United States
Environmental Protection
Agency

Office Of Air Quality
Planning And Standards
Research Triangle Park, NC 27711

EPA-454/R-00-038b
September 2000

Air

EPA

Source Characterization For Sewage Sludge Incinerators

Final Emissions Report
Volume I of III

Metropolitan Sewer District (MSD)
Mill Creek Wastewater Treatment Plant
Cincinnati, Ohio

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EPA-454/R-00-038b

SOURCE CHARACTERIZATION FOR SEWAGE SLUDGE INCINERATORS

FINAL EMISSIONS REPORT, VOLUME I OF III

METROPOLITAN SEWER DISTRICT (MSD)
MILL CREEK WASTERWATER TREATMENT PLANT
CINCINNATI, OHIO

Prepared for:

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Research Triangle Park, NC 27711

OVLC# 477705

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Office of Air and Radiation
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

September 2000

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The information in this document has been funded wholly or in part by the Office of Air Quality Planning and Standards, U.S. Environmental Protection Agency (EPA) under contract 68-D-99-009 to Battelle. It has been subjected to the Agency's review, and has been approved for publication as an EPA document. Mention of trade names or commercial products is not intended to constitute endorsement or recommendation for use.

ACKNOWLEDGMENTS

This report was prepared under Contract No. 68-D-99-009, Work Assignment WA 2-01, by Battelle and its subcontractor ETS, Inc. under the sponsorship of the U.S. Environmental Protection Agency. Mr. Eugene Crumpler was the EPA Program Manager and Mr. C. E. (Gene) Riley was the Work Assignment Manager. Their support on this test program was much appreciated. We would also like to acknowledge the assistance provided by the Hamilton County Metropolitan Sewer District and its employees, in particular Mr. Michael W. Heitz, who served as the MSD on-site coordinator for this test program.

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"SOURCE CHARACTERIZATION FOR SEWAGE SLUDGE INCINERATORS - FINAL EMISSIONS REPORT - VOLUME I OF III - METROPOLITAN SEWER DISTRICT (MSD) - MILL CREEK WASTEWATER TREATMENT PLANT, CINCINNATI, OHIO"
EPA -454/R-00-038b - September 2000

Please delete and replace the following text contained in the report.

Page 1-1 1.0 INTRODUCTION
1.1 SUMMARY OF TEST PROGRAM

Delete the first and second paragraphs starting with: "The Clean Air Act Amendments"

Replace the second paragraph as follows:

This test report summarizes testing of a multiple hearth incinerator at the Metropolitan Sewer District (MSD) Mill Creek Wastewater Treatment Plant in Cincinnati, Ohio in July, 1999. The emissions data collected in this program will be used to provide information for EPA's Office of Water (OW) to determine the need for further emissions standards in the Section CFR 503 - Subpart E, Standards for Incineration of Sewage Sludge.

1.0 INTRODUCTION

1.1 SUMMARY OF TEST PROGRAM

The Clean Air Act Amendments of 1990 require the U.S. Environmental Protection Agency's (EPA) Office of Air Quality Planning and Standards (OAQPS) to establish standards of performance for sewage sludge incineration. These standards are necessary to protect public health and the environment from any adverse effects of pollutant emissions from sewage sludge incineration. The regulations will contain general regulatory requirements, pollutant characterization, and emission limits. To assess control technologies as well as associated strategies for cost-effective standards, EPA requires data on emissions from sewage sludge incinerators . While some emission data exist for sewage sludge incinerators, data on coplanar polychlorinated biphenyls (PCBs) from sewage sludge incinerators are very limited.

This test report summarizes testing of a multiple hearth incinerator at the Metropolitan Sewer District (MSD) Mill Creek Wastewater Treatment Plant in Cincinnati, Ohio in July, 1999. The emission data collected in this test program will be used by EPA/OAQPS and EPA's Office of Water (OW) plans to use these data to support a decision about further data gathering efforts in support of MACT standards for sewage sludge incinerators.

The Mill Creek Wastewater Treatment Plant is a municipal wastewater treatment plant designed to process 120 to180 million gallons per day (MGD) of wastewater. Most of the wastewater received at the treatment plant comes from sanitary sources, with approximately 20 to 25 percent from industrial sources. The sludge generated from wastewater treatment is incinerated on site in six multiple hearth incinerators. Normally, three incinerators incinerate a combined total of 110 dry tons of sludge per day. The incinerators operate 24 hours a day and burn natural gas auxiliary fuel. The incinerators can burn digester gas but rarely do so. Emissions from each incinerator are controlled by a venturi scrubber followed by a three-tray impingement conditioning tower with a chevron style demister. The controlled emissions from each incinerator then exit through its own individual stack.

Table 1-1. Test Matrix

Sampling Location/ Matrix	No. of Runs	Sample Type	Sampling Method	Sampling Org.	Sample Run Time (min)	Analytical Method	Analytical Laboratory
Outlet Stack	3[b]	Coplanar PCBs	M-0010[a,b]	ETS	360	Draft PCB-Emissions[c]	Battelle
	3[b]	D/Fs	—0010[b]	ETS	360	M-8290[d]	Battelle
	3[b]	PAHs	M-0010[b]	ETS	360	CARB 429[e]	Quanterra
	Continuous	CO	M 10[f]	ETS	360	NDIR	NA
	Continuous	O_2/CO_2	M 3A[g]	ETS	360	Chemical Cell/ NDIR	NA
	Continuous	THC	M25A	MSD	360	Flame Ionization	NA
Sludge Feed	6 Grab Samples (1 per hour)	Coplanar PCBs	Composite of 60 min grabs	Battelle	360	Draft PCB Sludge[h]	Battelle
		D/F	Composite of 60 min grabs	Battelle	360	M-8290	Battelle
		Chlorine	Composite of 60 min grabs	Battelle	360	M-4500 G[i]	U.S. EPA T & E
		Total % Solids	Composite of 60 min grabs	Battelle	360	M-2540 B[j]	U.S. EPA T & E
		Ultimate/Proximate	Composite of 60 min grabs	Battelle	360	ASTM D3172, D5373	CT&E
Scrubber Water		Coplanar PCBs	Composite of 60 min grabs	Battelle	360	Draft PCB Water[k]	Battelle
	Inlet and Outlet 6 Grab Samples Each (1 per hour)	D/F	Composite of 60 min grabs	Battelle	360	M-8290	Battelle
		Chlorine	Composite of 60 min grabs	Battelle	360	M-4500 G	U.S. EPA T & E
		Total % Solids	Composite of 60 min grabs	Battelle	360	M-2540 B	U.S. EPA T & E
		pH/Temp	60 min grabs	Battelle	360	M-4500-H[l]	Battelle

a SW-846, Method 0010, Modified Method 5 Sampling Train.
b Three M-0010 runs total at outlet stack, single M-0010 run will generate sample for coplanar PCB, D/F, and PAH analysis.
c Draft Analytical Method for Determination of Toxic Polychlorinated Biphenyl Emissions from Sewage Incinerator Stationary Sources Using Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry.
d SW-846, Method 8290, Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography / High Resolution Mass Spectrometry (HRGC / HRMS).
e Air Resources Board, Method 429, Determination of Polycyclic Aromatic Hydrocarbons (PAH) Emissions from Stationary Sources.
f 40CFR60, Appendix A, Method 10, Determination of Carbon Monoxide Emissions from Stationary Sources.
g 40CFR60, Appendix A, Method 3A, Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources.
h Draft Method, Determination of Toxic Polychlorinated Biphenyls in Sewage Sludge Using Isotope Dilution High Resolution Gas Chromatography / High Resolution Mass Spectrometry.
i Standard Methods for Examination of Water and Wastewater, Method 4500 G, DPD Colorimetric Method.
j Standard Methods for Examination of Water and Wastewater, Method 2540 B, Total Solids Dried at 103-105°C.
k Draft Method, Determination of Toxic Polychlorinated Biphenyls in Sewage Incinerator Scrubber Water Using Isotope Dilution High Resolution Gas Chromatography / High Resolution Mass Spectrometry.
l Standard Methods for Examination of Water and Wastewater, Method 4500 H, pH Value.

The test program was conducted according to the "Site-Specific Test Plan (SSTP) - Sewage Sludge Incinerator, Metropolitan Sewer District, Cincinnati, Ohio", dated July 16, 1999. A matrix of the type and location of the samples collected is presented in Table 1-1. This test program included collection and analysis of stack gas from Incinerator No. 6 for polychlorinated biphenyl (PCB), polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (D/F), and polycyclic aromatic hydrocarbons (PAH), carbon monoxide (CO), carbon dioxide (CO_2), oxygen (O_2), and total hydrocarbon (THC).

Process samples consisting of sludge feed and scrubber water into and out of the venturi control system were also collected. Battelle analyzed both sludge feed and scrubber water samples for PCB, D/F, chlorine (Cl_2), and percent solids. The field team measured the temperature and pH of the scrubber water at the time of sample collection. Battelle arranged for ultimate/proximate analysis of the sludge feed.

Emission Measurement Center (EMC) of OAQPS coordinated emission measurement activities. Battelle and its subcontractor ETS, Inc. (ETS) performed the field sampling. Battelle laboratories in Columbus, Ohio, conducted the sample analysis except those noted below. EPA's Test and Evaluation Facility (T&E) on the MSD property did the chlorine and percent solids analyses of sewage sludge and water samples. Quanterra Environmental Services (California Lab) analyzed the PAH samples. Commercial Testing & Engineering Co. (CT&E) conducted the ultimate/proximate analysis of sewage sludge samples. Pacific Environmental Services (PES) collected process control data for the EPA under a separate contract.

1.2 TEST PROGRAM ORGANIZATION

Figure 1-1 presents the test program organization, major lines of communication, and names of responsible individuals and their functional role.

Mr. Gene Crumpler was EPA's Program Manager (PM) for this test. Mr. Gene Riley was EPA's Work Assignment Manager (WAM). Mr. Anthony Wisbith was Battelle's Work Assignment Leader (WAL). He was assisted by Mr. Andrew Hetz of ETS, Inc. as the Field Test Team Leader (FTTL). Ms. Karen Lesniak served as Battelle's Laboratory Coordinator, and Ms. Susan Abbgy as Battelle's QA Officer. Ms. Abbgy was assisted by Ms. Pamela Broadwell of ETS, Inc. in completing QA/QC activities. Mr. Michael Heitz of MSD served as the On-Site

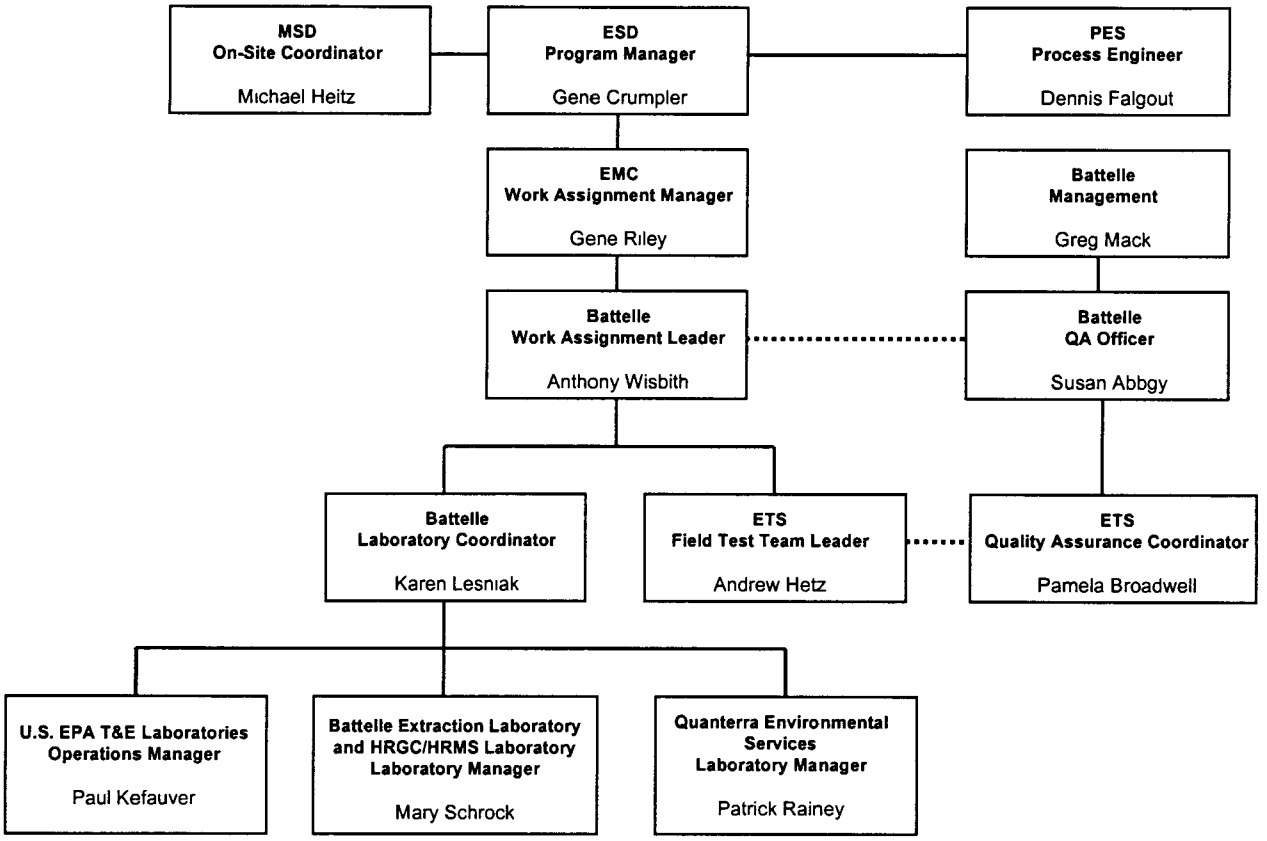

Figure 1-1. Program Organization

Coordinator. Mr. Dennis Falgout of PES collected process operating data during the test program.

