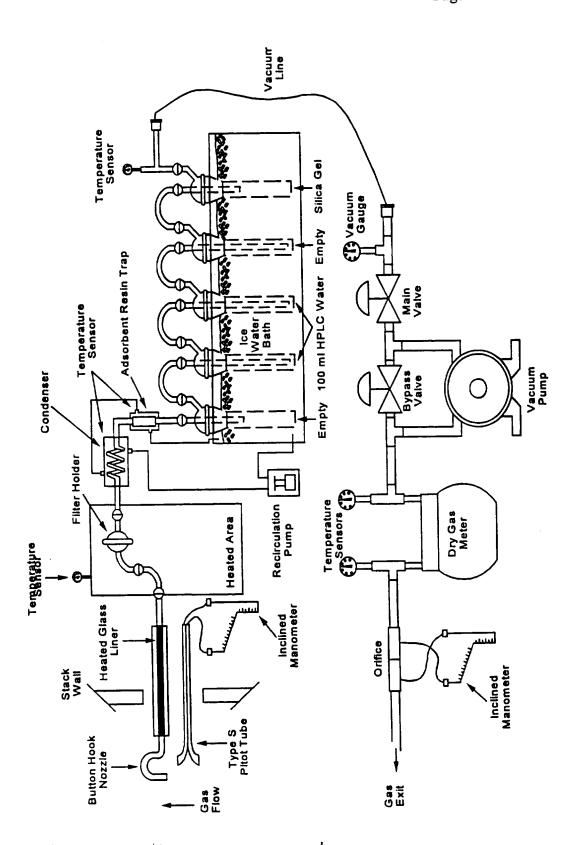


Figure 6.1 Modified Method 5 or Method 23 Sampling Train

TABLE 6.3

TARGET LIST OF CLEAN AIR ACT SVOHAPS

Acetophenone	3,3-Dichlorobenzidine	Methylene bis-chloroaniline	
4-Aminobiphenyl	bis-(2-Chloroethyl)ether	4,41-Methylenedianiline	
Aniline	N,N-Diethylaniline	Naphthalene	
o-Anisidine	N,N-Dimethylaniline	Nitrobenzene	
Benzidine	3,31-Dimethoxybenzidine	4-Nitrobiphenyl	
a,a,a-Trichlorotoluene	Dimethylaminoazobenzene	4-Nitrophenol	
Benzylchloride	Dimethylphthalate	N-Nitrosodimethylamine	
Biphenyl	4,6-Dinitro-2-methylphenol	N-Nitrosomorpholine	
bis(2-Ethylhexyl)phthalate	2,4-Dinitrophenol	Pentachloronitrobenzene	
2-Chloroacetophenone	2,4-Dinitrotoluene	Pentachlorophenol	
2-Methylphenol	3,31-Dimethylbenzidine	Phenol	
3/4-Methylphenol	Hexachlorobenzene	1,4-Phenylenediamine	
Cumene	Hexachlorocyclopentadiene	o-Toluidine	
Dibenzofuran	Hexachlorobutadiene	1,2,4-Trichlorobenzene	
,2-Dibromo-3-chloropropane	Hexachloroethane	2,4,5-Trichlorophenol	
Di-n-butylphthalate	Hydroquinone	2,4,6-Trichlorophenol	
1,4-Dichlorobenzene	Isophorone	Trifluralin	

TABLE 6.4

### TARGET LIST OF PAHS

Acenaphthene	Acenaphthylene	Fluorene
Phenanthrene	Anthracene	Fluoranthene
Pyrene	Benzo(ghi)perylene	Benz(a)anthracene
Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene
Benzo(a)pyrene	Dibenz(a,h)anthracene	Indeno(1,2,3-cd)pyrene
Naphtlene	2-Methylnaphthalene	2-chloronaphthalene
Acenaphthene	Fluorene	Acenaphthylene
Flouranthene	Phenanthrene	Anthracene
Pyrene	Benzo(b)fluoranthene	Perylene
Benzo(b)fluoranthene	Benzo (e) pyrene	

Sample recovery will follow procedures written in the May 31, 1995 Federal Register. The samples will be extracted and analyzed according to EPA Method 23. The sample components (filter, XAD<sup>®</sup>, and rinses) are Soxhlet extracted and combined. The sample is then split with half being archived and the other half analyzed. Analysis is performed on a high resolution gas chromatography/mass spectrometry (GC/MS).

#### 6.2.7 Determination of Particulate Matter and Trace Metals

EPA Method 29 will be used to collect PM and metals at the inlet and outlet of the cupola scrubber. A multi-point integrated sample will be extracted isokinetically from the 12 traverse points at the inlet and 24 traverse points at the outlet. At each traverse point, sampling will be performed for 10 minutes at the inlet and outlet for a total run time of 240 minutes per test. Readings will be taken every 5 minutes. The EPA Method 29 samples will be extracted through a glass nozzle, a heated glass lined probe, a tared quartz mat filter, 2 impingers containing  $H_2O_2/HNO_3$  and 2 impingers containing acidic KMnO<sub>4</sub>. A schematic of the EPA Method 29 sampling train is shown in Figure 6.3. The nozzle will be attached to the probe without the use of metal unions. Teflon® or Teflon® coated unions with Teflon® or graphite ferrules will be used.

Before performing the metals analysis, the filter sample and front half acetone rinse will be weighed for particulate loading according to EPA Method 5. For each sampling train sample run, seven individual analytical samples are generated; five for Hg, and two for all other metals. Samples are analyzed by SW-846 Method 6010 inductively coupled argon plasma (ICAP) and SW-846 Method 7470 cold vapor analysis (CVAAS) will be used for Hg analysis. The samples will be analyzed for the following metals: antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), manganese (Mn), mercury (Hg), nickel (Ni), phosphorous (P), selenium (Se), silver (Ag), thallium (Tl), and zinc (Zn).