Addresses and phone numbers for responsible individuals are provided below.

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1.3 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROCEDURES

QA/QC procedures implemented for this test program are described in the "Quality Assurance Project Plan (QAPP) - Sewage Sludge Incinerator, Metropolitan Sewer District, Cincinnati, Ohio", dated July 16, 1999. This QAPP addresses project management, measurement and data acquisition, assessment and oversight, and data validation. Section 6 of this report summarizes QA/QC results for the test program. Revisions to the QAPP were documented through the use of a Quality Assurance Project Plan Amendment Record Sheet, as described in Section A9.0 of the QAPP. Copies of the QAPP Amendment Record Sheets generated during this test program can be found in Appendix P to this report. A Post-Test Summary Report discussing the amendments and any other deviations from the SSTP which occurred in field sampling can also be found in this appendix.

1.4 DESCRIPTION OF REPORT SECTIONS

This test report consists of the following sections:

Section 1.0 provides an introduction to the test program. This section includes the general purpose and background of the test program, a brief overview of the facility and process tested, and the pollutants measured. A listing of the participants, an overview of QA/QC aspects of the test program, and this report content description are also contained in Section 1.0.

Section 2.0 provides a summary of test results. This section includes the test matrix; CEM results; analytical results for PCBs, D/Fs, and PAHs for emission, scrubber water, and sludge feed samples; and inorganic analysis results for scrubber water and sludge feed samples. A summary of field sampling procedural deviations from the SSTP and QAPP is also presented in this section.

Section 3.0 contains a description of the sampling locations for stack emissions, scrubber water, and sludge feed, a process diagram denoting the sampling locations, and a traverse point cross section of the stack.

Section 4.0 describes the process and facility operation, including control equipment specifications. Process operating data were furnished by PES directly to EPA.

Section 5.0 presents a summary of the field sampling and laboratory analysis procedures employed. These procedures include draft methods for PCB analysis of emissions, water, and sludge; standard methods for D/F and PAH analyses; and standard EPA methods for continuous emission monitoring (CEM) of combustion gases and stack parameters.

Section 6.0 summarizes results of internal QA/QC activities performed in the test program. This section lists the program and method specific performance targets and actual results achieved.

Appendices containing the raw field and laboratory data, including QA/QC results, are compiled in separate volumes to this report.

2.0 SUMMARY AND DISCUSSION OF TEST RESULTS

This section provides results of the test program conducted at the Cincinnati MSD on a sewage sludge incinerator and related processes from July 19 to July 22, 1999. Test results are provided in separate sections for air organic emissions, continuous emissions monitoring, and process sample measurements. Included in this section are a description of the test objectives and a discussion of changes and problems encountered during the field sampling.

2.1 OBJECTIVES AND TEST MATRIX

The overall objective of this test program was to quantify the toxic PCB, D/F, and PAH emissions from the stack of a sewage sludge incinerator. In addition, D/F and PCB measurements were performed on the sewage sludge feed and scrubber water. Complete test program parameters can be found in Table 1-1, Test Matrix. A field test log showing the dates of the sampling activities is provided in Appendix D-5.

The PCB and D/F emissions data collected from the MSD sewage sludge incinerator in this test program will be used by EPA's OAQPS and OW to:

(1) Conduct a comprehensive assessment of the risk to human health of the emissions of dioxin/furan/toxic PCBs from sewage sludge incinerators. This assessment is to determine if regulations on these emissions are required to reduce any unacceptable risk.

(2) Establish an emissions data base for toxic PCB and D/F emissions from sewage sludge incinerators.

2.2 SITE-SPECIFIC TEST PLAN CHANGES AND SAMPLE COLLECTION PROBLEMS

Field sampling modifications made to the Site-Specific Test Plan (SSTP) are summarized in the Post-Test Summary Report and in one SSTP Amendment. Copies of the Post-Test Summary Report and the SSTP Amendment are provided in Appendix P.

Specific sample collection procedures are described in Section 5.0 of this report and in the SSTP. Outstanding problems encountered during the test program are noted below for each medium sampled.

2.2.1 Air Emissions

A calibration gas audit (pretest and post test) was performed on the total hydrocarbon (THC) analyzer operated by the MSD during the test program. The THC calibration span gas (124.6 ppm) used for the audit was outside of the stated SSTP range (150 to 180 ppm). Substitution of the calibration span gas (124.6 ppm) was approved by the WAM since the total hydrocarbon analyzer was expected to operate on the low end of its calibration curve.

During the post-test calibration on July 22, 1999, moisture condensation was observed in the flow panel serving the analyzer. The post-test CGA revealed a slower response time and degraded accuracy. However, the accuracy was still within the allowed 15 percent. Condensation within the system could slow the analyzer response time and absorb some of the water-soluble organics in the span gas audit as well as the emissions being sampled.

The carbon monoxide (CO) analyzer was calibrated to a span of 6,000 ppm although provision was made to determine linearity up to 10,000 ppm if CO spikes above 6,000 ppm were encountered. No such exceedances of the specified calibration span were experienced during the test program.

The initial Proof Blank #1 was collected with an undersized nozzle since initial information on flowrate proved inaccurate. The nozzles were concurrently cleaned and similarly sized during the Modified Method 5 (MM5) runs such that the WAM accepted the Proof Blank #1 as being valid.

Concern was raised by the WAM on July 20, 1999, that gas stratification may be encountered in the exhaust stack. A stratification check was performed initially but the results were inconclusive. The WAM made a decision to traverse the same MM5 traverse point locations, using the CEM sampling probe simultaneous with each MM5 sampling run.

The initial MM5 sampling run on July 20, 1999, was invalidated for failing to meet post-test leak checking criteria after the midport change. As a result of this deviation, four MM5

sampling runs were performed such that three valid sampling runs (Runs 2, 3, and 4) and associated samples were made available for laboratory analyses.

Testing was interrupted at 105 minutes into MM5 Run 2 to troubleshoot high vacuum as indicated by the train vacuum gauge. The MM5 sampling train was leak-checked and the high vacuum was found to have been caused by compaction of the silica gel in the fifth impinger. The silica gel was loosened by shaking the fifth impinger and the train was again leak-checked. Testing resumed after a 15-minute delay, resulting in the interruption of data from 16:46-17:00 on July 20, 1999.

Throughout the MM5 sampling runs, isokinetic sampling was maintained using a "K Factor" or the ratio of the orifice meter differential pressure necessary to maintain isokinetic sampling to the differential pitot pressure. Known variables which are used to calculate the K factor include the orifice differential pressure during calibration (ΔH_a), the barometric pressure, and the nozzle diameter. Assumed variables include sampling meter temperature, and flue gas temperature, static pressure, and moisture.

The K factor was initially calculated for MM5 Run 1 based on preliminary sampling performed on July 19, 1999, and information obtained from Cincinnati MSD. Adjustments were made during each sampling run as actual data were obtained and compared to assumed values.

Although acceptable, the isokinetic sampling rate during MM5 Run 2 was lower than expected (91.7 percent), because of an incorrect assumption of flue gas moisture. The K factor was initially calculated for MM5 Run 2, based on an estimated moisture of 10 percent; a value measured during previous emissions tests. This estimated value was significantly higher than the actual flue gas moisture content of 4-5 percent measured during the actual sampling program. Although preliminary moisture measurements taken on July 19, 1999, supported the lower moisture values, these readings were thought to be anomalous at the time.

The data from the voided MM5 Run 1 also indicated 4.7 percent moisture. However, this value was also assumed invalid, because of suspected inleakage dilution from a failed leak check at the midpoint of the sampling run. It is now suspected that the leak originated during removal of the train from the sampling port and had no bearing on sample or moisture collection.

Following recovery of MM5 Run 2, the flue gas moisture of 4-5 percent was confirmed and the K factor was recalculated for MM5 Run 3 based on this measurement.

2.2.2 Scrubber Water

Upon review of the venturi scrubber operation during on-site activities on July 19, 1999, Mr. Eugene Crumpler, the EPA Program Manager, requested that the scrubber outlet water be characterized for comparison with the inlet water which was to be sampled according to the SSTP. Scrubber water samples were collected every hour as grab samples and were analyzed in the field for pH and temperature. Six grab samples for each location were composited into two scrubber water samples for each MM5 emission sampling run. The composited samples were submitted for analysis of toxic PCBs, D/Fs, chlorine, and total percent solids. The SSTP had specified a water sample collection frequency of one grab every half-hour during each MM5 sampling run. A modification was approved by the WAM to increase the sampling interval because a single sampling technician had been designated to collect the samples and time constraints were of a concern, as well as the addition of the outlet water sample.

2.2.3 Sewage Sludge

On July 20, 1999, the EPA Program Manager decided that additional analysis of the sewage sludge feed would be useful to evaluate the thermal characteristics of the sewage sludge during incinerator operation. Separate aliquots were generated from the composite sewage sludge feed samples for each of the three runs and submitted for ultimate/proximate analysis. The sewage sludge feed samples collected for proximate/ultimate analysis were handled and processed identically to the other sewage sludge feed samples collected.

2.3 AIR ORGANIC EMISSIONS SUMMARY AND DISCUSSION

The test results for air emissions are provided for toxic PCBs in Tables 2-1 through 2-4, for D/Fs in Tables 2-5 through 2-7, and for PAHs in Tables 2-8 and 2-9. Detailed analytical and field sampling results, including front and back half fractions for each MM5 sampling run are presented in the appendices. The PCB, D/F, and PAH analytical results can be found in Appendices E, F, and G, respectively.

The toxic PCB, D/F, and PAH results for Runs 2 and 3 are almost identical for most of the analytes. The back half emission concentrations for Run 4 are 50 to 60 percent lower than the back half emission concentrations for Runs 2 and 3 for all the analytes. As a result, emission concentrations for Run 4 are approximately half of the emission concentrations for Runs 2 and 3 for all three analyte classes.

Analyte loss may have occurred during sampling, during sample handling and transport, or prior to spiking the Run 4 sample with pre-extraction internal standards. This time period is based on a comparison of the pre-field surrogate spike recoveries to the pre-extraction internal standard recoveries. Recoveries of the pre-field surrogate spikes for Run 4 back half samples were approximately half of the recoveries for Run 2 and 3 back half samples for all PCB, D/F, and PAH field surrogate spikes, whereas recoveries of the spiked pre-extraction PCB, D/F, and PAH internal standards were comparable and generally acceptable across all three runs. Any losses in the pre-field surrogate spikes that may have occurred in sample extraction or cleanup of the Run 4 back-half sample would have also been reflected in similar losses of the spiked pre-extraction internal standards. Since the pre-extraction internal standard results are acceptable for Run 4 and consistent with the other two runs, this result suggests that the field surrogate spike and analyte losses likely occurred prior to extraction of the Run 4 emission samples.

Another indicator that analytes losses occurred prior to sample extraction is that the PCB, D/F, and PAH concentrations in the samples follow the same pattern as the pre-field surrogate spikes in that all measured back half analytes were approximately one half or less for the Run 4 sample. In addition, analyte concentrations for the Run 4 front half sample were somewhat lower than the Run 2 and 3 front half samples. This suggests that analyte levels may not have been consistent during sampling rather than a loss of analyte from the collected sample.

After review of sampling and analysis records, a definite explanation for the lower Run 4 emission concentrations could not be determined. Possible causes of the lower Run 4 concentrations and/or lower pre-field surrogate spikes that were considered include the following:

- Pre-field Surrogate Spike Performed Improperly. This does not seem possible in that the PCB, D/F, and PAH spikes were done independently (three separate solutions) and the same spiking error would have had to be made three times on the same XAD-2

resin. The laboratory logbooks do not reflect any problem with the pre-field surrogate spiking of the XAD-2 resin.

- Analytes Not Collected Consistently During Sampling. In Run 4, PCB, D/F, and PAH compounds in the gas stream may not have been collected consistently by the MM5 sampling train. This event would affect both the Run 4 front half and back half samples and is substantiated by emission concentrations for the Run 4 front half samples which are somewhat lower than emission concentrations for the Run 2 and 3 front half samples.

- XAD-2 Resin Lost After Sampling. During sample recovery, XAD-2 resin could have been lost from the XAD-2 resin cartridge. Field logs do not indicate any problem with the XAD-2 resin cartridge during Run 4 sample recovery so this is probably not the case.

- Temperature of XAD-2 Not Maintained During Sampling. The XAD-2 resin cartridge must be maintained at 20°C or lower temperature to avoid decomposition or volatilization of organic compounds. If the MM5 sampling train or XAD-2 resin portion thereof was exposed to ultraviolet light, high temperature, or other forms of energy, this might account for the low levels. A check of the field data sheet showed that the XAD-2 trap temperature was maintained below the required 20°C throughout Run 4. A review of the field log does not indicate any problems in recovering the MM5 sampling train at the completion of Run 4.

- XAD-2 Temperature Not Maintained During Sample Transportation. A review of the laboratory sample check-in record book shows that the Run 4 samples were received within the allowable ≤4°C. The temperatures of the coolers storing the XAD-2 resin traps as received from the test team for transport to Columbus were also within this limit.

- XAD-2 Temperature Not Maintained During Storage Prior to Extraction. All air emission samples were stored in the same locked refrigerated storage unit in the laboratory prior to extraction. The temperature control records for this period do not indicate any elevated temperatures.

- Emissions Concentrations During Run 4 Were Actually Lower. Lower Run 4 concentrations may be an accurate reflection of a change in incinerator emissions on the third day of sampling. The DQA report discussion indicated higher sewage sludge levels of PCB and D/F compounds on Run 4 which might serve to explain the lower air sample results. This, however, does not explain why the field surrogate spike recoveries were low.

- Run 4 XAD-2 Resin Lost Prior to Transfer to Soxhlet Extractor. This event would result in a volume loss of analytes including the pre-field surrogate spikes. However, laboratory record books do not collaborate such an event.

- Incorrect Sample Volume Used in Calculations. If an incorrect sample volume was used to calculate emission concentrations in ug/dscm, Run 4 emission concentrations could be affected. This would affect both front and back half results for Run 4. A check of field data reduction does not indicate any calculation error.

- Improper Spiking of Laboratory Internal or Recovery Standards. An incorrect amount of internal or recovery standards could have been added to the Run 4 samples. However, since native PCB concentrations are quantified against pre-extraction internal standards and PCB pre-field surrogate spikes are quantified against the pre-analysis recovery standard, this cause would require incorrect spiking on multiple occasions which is unlikely.

2.3.1 Toxic PCB Results

The toxic PCB results in ng/dscm are summarized in Tables 2-1 and 2-2. Toxic PCB results for Runs 2 and 3 are almost identical for most of the analytes. The back half emission concentrations for Run 4 are 50 to 60 percent lower than the back half emission concentrations for Runs 2 and 3 for all the analytes. Possible loss of PCBs may have occurred in the field as indicated by lower pre-field surrogate recoveries for Run 4 (use Section 6.1.3 for discussion). The PCB data have been reviewed extensively, and no reason can be found for the data differential. Alternatively, these lower Run 4 concentrations may be an accurate reflection of a change in incinerator emissions on the third day of sampling. Table 2-3 presents toxic PCB results in World Health Organization (WHO) Toxic Equivalencies which are an estimate of the concentration of 2,3,7,8-TCDD which would produce an equivalent toxicity as the PCB. The Toxic Equivalent Factors (TEFs) for the toxic PCBs are presented in Table 2-4.

Table 2-1. Toxic PCB Results - Stack Gas Concentrations (ng/dscm, as measured)

PCB Congener	Concentration (ng/dscm, as measured)[a]		
	Run 2	Run 3	Run 4
3,3',4,4'-tetrachlorobiphenyl (TCB) (PCB-77)[b]	15.6	10.7	4.27
2,3,3',4,4'-pentachlorobiphenyl (PeCD) (PCB-105)	2.67	2.45	0.945
2,3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-114)	0.389	0.340	0.137
2,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-118)	5.72	5.27	2.21
2',3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-123)	0.121	0.111	0.038
3,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-126)	0.700	0.584	0.210
2,3,3',4,4',5-hexachlorobiphenyl (HxCB) (PCB-156)	0.645	0.565	0.213
2,3,3',4,4',5'-hexachlorobiphenyl (HxCB) (PCB-157)	0.221	0.179	0.079
2,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-167)	0.388	0.337	0.136
3,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-169)	0.559	0.467	0.141
2,2',3,3',4,4',5-heptachlorobiphenyl (HpCB) (PCB-170)	1.08	0.966	0.437
2,2',3,4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-180)	2.69	2.37	0.856
2,3,3',4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-189)	0.095	0.076	0.044

[a] ng/dscm; nanogram per dry standard cubic meter.
Standard conditions: temperature - 20°C; pressure - 1 atm (760 mm Hg).

[b] Back half extracts diluted with additional internal standard and re-analyzed to bring the reported concentrations within the calibration range (see Section 6.1.3.1).

Table 2-2. Toxic PCB Results - Stack Gas Concentrations (ng/dscm, adjusted to 7% O_2)

PCB Congener	Concentration (ng/dscm, adjusted to 7% O_2)[a]		
	Run 2	Run 3	Run 4
3,3',4,4'-tetrachlorobiphenyl (TCB) (PCB-77)[b]	30.6	18.8	7.92
2,3,3',4,4'-pentachlorobiphenyl (PeCB) (PCB-105)	5.22	4.32	1.75
2,3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-114)	0.762	0.598	0.254
2,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-118)	11.2	9.27	4.10
2',3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-123)	0.237	0.195	0.070
3,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-126)	1.37	1.03	0.389
2,3,3',4,4',5-hexachlorobiphenyl (HxCB) (PCB-156)	1.26	0.994	0.395
2,3,3',4,4',5'-hexachlorobiphenyl (HxCB) (PCB-157)	0.433	0.315	0.146
2,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-167)	0.760	0.593	0.252
3,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-169)	1.09	0.822	0.261
2,2',3,3',4,4',5-heptachlorobiphenyl (HpCB) (PCB-170)	2.11	1.70	0.810
2,2',3,4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-180)	5.26	4.18	1.59
2,3,3',4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-189)	0.186	0.134	0.082

[a] ng/dscm; nanogram per dry standard cubic meter, adjusted to 7% oxygen. Standard conditions: temperature - 20°C; pressure - 1 atm (760 mm Hg).

[b] Back half extracts diluted with additional internal standard and re-analyzed to bring the reported concentrations within the calibration range (see Section 6.1.3.11.

Table 2-3. Toxic PCB Results - WHO Toxic Equivalent Stack Gas Concentrations (ng/dscm, adjusted to 7% O_2)

PCB Congener	WHO Toxic Equivalencies (ng/dscm, adjusted to 7%O_2)[a]			
	WHO[c] TEFs	Run 2	Run 3	Run 4
3,3',4,4'-tetrachlorobiphenyl (TCB) (PCB-77)[(b)]	1.0E-04	3.06E-03	1.88E-03	7.92E-04
2,3,3',4,4'-pentachlorobiphenyl (PeCB) (PCB-105)	1.0E-04	5.23E-04	4.32E-04	1.75E-04
2,3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-114)	5.0E-04	3.81E-04	2.99E-04	1.27E-04
2,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-118)	1.0E-04	1.12E-03	9.27E-04	4.10E-04
2',3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-123)	1.0E-04	2.37E-05	1.95E-05	7.04E-06
3,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-126)	0.1	1.37E-01	1.03E-01	3.89E-02
2,3,3',4,4',5-hexachlorobiphenyl (HxCB) (PCB-156)	5.0E-04	6.31E-04	4.97E-04	1.97E-04
2,3,3',4,4',5'-hexachlorobiphenyl (HxCB) (PCB-157)	5.0E-04	2.16E-04	1.57E-04	7.32E-05
2,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-167)	1.0E-05	7.60E-06	5.93E-06	2.52E-06
3,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-169)	0.01	1.09E-02	8.22E-03	2.61E-03
2,2',3,3',4,4',5-heptachlorobiphenyl (HpCB) (PCB-170)	1.0E-04	2.11E-04	1.70E-04	8.10E-05
2,2',3,4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-180)	1.0E-05	5.26E-05	4.18E-05	1.59E-05
2,3,3',4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-189)	1.0E-04	1.86E-05	1.34E-05	8.15E-06

[a] ng/dscm; nanogram per dry standard cubic meter, adjusted to 7% oxygen.
[b] Back half extracts diluted with additional internal standard and re-analyzed to bring the reported concentrations within the calibration range (see Section 6.1.3.11.
[c] WHO TEFs - World Health Organization, Toxic Equivalent Factors.
Standard conditions: temperature - 20°C; pressure - 1 atm (760 mm Hg).

Table 2-4. World Health Organization Toxic Equivalent Factors (TEFs) for Determining Toxic PCB TEQs

Type Compound	Congener		
	IUPAC[a] No.	Structure	TEF
Non-ortho	77	3,3',4,4'-tetrachlorobiphenyl (TCB)	0.0001
	126	2,3,3',4'5-pentachlorobiphenyl (PeCB)	0.1
	169	3,3',4,4',5,5'-hexachlorobiphenyl (HxCB)	0.01
Mono-ortho	105	2,3',3',4,4'-pentachlorobiphenyl (PeCB)	0.0001
	114	2,3,4,4',5-pentachlorobiphenyl (PeCB)	0.0005
	118	2,3',4,4',5-pentachlorobiphenyl (PeCB)	0.0001
	123	2,3,3',4,4',5-hexachlorobiphenyl (PeCB)	0.0001
	156	2,3,3',4,4',5'-hexachlorobiphenyl (HxCB)	0.0005
	157	2,3,3',4,4',5'-hexachlorobiphenyl (HxCB)	0.0005
	167	2,3',4,4',5,5'-hexachlorobiphenyl (HxCB)	0.00001
	189	2,3,3',4,4',5,5'-heptachlorobiphenyl (HpCB)	0.0001
Di-ortho	170	2,2',3,3',4,4',5-heptachlorobiphenyl (HpCB)	0.0001
	180	2,2',3,4,4',5,5'-heptachlorobiphenyl (HpCB)	0.00001

[a] IUPAC = International Union of Pure and Applied Chemistry.

Note: World Health Organization (WHO) TEFs for human risk assessment based on the conclusions of the WHO consultation in Stockholm, Sweden, 15-18 June 1997 (Van der Berg et al., 1998).

2.3.2 Dioxin/Furan (D/F) Results

The dioxin/furan results are summarized in Tables 2-5 through 2-7. A detailed discussion of the QA/QC results associated with these D/F data appears in Section 6.1.3. D/F results for Runs 2 and 3 are almost identical for most of the analytes. The back half emission concentrations for Run 4 are 50 to 60 percent lower than the back half emission concentrations for Runs 2 and 3 for all the analytes. These lower Run 4 concentrations may be due to analyte loss or an accurate reflection of a change in incinerator emissions on the third day of sampling. The D/F data have been reviewed extensively, and no reason can be found for the data differential.

Table 2-5. D/F Results - Stack Gas Concentrations (ng/dscm, as measured)

Congener	Concentration (ng/dscm, as measured)[a]		
	Run 2	Run 3	Run 4
Dioxins			
2,3,7,8-TCDD #	0.098	0.067	0.034
Total TCDD	3.02	3.53	0.719
1,2,3,7,8-PCDD	0.017	0.013	(0.005)
Total PCDD	0.706	0.658	0.187
1,2,3,4,7,8-HxCDD	0.015	0.015	0.006
1,2,3,6,7,8-HxCDD	0.038	0.040	0.013
1,2,3,7,8,9-HxCDD	0.039	0.035	0.018
Total HxCDD	0.606	0.584	0.324
1,2,3,4,6,7,8-HpCDD	0.204	0.202	0.093
Total HpCDD	0.459	1.45	0.237
Octa CDD	0.317	0.303	0.140
Total CDD Based on given numbers:	5.11	6.53	1.61
Furans			
2,3,7,8-TCDF #	1.58	1.24	0.533
Total TCDF	5.61	4.88	2.57
1,2,3,7,8-PCDF	0.195	0.150	0.068
2,3,4,7,8-PCDF	0.389	0.283	0.123
Total PCDF	4.93	3.67	1.54
1,2,3,4,7,8-HxCDF	0.226	0.177	0.092
1,2,3,6,7,8-HxCDF	0.081	0.067	0.035
2,3,4,6,7,8-HxCDF	0.126	0.097	0.051
1,2,3,7,8,9-HxCDF	ND<0.003	ND<0.003	ND<0.003
Total HxCDF	1.13	0. 880	0.410
1,2,3,4,6,7,8-HpCDF	0.224	0.185	0.103
1,2,3,4,7,8,9-HpCDF	0.023	0.017	0.009
Total HpCDF	0.329	0.261	0.137
Octa CDF	0.090	0.082	0.041
Total CDF Based on given numbers:	12.1	9.77	4.69
Total CDD + CDF Based on given numbers:	17.2	16.3	6.30

[a] ng/dscm; nanogram per dry standard cubic meter.
Standard conditions, pressure and temperature defined as 1 atm (760 mm Hg) and 20°C.