A flow chart showing how each fraction is analyzed is presented in Figure 6.4. Tables 6.5 and 6.6 show expected detection limits.

As stated previously, the metals emissions data at the scrubber outlet is critical to the success of the test program. The metals emissions data at the scrubber inlet is important information but non-critical to the success of the test program.

TABLE 6.5
EPA METHOD 29 ANALYTICAL DETECTION LIMITS

Metal	RDL (µg/mL)	Analysis Fractions				Total Detectable	
		1 (µg)	2 (μg)	3 (µg)	4 (μg)	5 (μg)	Amount (µg)
As	0.009	1.8	2.03	NA	NA	NA	3.93
Ba	0.004	0.8	0.9	NA	NA	NA	1.7
Be	0.002	0.4	0.45	NA	NA	NA	0.85
Cd	0.002	0.4	0.45	NA	NA	NA	0.85
Со	0.002	0.4	0.45	NA	NA	NA	0.85
Cu	0.011	2.2	2.49	NA	NA	NA	4.69
Cr	0.007	1.4	1.58	NA	NA	NA	2.98
Pb	0.004	0.8	0.9	NA	NA	NA	1.7
Sb	0.009	1.8	2.03	NA	NA	NA	3.83
Mn	0.004	0.8	0.9	NA	NA	NA	1.7
Ni	0.007	1.4	1.58	NA	NA	NA	2.98
P	0.067	1.34	1.51	NA	NA	NA	2.84
Ag	0.004	0.8	0.9	NA	NA	NA	1.7
Hg	0.0002	0.4	0.6	0.2	0.6	1.0	2.8
Se	0.011	2.2	2.49	NA	NA	NA	4.69
Tl	0.011	2.2	2.49	NA	NA	NA	4.69
Zn	0.004	0.8	0.9	NA	NA	NA	1.7

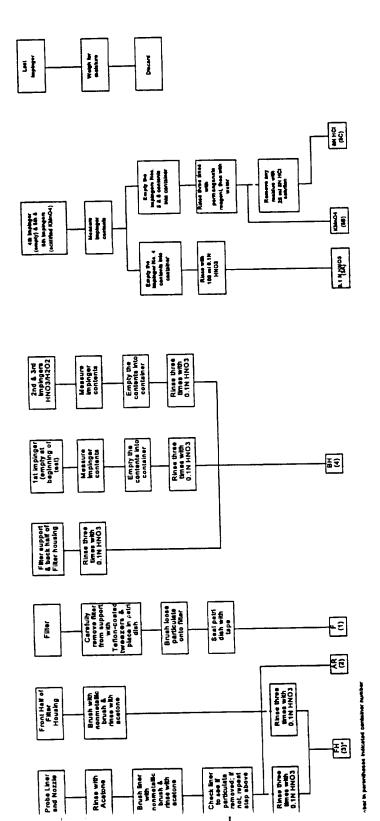
Note: Mercury (Hg) analysis by CVAAS Method 7470A, all others by Method 6010A (ICAP). CVAAS assumes an analysis volume of 10 mL.

 $\mu g = micrograms$ 

TABLE 6.6
EPA METHOD 29 ESTIMATED IN-STACK DETECTION LIMITS

Sampling Volume	3 m³		
Metals	MDL, μg/m³		
Hg	0.933		
As	1.31		
Ва	0.567		
Ве	0.283		
Cd	0.283		
Cr	0.993		
Cu	1.563		
РЬ	0.567		
Sb	1.277		
Со	0.283		
Mn	0.567		
Ni	0.993		
P	13.4		
Se	1.563		
Ag	0.563		
Tl	1.563		
Zn	0.567		

 $m^3$  = cubic meter



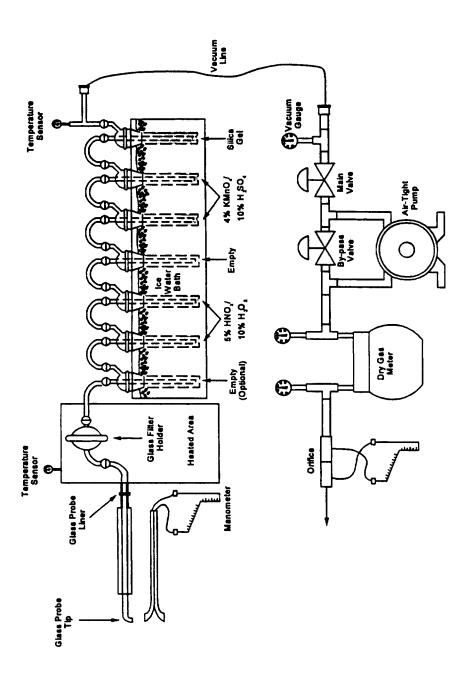


Figure 6.2 EPA Method 29 Sampling Train

#### 6.2.7 Determination of Volatile Organics

Direct Interface GC/MS will be used to monitor volatile organic hazardous air pollutants (VOHAPs) at the inlet and outlet of the cupola wet scrubber. A single point contant rate sample will be drawn from the gas streams using a heated probe and heated line, to prevent condensation. A conditioning unit will be used to protect the GC/MS from particulate matter and excess moisture. The gas chromatography (GC) portion of the GC/MS separates the gaseous mixtures of molecules by their affinity for the analytical column's stationary and mobile phases. The mass spectrometer (MS) portion of the GC/MS uniquely identifies each molecule by detecting individualized fragments. Complete details of the test method are found in Appendix A.