Note: (Below Detection Limit) values listed in parentheses.
ND = Non Detect. # = value from confirmation column. Non Detect and (Below Detection Limit) values not included in totals.

Table 2-6. D/F Results - Stack Gas Concentrations (ng/dscm, adjusted to 7% O_2)

Congener	Concentration (ng/dscm, adjusted to 7% O_2)[a]		
	Run 2	Run 3	Run 4
Dioxins			
2,3,7,8-TCDD	0.209	0.118	0.063
Total TCDD	5.92	6.22	1.33
1,2,3,7,8-PCDD	0.033	0.023	(0.009)
Total PCDD	1.38	1.16	0.348
1,2,3,4,7,8-HxCDD	0.029	0.026	0.011
1,2,3,6,7,8-HxCDD	0.074	0.069	0.024
1,2,3,7,8,9-HxCDD	0.076	0.062	0.030
Total HxCDD	1.19	1.03	0.600
1,2,3,4,6,7,8-HpCDD	0.399	0.357	0.172
Total HpCDD	0.899	2.560	0.439
Octa CDD	0.621	0.424	0.259
Total CDD	10.0	11.4	2.97
Furans			
2,3,7,8-TCDF	3.10	2.17	0.988
Total TCDF	11.00	8.58	4.76
1,2,3,7,8-PCDF	0.382	0.264	0.124
2,3,4,7,8-PCDF	0.762	0.496	0.228
Total PCDF	9.66	6.45	2.85
1,2,3,4,7,8-HxCDF	0.442	0.313	0.169
1,2,3,6,7,8-HxCDF	0.159	0.116	0.065
2,3,4,6,7,8-HxCDF	0.247	0.171	0.095
1,2,3,7,8,9-HxCDF	ND<0.0059	ND<0.0053	ND<0.0056
Total HxCDF	2.20	1.55	0.760
1,2,3,4,6,7,8-HpCDF	0.437	0.324	0.189
1,2,3,4,7,8,9-HpCDF	0.043	0.030	0.017
Total HpCDF	0.642	0.459	0.252
Octa CDF	0.176	0.146	0.078
Total CDF	23.7	17.2	8.71
Total CDD + CDF	33.7	28.6	11.7

[a] ng/dscm; nanogram per dry standard cubic meter, adjusted to 7% oxygen. Standard conditions, pressure and temperature defined as 1 atm (760 mm Hg) and 20°C.

Note: (Below Detection Limit) values listed in parentheses.
Non Detects and (Below Detection Limit) values not included in totals.
ND = Non detect, value is detection limit.

Table 2-7. D/F Results - TEQ Stack Gas Concentrations (ng/dscm, adjusted to 7% O_2)

Congener	2,3,7,8-TCDD Toxic Equivalence Factor	Concentration (ng/dscm, adjusted to 7% O_2)[a]		
		Run 2	Run 3	Run 4
Dioxins				
2,3,7,8-TCDD	1.000	0.209	0.118	0.063
Total TCDD				
1,2,3,7,8-PCDD	0.500	0.017	0.012	(0.005)
Total PCDD				
1,2,3,4,7,8-HxCDD	0.100	0.003	0.003	0.001
1,2,3,6,7,8-HxCDD	0.100	0.007	0.007	0.002
1,2,3,7,8,9-HxCDD	0.100	0.008	0.006	0.003
Total HxCDD				
1,2,3,4,6,7,8-HpCDD	0.010	0.004	0.004	0.002
Total HpCDD				
Octa CDD	0.001	0.00062	0.00053	0.00026
2,3,7,8-TEQ Total CDD		0.249	0.151	0.071
Furans				
2,3,7,8-TCDF	0.100	0.310	0.218	0.099
Total TCDF				
1,2,3,7,8-PCDF	0.050	0.019	0.013	0.006
2,3,4,7,8-PCDF	0.500	0.381	0.248	0.114
Total PCDF				
1,2,3,4,7,8-HxCDF	0.100	0.044	0.031	0.017
1,2,3,6,7,8-HxCDF	0.100	0.016	0.012	0.007
2,3,4,6,7,8-HxCDF	0.100	0.025	0.017	0.010
1,2,3,7,8,9-HxCDF	0.100	ND<0.0006	ND<0.0005	ND<0.0006
Total HxCDF				
1,2,3,4,6,7,8-HpCDF	0.010	0.004	0.003	0.002
1,2,3,4,7,8,9-HpCDF	0.010	0.000	0.000	0.000
Total HpCDF				
Octa CDF	0.001	0.000	0.000	0.000
2,3,7,8-TEQ Total CDF		0.799	0.542	0.255
2,3,7,8-TEQ Total CDD + CDF		1.05	0.693	0.326

[a] ng/dscm; nanogram per dry standard cubic meter, adjusted to 7% oxygen. Standard conditions, pressure and temperature defined as 1 atm (760 mm Hg) and 20°C.

Note: (Below Detection Limit) values listed in parentheses. Non Detects and (Below Detection Limit) values not included in totals.

2.3.3 PAH Results

The PAH results are summarized in Tables 2-8 and 2-9. A detailed discussion of the QA/QC results associated with these PAH data appears in Section 6.1.3. PAH Results for Runs 2 and 3 are somewhat similar for most of the analytes. The back half emission concentrations for Run 4 are 50 to 60 percent lower than the back half emission concentrations for Runs 2 and 3 for most of the analytes. These lower Run 4 concentrations may be due to analyte loss or an accurate reflection of a change in incinerator emissions on the third day of sampling. The PAH data have been reviewed extensively, and no definitive reason can be found for the data differential (see Sections 2.3 and 6.1.3).

Table 2-8. PAH Results - Stack Gas Concentrations (ng/dscm, as measured)

Compound	Concentration (ng/dscm, as measured)[a]		
	Run 2	Run 3	Run 4
Acenaphthene	109	83.6	15.5
Acenaphthylene	{1160}D	{1290}E	{155}
Anthracene	{144}	{52.7}	55.5
Benzo(a)anthracene	93.0	251	49.0
Benzo(b)fluoranthene	828	662	159
Benzo(k)fluoranthene	759	361	93.7
Benzo(g,h,i)perylene	233	134	50.7
Benzo(a)pyrene	75.2	34.1	NQ*
Chrysene	2390 E	{1340}E	288
Dibenzo(a,h)anthracene	71.8	48.9	12.5
Fluoranthene	2700 E	{2340}	557
Fluorene	1030	1090	77.6
Indeno(1,2,3-cd)pyrene	244	166	44.2
Naphthalene	{309000}D,E	{245000}D,E	{126000}D,E
Phenanthrene	{22600}D,E	{21200}D,E	{4780}E
Pyrene	1810 E	1560 E	292

[a] ng/dscm = nanogram per dry standard cubic meter. Standard conditions, pressure and temperature defined as 1 atm (760 mm Hg) and 20°C.

Note: Estimated Maximum Possible Concentration {EMPC} values listed in brackets.
D based on dilution.
E Exceeds calibration range.
NQ* d_{12}-benzo(a)pyrene recovery in Sample Run 4 was too low to quantify the compound. Maximum concentration of the compound is estimated by quantitation of benzo(e)pyrene at 161 ng/dscm.

Table 2-9. PAH Results - Stack Gas Concentrations (ng/dscm, adjusted to 7% O_2)

Compound	Concentration (ng/dscm adjusted to 7% O_2)[a]		
	Run 2	Run 3	Run 4
Acenaphthene	214	147	28.8
Acenaphthylene	{2280}D	{2260}E	{288}
Anthracene	{281}	{92.8}	103
Benzo(a)anthracene	182	441	90.7
Benzo(b)fluoranthene	1620	1160	294
Benzo(k)fluoranthene	1490	636	174
Benzo(g,h,i)perylene	455	236	94.0
Benzo(a)pyrene	147	60.0	NQ*
Chrysene	4690 E	{2350}E	534
Dibenzo(a,h)anthracene	141	86.0	23.2
Fluoranthene	5290 E	{4110}	1030
Fluorene	2010	1920	144
Indeno(1,2,3-cd)pyrene	478	292	81.9
Naphthalene	{605000}D,E	{431000}D,E	{233000}D,E
Phenanthrene	{44400}D,E	{37300}D,E	{8850}E
Pyrene	3540 E	2750 E	542

[a] ng/dscm = nanogram per dry standard cubic meter, adjusted to 7% oxygen. Standard conditions, pressure and temperature defined as 1 atm (760 mm Hg) and 20°C.

Note: Estimated Maximum Possible Concentration {EMPC} values listed in brackets.
D based on dilution.
E Exceeds calibration range.
NQ* d_{12}-benzo(a)pyrene recovery in Sample Run 4 was too low to quantify the compound. Maximum concentration of the compound is estimated by quantitation of benzo(e)pyrene at 96.1 ng/dscm.

2.4 CONTINUOUS EMISSIONS MONITORING SUMMARY AND DISCUSSION

Average daily results from continuous emission monitoring of carbon monoxide (CO), total hydrocarbons (THC), carbon dioxide (CO_2), and oxygen (O_2) are provided in Table 2-10. Plots of individual CO and THC data are provided in Figures 2-1, 2-2, and 2-3 for Runs 2, 3, and 4, respectively. The CO data scale appears along the left side of the plot with the THC data points plotted hourly along the bottom and scaled on the right-hand side of each figure. Appendix A (Tables A-3 through A-6) of the CEM data includes daily maximum and minimum values, arithmetic mean, and % standard deviation for each sampling period. The THC data presented in Appendix A were provided by MSD. THC, CO, and O_2 arithmetic means are calculated from data points collected during each sampling run as indicated from clock time. Detailed CEM data and calibration information are presented separately in Appendix L.

Table 2-10. CEM Daily Results

CEM	Daily Test Run Averages			
	Run 2	Run 3	Run 4	Average
CO[a], ppm_{dv}	1380	1170	1130	1230
THC[b], ppm_{dv}	70.6	54.2	37.5	54.1
CO_2[a], % v	5.16	5.50	5.07	5.24
O_2[a], % v	13.7	13.0	13.4	13.4

[a] CO, CO_2, and O_2 analyzer data calibration corrected from 1-minute averages during the 360 minute sampling run.

[b] THC analyzer data calibration corrected from the arithmetic average of hourly reported values from MSD during the sampling runs.

CO, CO_2, and O_2 emission concentrations appear to be relatively constant across the three runs. The THC concentrations for Run 4 are much lower than the THC concentrations for Runs 2 and 3.

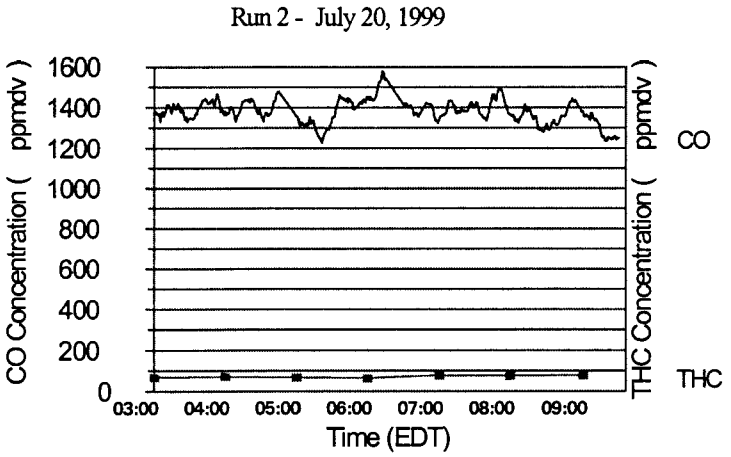


Figure 2-1. CO and THC Daily Test Results - Run 2

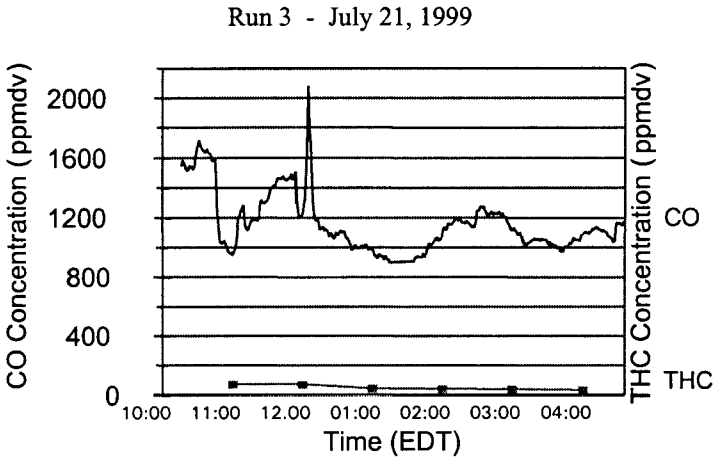


Figure 2-2. CO and THC Daily Test Results - Run 3

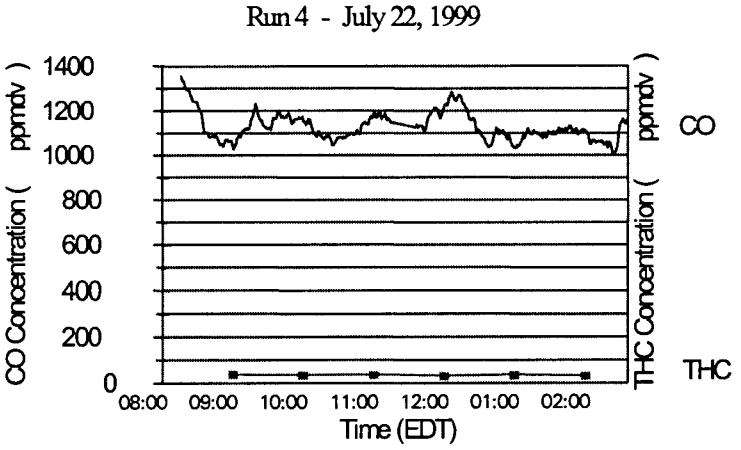


Figure 2-3. CO and THC Daily Test Results - Run 4

2.5 PROCESS SAMPLE MEASUREMENTS SUMMARY AND DISCUSSION

Test results for scrubber water and sewage sludge feed which were collected during the MM5 sampling runs are presented in this section. Detailed PCB and D/F analytical data for these matrices are provided in Appendix E and F, respectively. Appendices H, I, and J include detailed data and information for chlorine, ultimate/proximate, and percent solids analysis, respectively.

2.5.1 Scrubber Water Organic Results

Scrubber water samples were analyzed for PCB and D/F.

2.5.1.1 Toxic PCB Comparison of Scrubber Water In Versus Scrubber Water Out

Table 2-11 presents the PCB comparison between the inlet and outlet scrubber water samples for Run 2, 3, and 4 respectively. In general, the PCB concentrations in the inlet scrubber water samples are slightly lower than PCB concentrations in the outlet scrubber water samples although this result varies from run to run and from PCB congener to PCB congener.

2.5.1.2 Toxic PCB Results For Scrubber Water In

Table 2-12 compares the inlet scrubber water results for all three runs. The PCB concentrations in the inlet scrubber water samples were generally consistent throughout the three runs.

Table 2-11. Run 2, Run 3, and Run 4 Toxic PCB Results - Comparison of Inlet Versus Outlet Scrubber Water

PCB Congener	Concentration (ng/L, as measured)					
	Run 2 July 20, 1999		Run 3 July 21, 1999		Run 4 July 22, 1999	
	In	Out	In	Out[a]	In[a]	Out
3,3',4,4'-tetrachlorobiphenyl (TCB) (PCB-77)	0.158	4.18	0.166	3.63	0.138	2.52
2,3,3',4,4'-pentachlorobiphenyl (PeCB) (PCB-105)	0.690	0.946	0.491	1.87	0.504	0.854
2,3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-114)	0.233	0.157	0.068	0.155	0.045	0.124
2,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-118)	1.20	1.68	1.06	3.52	1.06	1.62
2',3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-123)	0.177	0.086	0.032	0.065	0.018	0.050
3,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-126)	0.024	0.135	0.007	0.118	0.004	0.098
2,3,3',4,4',5-hexachlorobiphenyl (HxCB) (PCB-156)	0.243 #	0.249 #	0.085 #	0.413 #	0.080 #	0.236 #
2,3,3',4,4',5'-hexachlorobiphenyl (HxCB) (PCB-157)	0.166 #	0.119 #	0.285 #	0.110 #	0.020 #	0.083 #
2,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-167)	0.186	0.158	0.044	0.164	0.036	0.125
3,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-169)	0.027	0.113	0.006	0.081	(0.002)	0.098
2,2',3,3',4,4',5-heptachlorobiphenyl (HpCB) (PCB-170)	0.105	0.264	0.080	0.414	0.078	0.298
2,2'3,4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-180)	0.338	0.686	0.190	1.00	0.177	0.653
2,3.3',4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-189)	0.029	0.030	0.009	0.024	{0.005}	0.044

[a] Re-analyzed result - see Section 6.1.3.2 discussion.
Note: # Values from second column confirmation.

Table 2-12. Toxic PCB Results - Inlet Scrubber Water

PCB Congener	Concentration (ng/L, as measured)		
	Run 2	Run 3	Run 4[a]
3,3',4,4'-tetrachlorobiphenyl (TCB) (PCB-77)	0.158	0.166	0.138
2,3,3',4,4'-pentachlorobiphenyl (PeCB) (PCB-105)	0.690	0.491	0.504
2,3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-114)	0.233	0.068	0.045
2,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-118)	1.20	1.06	1.06
2',3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-123)	0.177	0.032	0.018
3,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-126)	0.024	0.007	0.004
2,3,3',4,4',5-hexachlorobiphenyl (HxCB) (PCB-156)	0.243 #	0.085 #	0.080 #
2,3,3',4,4',5'-hexachlorobiphenyl (HxCB) (PCB-157)	0.166 #	0.285 #	0.020 #
2,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-167)	0.186	0.044	0.036
3,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-169)	0.027	0.006	0.002
2,2',3,3',4,4',5-heptachlorobiphenyl (HpCB) (PCB-170)	0.105	0.080	0.078
2,2',3,4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-180)	0.338	0.190	0.177
2,3,3',4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-189)	0.029	0.009	{0.005}

[a] Re-analyzed result - see Section 6.1.3.2 discussion.
Note: {EMPC} values listed in brackets.
Values from second column confirmation.

2.5.1.3 Toxic PCB Results For Scrubber Water Out

Table 2-13 compares the outlet scrubber water results for all three runs. The PCB concentrations in the three outlet scrubber water samples are comparable. The Run 3 sample was re-analyzed after a laboratory error using the archived sample. This is discussed further in Section 6.1.3.2.

2.5.1.4 Dioxin/Furan Results for Scrubber Water

D/F concentrations for scrubber water samples are presented in Tables 2-14 through 2-16. The comparison of D/F concentrations in inlet versus outlet scrubber water samples in Table 2-14 suggests that outlet concentrations are higher than inlet concentrations since most D/F congeners were not detected in the inlet water samples. However, the detection limit for the inlet scrubber water samples in many cases is higher than the concentration found in the outlet scrubber water samples so an evaluation of inlet versus outlet concentrations is difficult to make. As shown again in Table 2-15, most D/F concentrations in all three inlet scrubber water samples were below detection limits. The D/F concentrations in outlet scrubber water samples as shown in Table 2-16 were generally comparable across the three runs in that D/F congeners found at higher levels in one run (compared to other D/F congeners) would be found at relatively higher levels in the other two runs as well.

2.5.2 Sewage Sludge Organic Results

Sewage sludge samples were analyzed for PCB and D/F. Sewage sludge feed was sampled and analyzed for PCBs and D/Fs consistent with the scrubber water. Results for sewage sludge feed are presented in Table 2-17 for PCBs and in Table 2-18 for D/Fs. In general, PCB and D/F concentrations in the sludge feed are comparable across the three runs. However, the overall higher (20% above the mean) sewage sludge D/F concentrations in Run 4 may hint at a process variation which may have resulted in higher organic contaminant levels in the sludge which did not result in higher PCB, D/F, and PAH emission concentrations in Run 4.

Table 2-13. Toxic PCB Results - Outlet Scrubber Water

PCB Congener	Concentration (ng/L, as measured)		
	Run 2	Run 3[a]	Run 4
3,3',4,4'-tetrachlorobiphenyl (TCB) (PCB-77)	4.18	3.63	2.52
2,3,3',4,4'-pentachlorobiphenyl (PeCB) (PCB-105)	0.946	1.87	0.854
2,3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-114)	0.157	0.155	0.124
2,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-118)	1.68	3.52	1.62
2',3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-123)	0.086	0.065	0.050
3,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-126)	0.135	0.118	0.098
2,3,3',4,4',5-hexachlorobiphenyl (HxCB) (PCB-156)	0.249 #	0.413 #	0.236 #
2,3,3',4,4',5'-hexachlorobiphenyl (HxCB) (PCB-157)	0.119 #	0.110 #	0.083 #
2,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-167)	0.158	0.164	0.125
3,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-169)	0.113	0.081	0.098
2,2',3,3',4,4',5-heptachlorobiphenyl (HpCB) (PCB-170)	0.264	0.414	0.298
2,2',3,4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-180)	0.686	1.00	0.653
2,3,3',4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-189)	0.030	0.024	0.044

[a] Re-analyzed result - see Section 6.1.3.2 discussion.
Note: # Values from second column confirmation.

Table 2-14. D/F Results - Comparison of Inlet Versus Outlet Scrubber Water

Congener	Scrubber Water Concentrations (ng/L, as measured)					
	Run 2		Run 3		Run 4	
	In	Out	In	Out	In	Out
Dioxins						
2,3,7,8-TCDD	ND<0.013	0.009 #	ND<0.003	0.013 #	ND<0.008	0.014 #
Total TCDD	ND<0.029	0.475	(0.002)	0.821	ND<0.017	0.277
1,2,3,7,8-PCDD	ND<0.027	ND<0.005	ND<0.007	ND<0.013	ND<0.023	ND<0.004
Total PCDD	ND<0.046	0.046	ND<0.012	0.057	ND<0.039	{0.066}
1,2,3,4,7,8-HxCDD	ND<0.013	0.002	ND<0.002	ND<0.004	ND<0.006	0.003
1,2,3,6,7,8-HxCDD	ND<0.013	0.003	ND<0.002	ND<0.004	ND<0.006	0.006
1,2,3,7,8,9-HxCDD	ND<0.013	{0.003}	ND<0.002	ND<0.004	ND<0.006	0.006
Total HxCDD	ND<0.029	{0.029}	ND<0.005	0.054	ND<0.004	0.102
1,2,3,4,6,7,8-HpCDD	ND<0.033	{0.011}	ND<0.004	0.025	ND<0.012	0.032
Total HpCDD	ND<0.099	0.027	ND<0.013	0.053	ND<0.037	0.074
Octa CDD	0.040	0.030	0.012	0.059	0.016	0.047
Total CDD	0.040	0.578	0.012	1.040	0.016	0.500
Furans						
2,3,7,8-TCDF	ND<0.009	0.181 #	ND<0.002	0.222 #	ND<0.005	0.256 #
Total TCDF	0.092	0.824	(0.002)	1.120	ND<0.015	0.866
1,2,3,7,8-PCDF	ND<0.018	0.014	ND<0.004	0.025	ND<0.011	0.025
2,3,4,7,8-PCDF	ND<0.011	0.029	ND<0.002	0.045	ND<0.005	0.053
Total PCDF	ND<0.035	0.316	ND>0.006	0.493	ND<0.019	0.495
1,2,3,4,7,8-HxCDF	ND<0.014	0.012	ND<0.002	0.016	ND<0.006	0.025
1,2,3,6,7,8-HxCDF	ND<0.013	0.005	ND<0.002	0.007	ND<0.0006	0.011
2,3,4,6,7,8-HxCDF	ND<0.017	{0.006}	ND<0.002	0.013	ND<0.007	0.017
1,2,3,7,8,9-HxCDF	ND<0.014	ND<0.002	ND<0.002	ND<0.005	ND<0.008	ND<0.002
Total HxCDF	ND<0.053	0.048	ND<0.002	0.080	ND<0.025	0.113
1,2,3,4,6,7,8-HpCDF	ND<0.009	0.012	0.001	0.020	ND<0.003	0.025
1,2,3,4,7,8,9-HpCDF	ND<0.035	ND<0.003	ND<0.005	ND<0.008	ND<0.013	ND<0.003
Total HpCDF	ND <0.079	0.017	(0.004)	0.029	(0.003)	{0.035}
Octa CDF	ND<0.047	0.006	ND<0.006	0.010	ND<0.016	0.008
Total CDF	0.092	1.21	0.001	1.73	0.000	1.48
Total CDD + CDF	0.132	1.79	0.013	2.78	0.016	1.98

Note: (Below Detection Limit) values listed in parentheses and {EMPC} values listed in brackets.
ND = Non Detect, value is detection limit.
# = value from second column confirmation.
Non Detects and (Below Detection Limit) values not included in totals.