Figure 6.5 is a schematic of the sample interface system. Table 6.7 is a list of target compounds identifiable by the direct interface GC/MS.

TABLE 6.7

TARGET LIST OF VOLATILE HAZARDOUS AIR POLLUTANTS IDENTIFIABLE BY GC/MS

Bromodichloromethane	Bromomethane	Vinyl acetate	
Methyl iso-Butyl Ketone	Chloromethane	Vinyl chloride	
Ethyl chloride	1,1,1-Trichloroethane	1,1-Dichloroethene	
Benzene	Methylene chloride	m-, p-Xylene	
Bromoform	1,2-Dichloropropane	o-Xylene	
Methyl Ethyl Ketone	Styrene	Dibromochloromethane	
Carbon disulfide	1,1,2,2-Tetrachloroethane	cis-1,2-Dichloroethene	
Carbon tetrachloride	Tetrachloroethylene	trans-1,2-Dichloroethene	
Chlorobenzene	Toluene	2-Hexanone	
Chloroform	1,1,2-Trichloroethane	Ethyl benzene	
Cumene	Trichloroethene	1,1-Dichloroethane	
cis-1,3-Dichloropropene	trans-1,3-Dichloropropene	1,2-Dichloroethane	

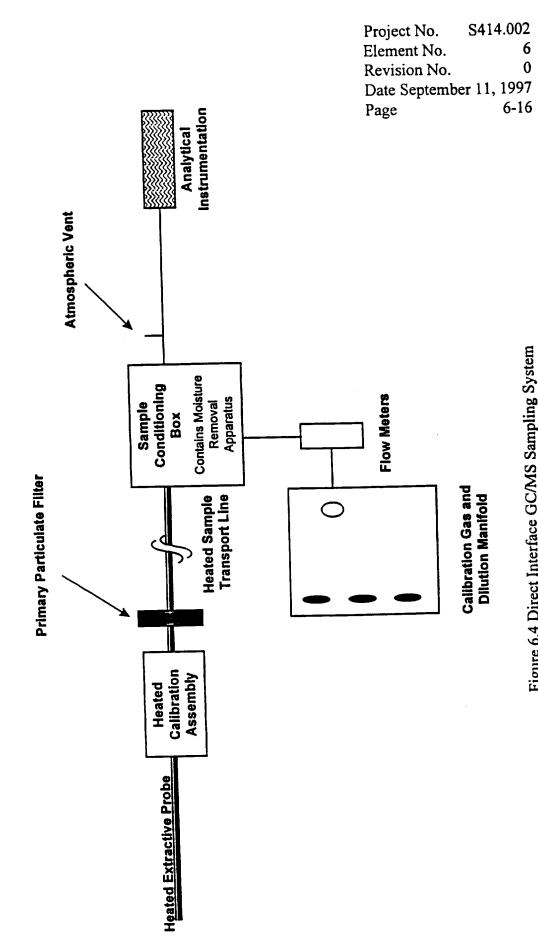


Figure 6.4 Direct Interface GC/MS Sampling System

### 7.0 SAMPLE HANDLING AND CUSTODY

Samples will be collected on site, and then transported by PES to PES' subcontract laboratory, TLI, for analysis. The custody procedures emphasize careful documentation of constant secure custody during the field, transport, and analytical stages of the test program. While in the field the samples will be either under the direct control of PES personnel or locked in the field laboratory. During transport, boxes and coolers containing samples and chain of custody will be sealed with custody tape or a custody tag. While at PES, samples will be kept locked in the laboratory section of the shop and refrigerated if necessary. After signed transfer of the sample to the sample custodian, the samples will be tracked using TLI's sample tracking form. After analysis, the samples are archived at TLI and all analytical results and associated sample custody and sample tracking are kept on file at TLI. PES sample custody form and labels are presented as Figures 5.2 and 7.1.

# 7.1 Sample Custodians

The following are the names of each sample custodian who will be responsible for the proper chain of custody during this testing program.

Daniel Sheffel

Bill Hurst / Chuck Livengood

PES, Inc.

Triangle Laboratories, Inc.

4700 Duke Drive, Suite 150

801 Capitola Drive

Mason, Ohio 45040

Durham, North Carolina 27713

513/398-2556 (ph) 513/398-3342 (fax)

919/544-5729 (ph) 919/544-5497 (fax)

## 7.2 Sample Numbering Scheme

Each test run will have a unique alpha-numeric code to be used only for sample components from that test run. This code will include information on the test location, test method, and test run. An "I" or "O" represents the inlet or outlet location, respectively. The methods are abbreviated as M29, M23, MM5. The sample components are described in a one or two word phrase such as: filter, impinger catch, acetone rinse, etc. In addition to this numbering scheme, the field laboratory will label each sample with a unique sample identifier number and

will generate a key for these sample identification numbers.

Example:

Test Location - Test Method - Run Number - Sample Component

I - M29 - 2 - Filter

# 7.3 Sample Preservation

All samples will be stored and transported in sealed containers away from dust and other ambient causes of contamination. Table 7.1 outlines the containers and preservation required.

Pacific Environmental Services, Inc
Sample ID No.:
Recovered by (Initials):
Plant:
Project Number:
Date/Time:
Sample Media/Description:
Comments:

Figure 7.1 Typical Sample Label

TABLE 7.1
SAMPLE PRESERVATION

Sample Type	Containers	Preservation
EPA Method 23	Filter - Petri dish wrapped in aluminum foil,  XAD® - Wrapped in aluminum foil  Impinger catch and reagents - 500 mL amber glass jars with Teflon lids.	Chilled on ice. Jars should be sealed against contamination.
EPA Method 29	Filter - Glass petri dishes HNO3 reagent - 500 mL glass with Teflon lids. KMnO4 reagent - 500 mL glass with Teflon lids.	Jars should be sealed against contamination.
SW-846 Method 0010	Filter - Petri dish wrapped in aluminum foil,  XAD® - Wrapped in aluminum foil  Impinger catch and reagents - 500 mL amber glass jars with Teflon lids.	Chilled on ice. Jars should be sealed against contamination.

Note: PES uses precleaned and certified sample bottles.

## 7.4 Field Test Data

All data sheets from the test locations and field laboratories will be collected at the end of each test day by the PM and kept in his custody throughout the test program. The data sheets will be given over to the report writer after a thorough debriefing. The original field data will remain in PES custody and eventually stored on file with the final report.

# 8.0 QUALITY CONTROL REQUIREMENTS

The goal of the QC program is to ensure, to the highest degree possible, the accuracy of the data collected and quantify the accuracy. This section describes specific procedures for equipment preparation, on-site sampling, and sample recovery that affect sample quality, and laboratory analysis.

## 8.1 Reagents and Glassware Preparation

## 8.1.1 Supplies and Consumables

Most reagents arrive sealed from manufacturer with stated purity levels on the label. Other reagents come from the analytical laboratory which has documented the production of the reagents from known components. In each case reagents (including: XAD resin, filters, and liquid reagents) have QC blanks associated with them as a check against contamination.

#### 8.1.2 <u>MM5</u>

All sample train glassware and sample recovery apparatus will be preconditioned following the procedures of Method 0010. All sampling train compounds and sample recovery apparatus will be soaked in hot soapy water (Alconox<sup>®</sup>), followed by three rinses each with tap water, distilled/deionized water, pesticide grade methanol, and methylene chloride. The components will then be dried using prepurified dry nitrogen followed by oven drying for two hours at 450°F. All glassware will then be capped with aluminum foil. Quartz-fiber filters, without organic binders, will be used.

The XAD®-2 sorbent resin traps will be prepared by TLI. Once cleaned, the traps are prespiked with surrogates, and capped with glass balls and sockets until use in the field. Impinger water will be organic-free reagent grade. Distilled-in-glass grade methylene chloride and methyl alcohol will be used as recovery solvents.

## 8.1.3 **EPA Method 23**

All sample train glassware, filters and sample recovery apparatus will be preconditioned following the procedures of Method 23. All sampling train compounds and sample recovery apparatus will be soaked in hot soapy water (Alconox®), followed by three rinses each with tap water, distilled/deionized water. All glassware will then be capped with aluminum foil. Precleaned quartz-fiber filters, without organic binders, will be used.

The XAD®-2 sorbent resin traps will be prepared by Triangle Laboratories, Inc. Once cleaned, the traps are pre-spiked with surrogates, and capped with glass balls and sockets until use in the field. Impinger water will be organic-free reagent grade. Distilled-in-glass grade acetone chloride and toluene will be used as recovery solvents.

## 8.1.4 EPA Method 29

All sample train glassware, filters and sample recovery apparatus will be preconditioned following the procedures of Method 29. First, all sampling train glassware are rinsed with hot tap water and then washed in hot soapy water. Next, all glassware is rinsed three times with tap water, followed by three additional rinses with water. Then all glassware is soaked in a 10 percent (V/V) nitric acid solution for a minimum of 4 hours, rinsed three times with water, then rinsed a final time with acetone, and allowed to air dry. All glassware openings are covered where contamination can occur until the sampling train is assembled for sampling.

Quartz-fiber filters, without organic binders, will be used. Nitric acid and hydrochloric acid (Baker Instra-analyzed or equivalent) will be used as clean up reagents.

## 8.2 On-Site Sampling

- **8.2.1** General PES will provide an onsite QA auditor to observe testing and audit personnel, equipment, procedures and record keeping. The auditor will record his observations on the form shown in Figure 12.1. PES will perform the following checks on the sampling equipment.
- **8.2.1.1** <u>Barometric Pressure</u>. Barometric pressure will be recorded at the beginning of each test day. If necessary, adjustments will be made for elevation depending on the actual location of the barometer.

- **8.2.1.2** Dry Gas Meter. Prior to sampling, the MM5, Method 23, and Method 29 meter systems will be leak-checked both before and after the vacuum pump. The meter orifice check of section 4.4.1 of Method 5 will then be performed on the MM5, Method 23, and Method 29 meter systems.
- 8.2.1.3 <u>Sampling Equipment Assembly and Recovery</u>. MM5, Method 23, and Method 29 sampling trains will be assembled and recovered in on-site laboratory trailers away from the sampling site. To prevent cross contamination, EPA Method 29 will be assembled and recovered in a separate laboratory trailer. The sampling trains glassware openings will be sealed with Teflon<sup>®</sup> tape for transport to and from the sampling site.

### 8.2.2 Measurement Sites

Prior to sampling, all stack dimensions will be checked against preliminary information to verify measurement site locations, location of test ports, and stack inside dimensions. Inside dimensions will be checked to determine uniformity of the stack cross-sectional area and the sample test ports will be checked to verify that they do not extend beyond the inside wall. The stack inside dimensions, wall thickness, and sample port depths will be measured to the nearest 1/16 inch.

### 8.2.3 <u>Velocity Measurements</u>

All velocity measurement apparatus will be assembled, leveled, zeroed, and leak-checked prior to use and at the end of each test run. The static pressure will be determined at a single point near the center of the stack cross-section.