Table 2-15. D/F Results for Inlet Scrubber Water

Congener	Concentration (ng/L, as measured)		
	Run 2	Run 3	Run 4
Dioxins			
2,3,7,8-TCDD	ND<0.013	ND<0.003	ND<0.008
Total TCDD	ND<0.029	(0.002)	ND<0.017
1,2,3,7,8-PCDD	ND<0.027	ND<0.007	ND<0.023
Total PCDD	ND<0.046	ND<0.012	ND<0.039
1,2,3,4,7,8-HxCDD	ND<0.013	ND<0.002	ND<0.006
1,2,3,6,7,8-HxCDD	ND<0.013	ND<0.002	ND<0.006
1,2,3,7,8,9-HxCDD	ND<0.013	ND<0.002	ND<0.006
Total HxCDD	ND<0.029	ND<0.005	ND<0.004
1,2,3,4,6,7,8-HpCDD	ND<0.033	ND<0.004	ND<0.012
Total HpCDD	ND<0.099	ND<0.013	ND<0.037
Octa CDD	0.040	0.012	0.016
Total CDD	0.040	0.012	0.016
Furans			
2,3,7,8-TCDF	ND<0.009	ND<0.002	ND<0.005
Total TCDF	0.092	(0.002)	ND<0.015
1,2,3,7,8-PCDF	ND<0.018	ND<0.004	ND<0.011
2,3,4,7,8-PCDF	ND<0.011	ND<0.002	ND<0.005
Total PCDF	ND<0.035	ND>0.006	ND<0.019
1,2,3,4,7,8-HxCDF	ND<0.014	ND<0.002	ND<0.006
1,2,3,6,7,8-HxCDF	ND<0.013	ND<0.002	ND<0.0006
2,3,4,6,7,8-HxCDF	ND<0.017	ND<0.002	ND<0.007
1,2,3,7,8,9-HxCDF	ND<0.014	ND<0.002	ND<0.008
Total HxCDF	ND<0.053	ND<0.002	ND<0.025
1,2,3,4,6,7,8-HpCDF	ND<0.009	0.001	ND<0.003
1,2,3,4,7,8,9-HpCDF	ND<0.035	ND<0.005	ND<0.013
Total HpCDF	ND <0.079	(0.004)	(0.003)
Octa CDF	ND<0.047	ND<0.006	ND<0.016
Total CDF	0.092	0.001	0.000
Total CDD + CDF	0.132	0.013	0.016

Note: (Below Detection Limit) values listed in parentheses.
ND = Non Detect.
Non Detects and (Below Detection Limits) values not included in totals.

Table 2-16. D/F Results for Outlet Scrubber Water

Congener	Concentration (ng/L, as measured)		
	Run 2	Run 3	Run 4
Dioxins			
2,3,7,8-TCDD	0.009 #	0.013 #	0.014 #
Total TCDD	0.475	0.821	0.277
1,2,3,7,8-PCDD	ND<0.005	ND<0.013	ND<0.004
Total PCDD	0.046	0.057	{0.066}
1,2,3,4,7,8-HxCDD	0.002	ND<0.004	0.003
1,2,3,6,7,8-HxCDD	0.003	ND<0.004	0.006
1,2,3,7,8,9-HxCDD	{0.003}	ND<0.004	0.006
Total HxCDD	{0.029}	0.054	0.102
1,2,3,4,6,7,8-HpCDD	{0.011}	0.025	0.032
Total HpCDD	0.027	0.053	0.074
Octa CDD	0.030	0.059	0.047
Total CDD	0.578	1.04	0.500
Furans			
2,3,7,8-TCDF	0.181 #	0.222 #	0.256 #
Total TCDF	0.824	1.120	0.866
1,2,3,7,8-PCDF	0.014	0.025	0.025
2,3,4,7,8-PCDF	0.029	0.045	0.053
Total PCDF	0.316	0.493	0.495
1,2,3,4,7,8-HxCDF	0.012	0.016	0.025
1,2,3,6,7,8-HxCDF	0.005	0.007	0.011
2,3,4,6,7,8-HxCDF	{0.006}	0.013	0.017
1,2,3,7,8,9-HxCDF	ND<0.002	ND<0.005	ND<0.002
Total HxCDF	0.048	0.080	0.113
1,2,3,4,6,7,8-HpCDF	0.012	0.020	0.025
1,2,3,4,7,8,9-HpCDF	ND<0.003	ND<0.008	ND<0.003
Total HpCDF	0.017	0.029	{0.035}
Octa CDF	0.006	0.010	0.008
Total CDF	1.21	1.73	1.48
Total CDD + CDF	1.79	2.78	1.98

Note: {EMPC} values listed in brackets.
ND = Non Detect, value is detection limit
# = value from second column confirmation.
Non Detects and {EMPC} values not included in totals.

Table 2-17. Toxic PCB Results for Sewage Sludge

PCB Congener	Concentration (ng/g, dry)		
	Run 2	Run 3	Run 4
3,3',4,4'-tetrachlorobiphenyl (TCB) (PCB-77)	40.9	41.1	45.4
2,3,3',4,4'-pentachlorobiphenyl (PeCB) (PCB-105)	7.01	7.39	7.29
2,3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-114)	0.691	0.674	0.738
2,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-118)	12.2	13.5	12.9
2',3,4,4',5-pentachlorobiphenyl (PeCB) (PCB-123)	0.231	0.276	0.241
3,3',4,4',5-pentachlorobiphenyl (PeCB) (PCB-126)	1.12	1.21	1.48
2,3,3',4,4',5-hexachlorobiphenyl (HxCB) (PCB-156)	1.77 #	1.88 #	1.88 #
2,3,3',4,4',5'-hexachlorobiphenyl (HxCB) (PCB-157)	0.472 #	0.565 #	0.536 #
2,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-167)	0.878	0.968	0.959
3,3',4,4',5,5'-hexachlorobiphenyl (HxCB) (PCB-169)	0.453	0.601	0.656
2,2',3,3',4,4',5-heptachlorobiphenyl (HpCB) (PCB-170)	2.53	2.57	2.78
2,2',3,4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-180)	6.00	6.78	6.71
2,3,3',4,4',5,5'-heptachlorobiphenyl (HpCB) (PCB-189)	0.181	0.198	0.218

Note: # Values from second column confirmation.

Table 2-18. D/F Results for Sewage Sludge

Congener	Concentration (ng/g, dry)		
	Run 2	Run 3	Run 4
Dioxins			
2,3,7,8-TCDD	(0.003) #	(0.003) #	(0.005) #
Total TCDD	0.068	0.083	0.095
1,2,3,7,8-PCDD	ND <0.015	ND <0.017	ND<0.017
Total PCDD	0.030	0.023	0.030
1,2,3,4,7,8-HxCDD	ND<0.005	ND<0.005	0.008
1,2,3,6,7,8-HxCDD	0.015	0.018	0.031
1,2,3,7,8,9-HxCDD	0.027	0.024	0.040
Total HxCDD	0.128	0.135	0.020
1,2,3,4,6,7,8-HpCDD	0.229	0.281	0.384
Total HpCDD	0.431	0.520	0.702
Octa CDD	2.51	2.69	3.69
Total CDD	3.17	3.45	4.53
Furans			
2,3,7,8-TCDF	0.021 #	0.024 #	0.035 #
Total TCDF	0.076	0.096	0.120
1,2,3,7,8-PCDF	ND<0.008	ND<0.008	0.013
2,3,4,7,8-PCDF	0.008	0.009	0.013
Total PCDF	0.030	0.095	0.163
1,2,3,4,7,8-HxCDF	0.014	0.019	0.030
1,2,3,6,7,8-HxCDF	ND<0.005	0.006	0.010
2,3,4,6,7,8-HxCDF	0.006	0.008	0.013
1,2,3,7,8,9-HxCDF	ND<0.006	ND<0.007	ND<0.007
Total HxCDF	0.098	0.117	0.171
1,2,3,4,6,7,8-HpCDF	0.132	0.159	0.222
1,2,3,4,7,8,9-HpCDF	ND<0.012	ND<0.013	ND<0.013
Total HpCDF	0.239	0.284	0.377
Octa CDF	0.313	0.340	0.441
Total CDF	0.756	0.932	1.27
Total CDD + CDF	3.92	4.38	5.80

Note: (Below Detection Limit) values listed in parentheses.
ND = Non Detect, value is detection limit.
# = Value from second column confirmation.
Non Detects and (Below Detection Limit) values not included in totals.

2.5.3 Scrubber Water and Sewage Sludge Inorganic Results

Table 2-19 provides analytical results for inorganic parameters (chlorine, total percent solids, temperature, and pH) in scrubber water samples. No significant differences are apparent between inlet and outlet, or across the three runs, for these parameters in the scrubber water. Chlorine and percent solids results for sewage sludge samples are presented in Table 2-20. Again, results across the three runs appear generally comparable. However, in several instances the free chlorine values exceed the reported total chlorine values. The explanation for this is indeterminent.

Ultimate/proximate analysis was also performed on the sludge feed samples to determine thermal properties. Results from the ultimate/proximate analysis are presented in Tables 2-21 and 2-22. Laboratory data sheets for this analysis are provided in Appendix J. As shown, ultimate/proximate results are consistent for all three runs.

Table 2-19. Chlorine, Percent Solids, Temperature, and pH Results - Comparison of Inlet Versus Outlet Scrubber Water

Measurement	Run 2		Run 3		Run 4	
	In	Out	In	Out	In	Out
Chlorine (mg/L)						
- Free	0.01	0.03	0.05	0.05	0.03	0.01
- Total	ND<0.01	0.05	0.08	0.03	0.05	0.02
Total Percent Solids (%)	0.146	0.154	0.129	0.142	0.146	0.158
Temperature (°F[a])	87	116	87	120	87	123
pH[a]	7.37	6.49	7.48	6.57	7.43	6.51

[a] Temperature and pH were calculated as an average of six grab samples collected during each of the 360 minute sampling runs.

Note: ND = Non Detect, value is detection limit.

Table 2-20. Chlorine and Percent Solids Results for Sewage Sludge

Measurement	Run 2	Run 3	Run 4
Chlorine (mg/kg)			
- Free	18.5	4.84	17.1
- Total	ND<0.5	4.84	4.29
Total Percent Solids (%)	23.5	20.0	20.4

Note: ND = Not detected, value is detection limit.

Table 2-21. Ultimate Analysis Results for Sewage Sludge

Ultimate Measurement	Dry Basis, %		
	Run 2	Run 3	Run 4
Hydrogen	4.95	4.93	5.16
Nitrogen	4.76	4.51	4.91
Oxygen	12.5	15.1	13.5
Carbon	38.2	37.0	39.3
Sulphur	1.33	1.33	1.35
Ash	38.3	37.1	35.7

Table 2-22. Proximate Analysis Results for Sewage Sludge

Proximate Measurement	Run 2		Run 3		Run 4	
	As Received	Dry Basis	As Received	Dry Basis	As Received	Dry Basis
Moisture, %	76.2	--	79.1	–	78.3	--
Volatile Matter, %	13.9	58.3	12.2	58.7	13.0	60.0
Fixed Carbon, %	0.80	3.39	0.89	4.26	0.93	4.29
Sulphur, %	0.32	1.33	0.28	1.33	0.29	1.35
BTU Content (BTU/lb)	1630	6850	1480	7100	1590	7300

3.0 SAMPLING LOCATION DESCRIPTIONS

Sampling locations for flue gas emissions, scrubber water, and sewage sludge feed are indicated on the process flow diagram provided in Figure 3-1.

3.1 FLUE GAS SAMPLING LOCATION

Flue gas sampling was conducted in the exhaust stack following the venturi scrubber and conditioning tower scrubber to control emissions from Incinerator No. 6. Figure 3-2 is a schematic of the sampling location. As shown in Figure 3-2, the incinerator exhausts from the scrubber through a 40-in. i.d. stack extending from the roof of the incinerator building. The stack is constructed of 3/8-in. steel. Four ports, 3 inches in diameter and 5 inches deep are located approximately 13 ft, 6 in. above the roof line. The sampling ports are located approximately 204 in. (5.1 duct diameters) downstream of the nearest flow disturbance (center shaft cooling air duct). The stack exhaust end is located approximately 216 in. (5.4 duct diameters) downstream of the sampling ports.

Sampling location flue gas characteristics were determined using individual sampling and traverse points for measurement of flue gas velocity, flue gas composition, and the matrix parameters listed in Table 1-1. Testing was conducted according to EPA standard methods as described in the following paragraphs.