Apparatus	QC Check	Criteria	Corrective Action
Manometer	Leveled and zeroed before and after each test.	Bubble leveler must indicate level conditions.  Manometer must read zero.	Relevel and rezero.  Note in test log.
Pitot	Check for chips in Pitot. Perform leak check on Pitot lines before and after each test run.	Pitot lines must indicate no leak over a 15 second period at a $\triangle$ P of 3 inches of water.	Repair or replace Pitots. A post test leak check failure indicates the run must be voided and repeated.

## 8.2.4 Flue Gas Composition

Integrated multi-point flue gas samples will be collected in Tedlar® gas bags and analyzed for CO<sub>2</sub> and O<sub>2</sub> content. Prior to sampling, the gas bags will be leak-checked. At the sampling site, the sampling apparatus will be assembled and leak-checked. The gas bag will be completely evacuated. The sampling train will be started and once the entire sample train is purged, the sample flow rate will be set and the gas bag will be attached. At the completion of the sampling period the gas bag will be removed and the bag sealed.

Apparatus	QC Check	Criteria	Corrective Action
Tedlar <sup>®</sup> gas bags	Evacuated and leak checked.	Must be able to hold 2-4 inches of water vaccum for 10 minutes.	Discard bag and replace.

### 8.2.5 Moisture

The MM5, Method 23, and Method 29 trains will be used to determine stack gas moisture. During sampling, the exit gas of the last impinger will be maintained below 68°F to ensure complete condensation of the stack gas water vapor. The total moisture will be determined on-site gravimetrically using an electronic platform balance with 0.1 gram sensitivity. The MM5 and Method 23 sampling trains include a XAD sorbent module which will be also

been weighed for moisture catch.

Apparatus	QC Check	Criteria	Corrective Action
Impinger Glassware	Monitor Temperature	Exit temperature to be kept below 68°F.	Add more ice.
Electronic Balance	Reference Weight	Reading within 0.1 grams of known standard	Recalibrate and reweigh

### 8.2.6 Method 23. Method 29 and Modified Method 5

The field sampling QA/QC for MM5, Method 23, and Method 29 will begin in the sample recovery area. The sample trains will be set up without attaching the sample probe and leak-checked to verify sample train integrity before transport to the sampling site. At the sampling site the sample probe will be attached to the rest of the train and leak-checked a second time. Any leaks found in excess of 0.02 cfm will be corrected prior to beginning the test runs. Leak checks will also be conducted before and after any sample train component changes, between sample ports, and upon completion of each test run. Probe and filter heaters will be maintained within allowable ranges throughout the test runs.

One MM5 field blank, two Method 23 field blanks, and one Method 29 field blank will be obtained and checked for field contamination. Complete sample trains will be assembled in the sample recovery area and transported to and from the sampling location in the same manner as the sample trains used for the test runs. The inlet and outlet sampling locations are adjacent to one another. Therefore, only one field blank for each method will be required. Each field blank train will be subjected to a minimum of one leak check in the laboratory and five at the sample location.

Apparatus	QC Check	Criteria	Corrective Action
Probe and Filter Heaters	Monitor Temperature	Temperature to be kept 248 ± 25°F.	Adjust Heater Setting
Sampling Train	Leak Check	Leak Rate of less than 0.02 cfm	Adjust Sample Volume or Retest

### 8.2.7 Direct Interface GC/MS

The MS will be tuned according to the manufacturer's written instructions for the set up, tune, operation, and calibration of the GC/MS and sample interface equipment. All hardware and software setting of temperatures, pressures, and other operational parameters used for sample acquisition shall be the same as those used when constructing the three point calibration. Manufacturer's certificates of calibration standards and internal standards, will be stored and included in the test report.

## 8.3 Sample Recovery

Sample recovery will be performed in on site laboratory trailers under the supervision of the PES On-Site Laboratory Supervisor.

#### 8.3.1 MM 5

Recovery of the MM5 sample train will be performed following the procedures of Method 0010. In addition to the field blank discussed in Section 5.3.6 of Method 0010, blanks will be taken of reagent grade water, methanol, methylene chloride, unused filters, and XAD<sup>®</sup>-2 resin cartridge. The sample recovery apparatus will be made of Teflon<sup>®</sup> or glass and precleaned as described in Section 5.2.1 of Method 0010. The sample train cleanup will be accomplished by rinsing each sample train component three times with a 50/50 mixture of methanol and methylene chloride. All sample containers will be tared and then reweighed following sample recovery, so that sample integrity can be ascertained.

## 8.3.2 Method 23

Recovery of the Method 23 sample train will be performed following the procedures of Method 23. In addition to the field blank, blanks will be taken of reagent grade water, toluene, acetone, unused filters, and XAD®-2 resin cartridge. The sample recovery apparatus will be made of precleaned Teflon® or glass. The sample train cleanup will be accomplished by rinsing each sample train component from the nozzle to the front half of the filter holder three times with

acetone, followed by three toluene rinses. The back half of the filter and condenser is then rinsed three times with acetone. The condenser is soaked three times with toluene, for five minutes each time. All sample containers will be tared and then reweighed following sample recovery, so that sample integrity can be ascertained.

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#### 8.3.3 Method 29

Recovery of the Method 29 sample train will be performed following the procedures of Method 29. In addition to the field blanks, blanks will be taken of the reagent solutions, acetone, nitric acid, hydrochloric acid, and unused filters. The sample recovery apparatus will be nonmetal. The sample train clean up will be accomplished by rinsing each sample train component from the nozzle to the front half of the filter holder three times with 100 mL of acetone, followed by 100 mL of nitric acid. The first three impingers will be rinsed with 100 mL of nitric acid. The fourth impinger will be rinsed separately with 100 mL of nitric acid. The fifth and sixth impinger will be rinsed with 100 mL of KMnO<sub>4</sub>, followed if necessary by 25 mL of hydrochloric acid. All sample containers will be tared and then reweighed following sample recovery, so that sample integrity can be ascertained.