3.1.1 Sampling Point Determination - EPA Method 1

In accordance with EPA Method 1, a total of 24 traverse points (twelve per axis) were used for MM5 sampling and CEM measurements. To minimize handling of the MM5 sampling train during the sample collection, data were recorded at 5 minute intervals, however, the collection train was only move every 15 minutes (180 minutes per each traverse). Sample collection consisted of 12 sampling points in each of two sampling ports (port A and port B). A diagram of the sampling points is provided in Figure 3-3.

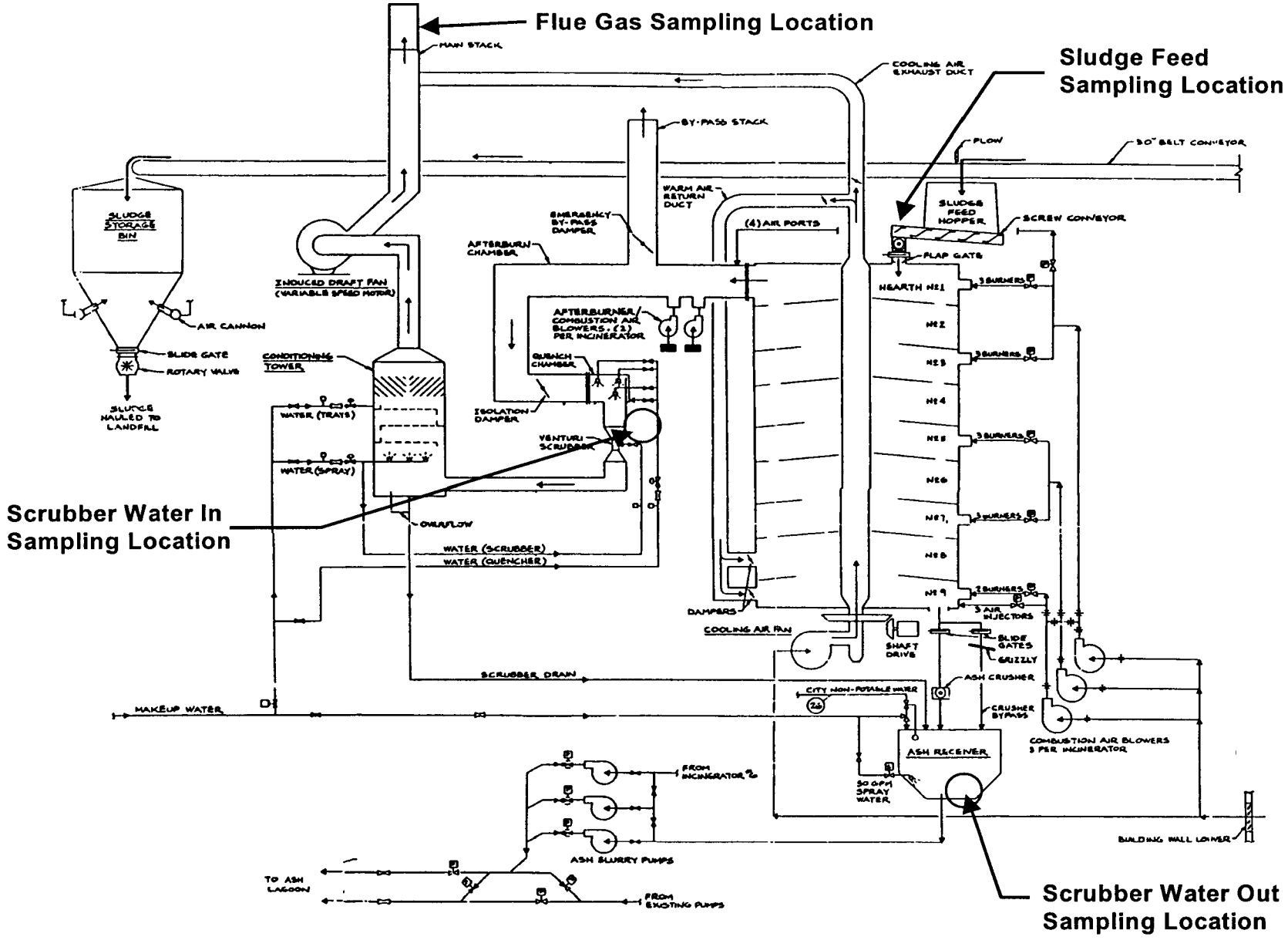


Figure 3-1. Plant Process Sampling Locations

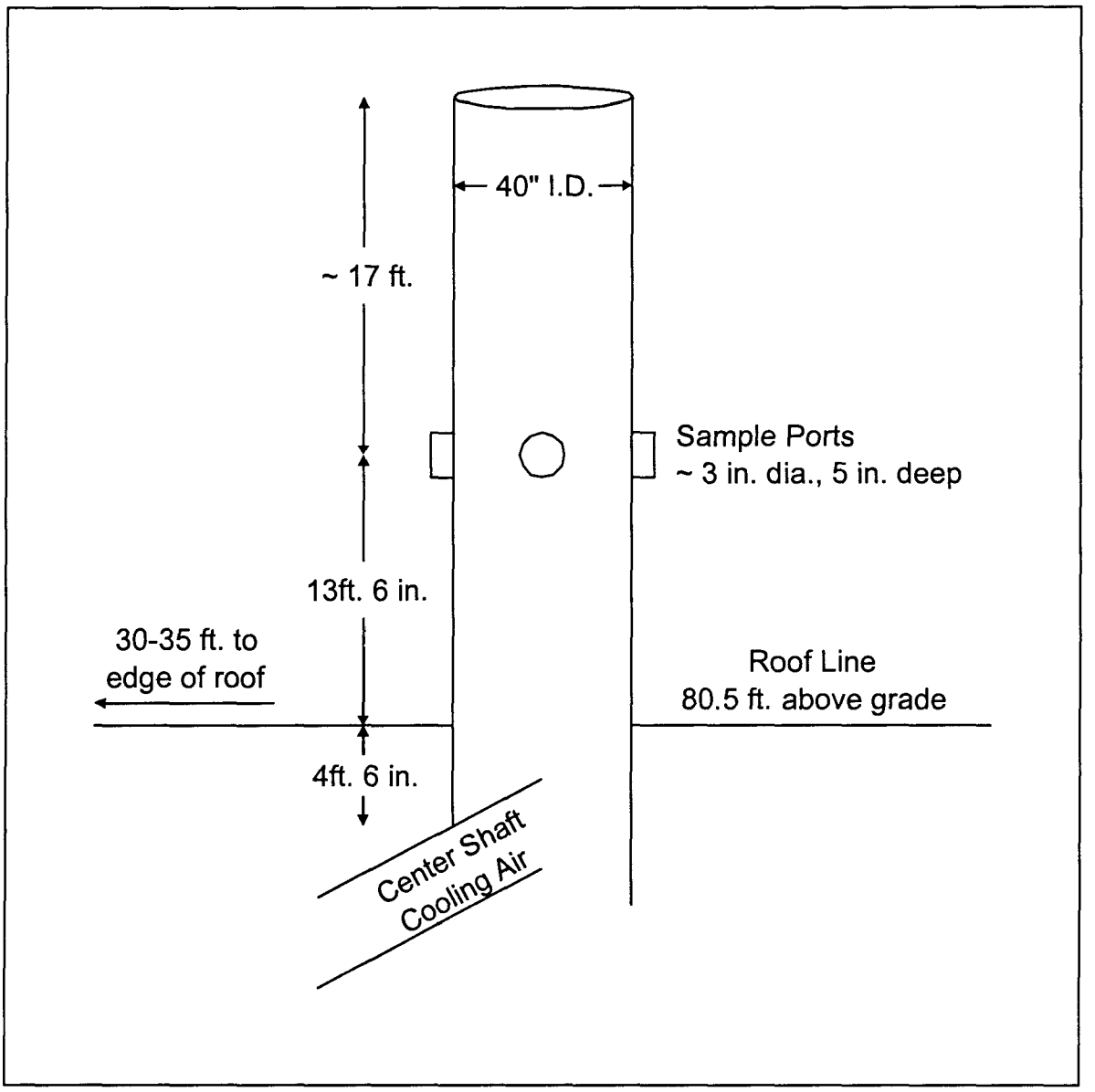
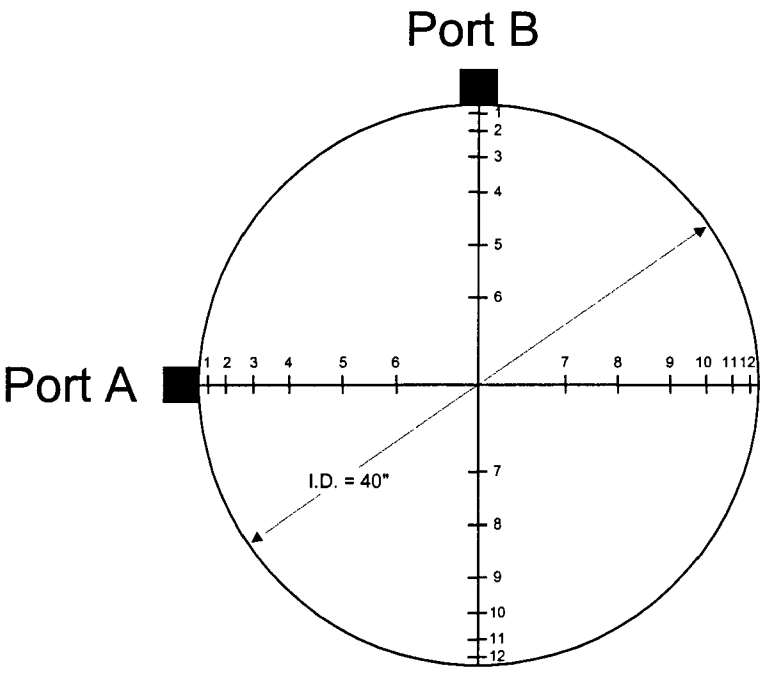


Figure 3-2. Schematic of Sampling Location Exhaust Stack for Incinerator No. 6



Point	% of ID	Distance from Inside of Port (inches)
1	2.1	0.8 (1.0)[a]
2	6.7	2.7
3	11.8	4.7
4	17.7	7.1
5	25	10
6	35.6	14.2
7	64.4	25.8
8	75	30
9	82.3	32.9
10	88.2	35.3
11	93.3	37.3
12	97.9	39.2 (39)[a]

[a] Points 1 and 12 were adjusted to one inch from the stack wall to comply with EPA Method 1

Inside Diameter	40 in.	3.3 ft.
Distance Port Downstream from Disturbance	204 in.	5.1 dia.
Distance Upstream from Port Disturbance	216 in.	5.4 dia.

Figure 3-3. Sampling and Traverse Points for Incinerator Stack

3.1.2 Volumetric Measurements - EPA Method 2

EPA Method 2 was used to determine the velocity and volumetric flow rates of the stack gas. The gas velocity was measured using a stainless steel Type-S pitot tube. The pitot tube was calibrated against a NIST-traceable pitot tube in accordance with Method 2. Prior to the field test a type-K thermocouple was calibrated against know gas temperatures.

Velocity and temperature measurements were made at each of the 24 traverse points as shown in Figure 3-3. These measurements were performed in conjunction with the MM5 sampling. Measurement data were documented on field data sheets provided in Appendix B to this report.

3.1.3 Molecular Weight Determination - EPA Method 3A

O_2 and CO_2 gas measurements for determining the average molecular weight of the stack gases were performed in accordance with EPA Method 3A. Sampling was conducted by obtaining integrated gas samples as part of the continuous instrumental analyzer methods discussed in Section 5.1.1.6. Sampling for carbon monoxide, oxygen, and carbon dioxide were originally to be conducted at a single point in the centroid area of the duct. However, a gas stratification determination performed on July 19, 1999, was inconclusive. The gas stratification was not resolved since a single CEM system was used which did not allow for comparisons between two CEM systems for variability. Since gas stratification was a possibility, the WAM decided to traverse the CEM sampling probe using the same traverse points simultanous with the MM5 sampling train.

3.1.4 Flue Gas Moisture Content - EPA Method 4

The flue gas moisture was measured in conjunction with each of the MM5 sampling runs according to procedures outlined in EPA Method 4. The flue gas moisture for each run was determined by volumetric analysis of the water collected in the XAD absorbent and impingers of the MM5 sampling train. All impingers were contained in an ice bath throughout the testing to

ensure complete condensation of the moisture in the flue gas stream. Any moisture not condensed in the impingers was captured in the silica gel contained in the final impinger.