# 8.4 On-Site Analyses of Flue Gas Composition

Integrated flue gas samples, collected in Tedlar® bags, will be analyzed within four hours after sample collection as per Method 3B.  $CO_2$  and  $O_2$  content will be determined using an Orsat® analyzer with 0.1% graduations. Prior to analysis, the Orsat® analyzer will be replenished with fresh reagents and leak-checked in accordance with the manufacturer's instructions. Known concentrations of  $CO_2$  and  $O_2$  will be analyzed to confirm proper instrument operation. The measured  $CO_2$  and  $O_2$  concentrations must be within 0.5% (absolute) of the known concentration of each gas.

Apparatus	QC Check	Criteria	Corrective Action
Orsat <sup>®</sup>	Leak Check	Liquid level in each pipette must not fall below bottom of capillary tubing over a 4-minute period and	Adjust Orsat® and Recheck
		Menisus in burette must mot change by more than 0.2 mL during 4-minute period	
Orsat <sup>®</sup>	Analysis of Audit samples	Within 0.5% of known	Adjust Orsat® and Retest

#### 8.5 Laboratory Analysis

Table 8.1 lists the QC checks to be performed.

### 8.5.1 Modified Method 5 (MM5)

The MM5 samples will be analyzed following the procedures of SW-846 Method 8270. Field blanks and laboratory blanks will be used to check for contamination. They will be processed in the same way the field samples are processed.

A five point initial calibration will be performed using internal standards and instrument response factors developed for the target analytes. Method 8270 procedures require the percent standard deviation of the initial calibrations (%RSD) to be within 50% for most analytes and 30% for some analytes. Continuing calibrations will be performed daily during analysis and compared against the initial calibrations. If the continuing calibration values deviate from the initial calibration by more than 30% to 50%, depending on the analyte, the initial calibration will be repeated.

The XAD®-2 resins will be pre-spiked with surrogate standards prior to the field sampling. The recovery of these surrogates will be used to give an indication of the overall sampling and analytical recovery efficiency. After the field sampling and prior to sample extraction, the field samples and laboratory blanks will be spiked with a second set of surrogate standards. The recovery of these surrogate standards will be used to give an indication of the extraction and analytical recovery efficiency. The criteria for surrogate recoveries are presented in Table 8.1. After extraction and just prior to analysis, internal standards will be added to the sample extracts and used as an indicator of instrument response.

### 8.5.2 Method 23

The Method 23 samples will be analyzed following the procedures of EPA Method 23. Field blanks and laboratory blanks will be used to check for contamination. They will be processed in the same way the field samples are processed.

TABLE 8.1
QUALITY CONTROL SAMPLES

Test Method and QC Sample	Criteria	QC Limits
EPA Method 29		
Blanks - FB, RB, LB (1 of each)	Concentrations of analytes	Below detection limits
Spikes - MS, MSD (1 of each)	Percent Recovery	80%-120%
Dups (1 per test program)	Relative Percent Difference	<20% RPD
EPA Method 23		

### 8.5.3 Method 29

The M29 samples will be analyzed following the procedures of EPA Method 29. Field blanks and laboratory blanks will be used to check for contamination. They will be processed in the same way the field samples are processed. Calibration procedures vary by analyzer. The most sensitive method for each metal will be used. An overview of two analyzers are given below.

- **8.5.3.1** ICAP Calibration. The instrument is profiled and calibrated according to the manufacturer's recommended procedures using those standards. The calibration is checked once per hour. If the instrument does not reproduce the standard concentrations within 10 percent, the complete calibration procedures are repeated.
- **8.5.3.2** <u>CVAAS Calibration</u>. The standards are prepared and used to calibrate the spectrometer. Calibration procedures are also outlined in the EPA methods referred to in Table 29-2 (See Appendix A). Each standard curve is run in duplicate and the mean values are used to calculate the calibration line. The instrument is recalibrated approximately once every 10 to 12 samples.

# 9.0 CALIBRATION AND FREQUENCY

PES's sampling equipment is calibrated according to the criteria specified in the reference method. The range of calibration is specified for all environmental measurements to encompass the range of probable experimental values.

# 9.1 Calibration and Preparation of Sampling Apparatus

The preparation and calibration of source sampling equipment is essential in maintaining data quality. Brief descriptions of the calibration procedures used by PES follow. Detailed procedures as presented in the EPA test methods are presented in Appendix A.

## 9.1.1 Barometers

PES uses aneroid barometers which are calibrated against a barometer pressure value reported by a nearby National Weather Service Station and is corrected for elevation.

## 9.1.2 Temperature Sensors

Bimetallic dial thermometers and Type K thermocouples are checked for calibration using the procedure described in Section 3.4.2 of the Quality Assurance Handbook, Volume III, 1994. Each temperature sensor is checked over the expected range of use against an ASTM 3C or 3F thermometer. Table 9.1 summarizes the types of calibration checks performed and the acceptable levels of variance. Electronic temperature readouts are calibrated using a thermocouple simulator having a range of 0-2400°F.

#### 9.1.3 Pitot Tubes

PES uses Type S pitot tubes which are constructed to EPA Method 2 specifications. Pitot tubes meeting these criteria are assigned a baseline coefficient of 0.84 and need not be calibrated.

TABLE 9.1

SUMMARY OF TEMPERATURE SENSORS CALIBRATION REQUIREMENTS

	Tolerances	±1.5% of reference temperature	1.5% of reference temperature	±1°C	±1°C	±3°C
	Heated Oil (150-200°C)	*	*			
ſEDIA	Boiling Water (100°C)	*	*			
CALIBRATION MEDIA	Hot Water (40-50°C)					*
CA	Ambient Air (20-25°C)			*	#	*
	Ice Bath (0°C)	*		*	*	
	Number of Calibration Points	3	2	2	2	2
	Temperature Sensor	Stack Gas	Filter	XAD Trap	Impinger Outlet	Dry Gas Meter

TABLE 9.3

PCDD/PCDF ANALYTICAL EQUIPMENT CALIBRATION REQUIREMENTS

Analytical Parameter	Quality Parameter	Method of Determination	Frequency	Criteria
PCDD/PCDF - HRGC/HRMS	Calibration - Quantitative	Initial analysis of standards at 5 levels bracketing sample concentrations	Prior to sample analysis	Variability of avg. RRF must be <25 -30% RSD for each unlabeled analyte and internal surrogate & alternate std. S/N ratio must be >2.5; Ion abundance ratios must be within control limits
	Calibration - Column Performance Check	Analysis of column performance check solution mixture of PCDD/PCDF congeners	At start of each 12-hr shift	Document resolution between 2378-TCDD and other TCDDs (25% valley)
	Calibration - Continuing	Analysis of mid-range calibration standard	Every 12 hours	Variability of avg. RRF must be <25 -30% RSD for each unlabeled analyte and internal surrogate & alternate std. S/N ratio must be >2.5; Ion abundance ratios must be within control limits
	Calibration - Confirmation Analysis - Quantitative	Initial analysis of standards at 5 levels bracketing sample concentrations	Prior to sample analysis	Variability of avg. RRF must be <25 -30% RSD for each unlabeled analyte and internal surrogate & alternate std. S/N ratio must be >2.5; Ion abundance ratios must be within control limits

RRF = Relative Response Factor S/N = Signal to Noise Ratio

## 10.0 DATA ACQUISITION

### 10.1 Use of Non-Measurement Data

All data used in this project will be measured directly or calculated from direct measurement data. Indirect or non-measurement techniques such as: previous test data, fan amperage charts, engineering estimates, blue prints, and psychometric charts will not be used as final data. Such techniques may be used in the planning stages of the test program to properly size equipment and set analytical instrumental parameters.

### 10.2 Use of Calculated Data

Most of the data of interest will be calculated from direct measurements. For example, some of the direct measurements used in calculating emissions data include: pollutant catch weight, velocity pressure head, dry gas meter volume, barometric pressure, volume of condensate, average delta H during test run, size of nozzle used, and many others. The direct measurements are initially recorded on field data sheets. This information is input into spreadsheets at PES's office in Cincinnati. When analysis is complete, that data is also input. The calculated values are presented in the text of the final report. See Section 11 for details on data management and Section 14 for data verification and validation.

## 11.0 DATA MANAGEMENT

### 11.1 Life Cycle of Data

Project data is first generated in the field by direct observations. These observations are recorded in ink on preformatted data sheets. These data sheets and samples are returned from the field to PES. The samples are sent to Triangle Laboratories, Inc where they are analyzed for the prescribed pollutants. The field data are sent to data reduction where it is input into software designed to generate information on gas stream conditions based on data provided. When analysis is complete, the analytical data are combined with the field data to produce pollutant emission rates and concentrations. Calculated results, copies of field data sheets, and raw analytical data are all included in the final report. The original data sheets, and analytical data, and disk copies of the software used in the calculations are stored on file at PES for a period of no less than three years.

## 11.2 Data Recording

Extreme care will be exercised to ensure hand recorded data are written accurately and legibly. Additionally, the forms have been formatted for easy use. PES will provide an onsite QA observer to audit the data recording process. See Section 13 for details on the role of the onsite QA personnel. Errors and discrepancies will be noted in the PM field log book.

# 11.3 Data Collection and Handling Validation

In addition to the project undergoing validation after data collection and analysis (See Section 15), the data is validated as it is collected. The PM will review field data sheets looking for inconsistencies in such parameters as: temperatures, pressures, velocity head, sample volume, etc. The PM will also review sample components, and sample recovery sheets looking for inconsistencies in such parameters as: sample volume, amount of catch on filter, color of samples, labeling, mathematics on data sheets, etc. Errors and discrepancies will be noted in the PM field log book.

### 12.0 PROJECT ASSESSMENT

### 12.1 Onsite Procedure Audit

QA personnel will be onsite to observe testing and audit personnel, equipment, procedures and record keeping for compliance with the QAPP. This audit will be conducted after work has commenced, and at regular intervals, thus giving an opportunity for corrective action. An example of an onsite audit sheet appears as Figure 12.1. These forms will be included in the final report.

### 12.2 Internal Data Review

PES personnel with technical expertise in the field of air emissions testing, who have had minimal or no involvement with this test program will provide a thorough review of the data. Reviewers will determine if the project activities were: technically adequate, competently performed, and properly documented. Reviewers will also consider if the project data meets the technical and quality assurance requirements of the QAPP. Reviewers' assessment of the assumptions, calculations, extrapolations, interpretations, methods, acceptance criteria and conclusions will be documented in the final report. The names, titles and positions of the reviewers will also be included in the final report.

Quality Control Check	Observation
After Testing	
Visually inspect sampling nozzle	
Visually inspect Type S Pitot tube	
Leak check each leg of the Type S Pitot tube	
Leak check the entire sampling train	
Record observations if any	,
Field Log	
Project name/ID and location	
Sampling personnel (names/position)	
Geological observations including map	
Sample run times and dates	
Sample descriptions	
Description of QC samples	
Deviations from QAPP	
Difficulties in sampling or unusual conditions	
Sample Labels	
Sample ID	
Date and time of collection	
Lab technician initials	
Analytical parameter	
Preservative required	

Figure 12.1. Onsite QA/QC Observations (concluded)

## 13. REPORTS TO MANAGEMENT

Effective communication is an integral part of a quality system. Planned reports provide a structure for apprising management of the project schedule, the deviations from the approved QAPP, the impact of the deviations, and the potential uncertainties in decisions based on the data.

## 13.1 On Site Reporting

During testing, personnel will be in constant communication with the PM. The test crew will apprise the PM of sample start and stop times, difficulties in sampling, and any abnormal conditions. Onsite laboratory technicians will apprise the PM of observations on sample appearance and sample volume. The PM will document these observations in the bound project logbook.

### 13.2 Data Processing

Data processing and report writing personnel will continually provide brief informal progress reports to the PM. When necessary, corrective action reports will be submitted describing the nature of the occurance and suggested solutions.

### 13.3 Reports to EPA

The PM will provide monthly progress reports to the EPA WAM. These reports will include pertinent information from the data processing and report writing progress reports and corrective action reports, as well as reports on the status of analytical data as determined from conversations with TLI. PES will promptly advise the EPA WAM on any items that may need corrective action.

#### 13.4 Internal Review Assessment

Internal reviewers will develop a report based on their assessment of the project data. This report will be given to the PM. All pertinent findings will be reported to the EPA WAM.

#### 14. DATA VALIDATION AND USABILITY

The purpose of validation is to assess the degree to which the data meets the quality specifications outlined in the QAPP. If deviations are noted, the validation procedures can be used to assess the effect the deviation will have on data usability. Forms used in data validation appear as Figures 14.1 through 14.5.

### 14.1 Sampling Design

The first step in validating the data set is to assess if the project, as executed, meets the sampling design. Process data are reviewed to ensure the unit was operating within the limits outlined in the project. Test dates and times are reviewed to ensure that testing was performed as scheduled with test methods performed sequentially or simultaneously as designed. Test locations are checked against the project design. The method performed and the pollutants tested for are checked against the project design.

### 14.2 Sample Collection Procedures

Actual procedures documented in field logs and data sheets are checked against the procedures described in the QAPP. Deviations from the QAPP will be classified as acceptable or unacceptable, and critical and non-critical.

### 14.3 Sample Handling

Sample custody and holding times will be checked for each sample component. Sample custody from the field to the lab and within the departments of the lab will be checked. Holding times and sample preservation will be checked against the QAPP. All deviations will be documented in the final report.

### 14.4 Analytical Procedures

The analytical procedures performed during the test program will be checked against those described in the QAPP. Deviations from the QAPP will be classified as acceptable or unacceptable, and critical and non-critical.

Location / Condition				
Method				
Run IDs				
Date				
Time				
				***************************************
ts				
%CO2				
%02				
Po				

Figure 14.1. Flue Gas Validation Worksheet

Run ID	Nozzle Number	Filter Number	Post Test Leak Check	Post Test Pitot Leak Check	Start &Stop Time	Pitot Number
		-				

Figure 14.2. Data Sheet Completeness Worksheet

RUN ID	PITOT ID	CALIBRATION COEFF.	DATA SHEET COEFF.	CALIBRATION DATE	TEST DATE
		7			

Figure 14.4. Pitot Calibration Worksheet

RUN ID						
METHOD						
QC CHECK	QCLIMIT	ACTUAL	QC LIMIT	ACTUAL	QC LIMIT	ACTUAL
FB						
TB						
RB1						
RB2						
RB3						
LB						
MS						
MSD						
SS						
DUP						
MISC1						
MISC2						
MISC3						

FB= Field Blank, TB = Trip Blank, RB= Reagent Blank, LB= Lab Blank, MS= Matrix Spike, MSD= Matrix Spike Duplicate, SS = Surrogate Spikes, DUP = Duplicate Analysis, MISC = Other miscellaneous QC checks.

NOTES: If a QC check does not apply to a method place a "-" in the box. There may be several surrogate standards. Use the form as a prompt to check over all surrogate spike recoveries.

Figure 14.6. QC Check Worksheet

## 15. RECONCILIATION WITH DATA QUALITY OBJECTIVES

Results obtained from the project will be compared to the data quality objectives described in this document. Results will be checked for accuracy, precision, and completeness. The following subsections show the calculations involved.

As stated in Section 2.0 the end use of this data is to quantify emissions from the cupola process at the host facility and to determine fabric filter performance in controlling emissions of HAPs from cupolas. As the project did not set a standard for emissions or performance levels to meet, the data will be compared only to the quality assurance objectives in this document. Using the results obtained from the project and the comparison with quality assurance objectives, PES will investigate to identify any patterns, relationships, or potential anomalies. PES will evaluate whether the project met the objectives of the sampling design, and whether departures from the QA/QC guidelines are acceptable. The conclusions will be presented to the EPA in the final report.

#### 15.1 Accuracy

Accuracy refers to the closeness of the result to the actual values. In this project, accuracy will be measured using matrix spikes and surrogate spikes. A known amount of a matrix or surrogate spike is added to a sample. The sample is then analyzed for the compound. The result is referred to as a matrix or surrogate spike recovery.

#### 15.2 Precision

Precision refers to the repeatability of the result. In this project, precision will be measured using duplicate analysis. The precision is expressed as relative percent difference.