3.2 PROCESS SAMPLING LOCATIONS

3.2.1 Scrubber Water

Scrubber water samples were collected at two locations: (1) inlet sample - the main header supplying the venturi and three-tray impingement scrubber on level 1 of the incinerator building, and (2) outlet sample - from the bottom of the ash tank located at the bottom of the venturi scrubber. Both sources were located in the center of the far north side of the incinerator building.

3.2.1.1 Inlet Sampling Location

Inlet water samples were collected from a 6-in. nonpotable water supply pipe used to feed the venturi scrubber. Water pressure in the pipe was at 10 psig. A 0.5-in. pipe valve located on the side of the feed pipe was used to drain the sample. This sampling site was located on level 1 of the incinerator building. Prior to collecting a sample the valve would be opened and allowed to purge for 3 minutes. The sample collection container would then be filled twice and then dumped. The third filling was retained as a valid grab sample.

3.2.1.2 Outlet Sampling Location

Scrubber water from the venturi scrubber was collected from a drain valve located at the bottom of the ash collection tank. The tank was located in the basement of the incinerator building just below the impinger trays. The drain valve was left partially open to allow a constant flow of water from the drain. This was done to prevent plugging. When collecting a sample, the sample container would be filled twice and dumped. A third filling would then be retained as a valid grab sample.

3.2.2 Sludge Feed

Sludge feed samples were collected from the screw conveyor transferring the sludge from the sludge feed hopper to Hearth No. 1 of the incinerator. Samples were taken from an open end of the screw conveyor.

4.0 PROCESS DESCRIPTION AND OPERATION

4.1 PROCESS DESCRIPTION

4.1.1 General

The Mill Creek Wastewater Treatment Plant is a municipal waste water treatment plant that incinerates sewage sludge in six multiple hearth incinerators. The facility can treat from 120 to 180 million gallons per day (MGD) of wastewater. It currently processes about 140 MGD. The Authority serves a population of approximately 750,000. Residential and light commercial sources contribute most of the wastewater received at the treatment plant. Approximately 20 to 25 percent by volume (containing 40 percent of the received solids) comes from industrial sources. Figure 4-1 is a schematic diagram of the process.

4.1.2 Preliminary Treatment

Wastewater entering the plant first passes through a screening process to remove any large objects. It then moves through grit tanks that allow the heavy, untreatable inorganic materials, called grit, to settle from the wastewater. Next, the wastewater flows through fine screens that remove small, fibrous material. The Mill Creek WWTP sends all solid materials collected during the preliminary treatment steps to a landfill.

4.1.3 Primary Treatment

Wastewater then flows to the primary clarifiers. Here, these clarifiers hold the wastewater under quiescent conditions to allow the heavy solids and floatable solids to separate from the wastewater. Afterwards, the wastewater goes to the secondary treatment process and the primary sludge goes to the sludge thickening system. The skimmings go to special skimmings handling facilities.

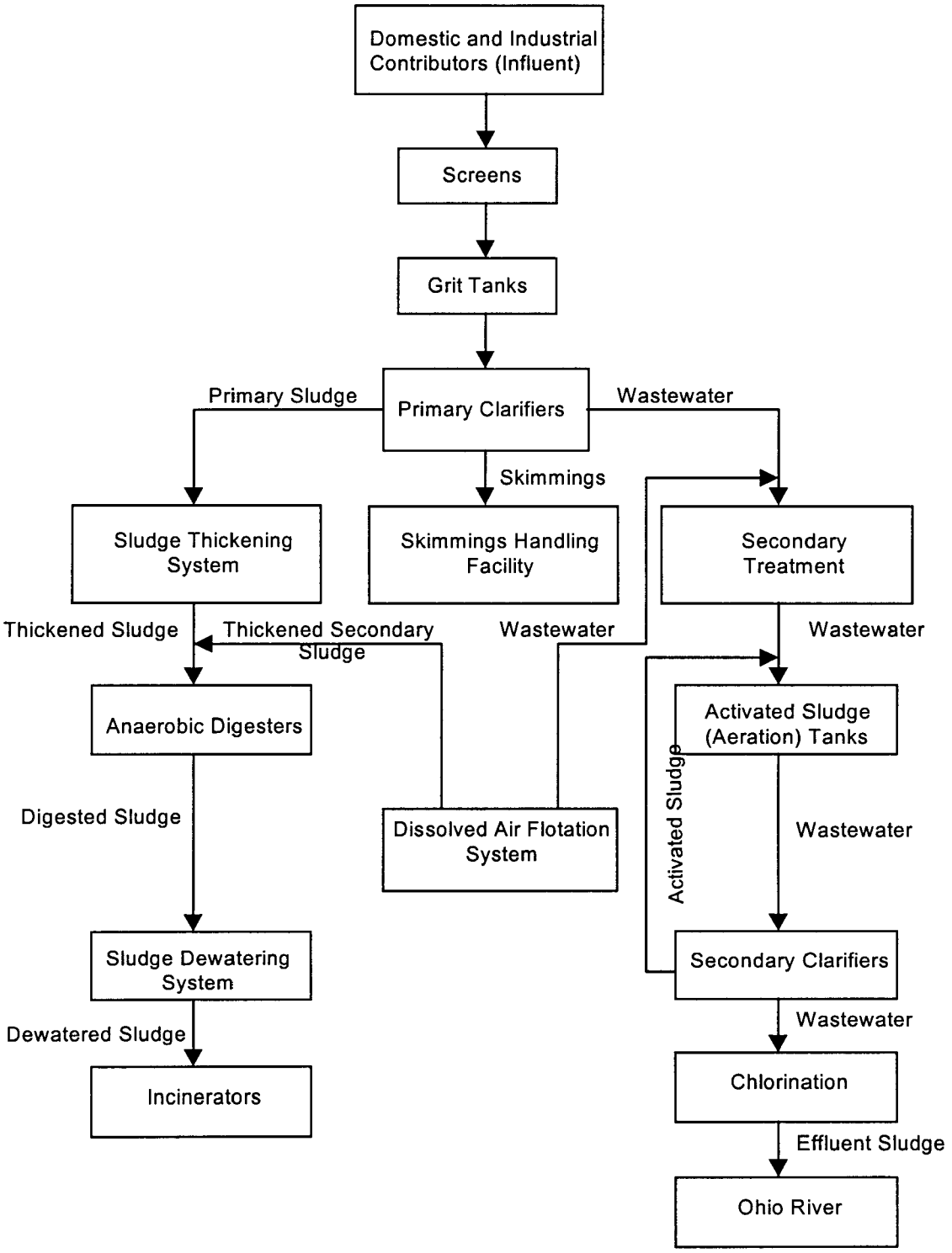


Figure 4-1. Schematic Diagram of Mill Creek Wastewater Treatment Plant Process

4.1.4 Secondary Treatment

Wastewater entering the secondary treatment process contains colloidal particles and soluble organic material. This wastewater flows into the activated sludge tanks. These tanks contain a large population of microscopic organisms that digest the organic material using it for energy and incorporating it into new cellular material. The contents of the tank (wastewater, returned activated sludge and air) are maintained and mixed in the tank for 6 to 8 hours. This is sufficient time for the microorganisms to extract the colloidal particles and soluble organic material from the water. The discharge from the aeration tanks flows to the secondary clarifiers.

Most of the resulting secondary sludge (composed of microorganisms) returns to the head of the aeration tank to resume consumption of organic material from the wastewater. The aeration tanks produce more activated sludge than the organic material in the incoming wastewater can support. Pumps send the excess activated sludge (EAS) to the sewage sludge thickening facility.

4.1.5 Tertiary Treatment

Tertiary treatment consists of chlorination to approximately 3 ppm. Chlorination consumes approximately 3,000 pounds of chlorine per day. The treated wastewater is then discharged into the Ohio River.

4.1.6 Sewage Sludge Thickening System

The primary sewage sludge that enters the sludge thickening facility is approximately 0.5 to 1 percent solids. Circular basins that allow settling under quiescent conditions thicken the sewage sludge to a solids content to approximately 8 percent. The thickened sludge flows to the anaerobic digesters.

A dissolved air flotation (DAF) system thickens the secondary sewage sludge. The DAF combines the excess activated sludge with a pressurized, supersaturated solution of air in water. Release of the pressure allows the excess air to leave the solution. This air forms small bubbles

that attach to particles of sludge, causing them to float to the surface. Pumps remove the sludge blanket and pump the sludge, now about 6 percent solids, to the anaerobic digesters.

4.1.7 Sewage Sludge Digestion

Anaerobic digestion, as the name infers, allows anaerobic bacteria to digest the mixture of primary and secondary sludges in air-tight tanks. The digestion process includes two heaters. The first heater raises the temperature of the material entering the digesters to about 95 °F; the second maintains the temperature throughout the 30- to 40-day digestion period. Higher temperature enhances the rate of consumption of the sewage sludge solids by the anaerobes. The anaerobes partially reduce the organic matter in the sewage sludge to carbon dioxide, water and methane.

The resulting digester gas has a high enough heat content to fuel the digestion process heaters and the sewage sludge incinerators. Flares burn the excess digester gas during nonoperational periods. The digested sludge flows to the dewatering system. The facility plans to stop the digested sewage sludge burning process in the future, but was burning digested sewage sludge during these tests.

4.1.8 Dewatering System

The dewatering system consists of 16 belt filter presses. These increase the solids content of the sludge to approximately 22-26 percent solids. Mill Creek has begun replacing the belt filter presses with centrifuges. The centrifuges will increase the solids content of the dewatered sewage sludge to 27-29 percent. Belt filter presses provided all of the dewatered sewage sludge burned during these tests. Dewatered sludge travels to the incineration facility via conveyor belts.

4.1.9 Incinerator Ash

Incinerator ash is slurried and pumped into ash lagoons for storage. The operators periodically dredge the ash from the lagoons and send it to a landfill.

4.2 DESCRIPTION OF INCINERATORS

Mill Creek has six multiple hearth incinerators. Four of the units were installed in 1959. Mill Creek rehabilitated the four original incinerators in 1973 and again in 1992. The plant added two additional incinerators in 1990. The incinerators operate 24-hours a day. Design capacity is 18,000 wet pounds per hour of sludge for each incinerator. Permitted capacity is 303 dry tons of sewage sludge per day. Normal operating capacity is 14,000 to 16,000 wet pounds (at 22 percent solids) per hour per incinerator. The plant usually operates three incinerators at a time, incinerating a total of approximately 110 dry tons per day of sewage sludge.

Mill Creek began operation of incinerators 5 and 6 in 1990. Crouse Combustion Systems, Inc. designed the units and began construction. MMR/Wallace with Combustion Systems took over design and construction after the bankruptcy of Crouse. University Mechanical/Zack took over and completed the construction after the bankruptcy of the second design/construction team. The units have no single manufacturer or model numbers. They are 22-ft 3-in. outside-diameter, nine hearth multiple hearth incinerators.

A positive displacement, screw conveyor delivers sewage sludge to the incinerator. Plant operators have calibrated the delivery of the screw conveyors and calculate the rate of wet sludge burning based upon the revolutions per minute of the feed screw. This system provides an accurate measurement of the rate of delivery of sewage sludge. The system also provides a steady rate of delivery of fuel to the incinerator.

The incinerator can burn either natural gas or digester gas, though the operators rarely use digester gas.

4.3 DESCRIPTION OF EMISSIONS MONITORING, CONTROL AND EMERGENCY EQUIPMENT

4.3.1 Emissions Control Equipment

Incinerators 5 and 6 have variable throat Venturi contractors and three-tray impingement tray scrubbers with a stainless steel, chevron-style demister. Design pressure drop for the Venturi is 15 in. to 20 in. of water; design pressure drop for the tray scrubbers is 6 in. to 10 in. of water. The plant uses chlorinated final effluent as scrubber water. (Note: The Daily Reports contained in Appendix M list the pressure drop across the venturi portion of the scrubber system, not across the entire system.) The incinerator complies with its permit limit of 100 ppm THC (@ 7% O_2, Dry, 30-day average) without using the detached afterburner. The afterburner, when last used in 1996, operated at approximately 1050°F.

4.3.2 Emissions Monitoring Equipment

Continuous emission monitors record the stack-gas concentrations of O_2, and THC continuously. The concentration of THC in the stack gas must be expressed in parts per million with the concentration adjusted to 7 percent O_2 dry gas. The emission monitoring computer calculates the moisture concentration in the stack gas by assuming saturation and using a lookup table of percentage moisture vs. temperature. This conservative assumption will cause a slight overestimation of the calculated emission rate of THC. The assumption of saturation is conservative because the shaft cooling air, which is unsaturated, heated ambient air, is combined with the saturated scrubber exhaust upstream of the thermocouple stack temperature measurement location.

4.3.3 Emergency Equipment

Emergency bypass stacks protect the scrubber from overheating in case of a process upset. These stacks can route the incinerator exhaust gas directly to the atmosphere. The Programmable Logic Controllers (PLC) will shut down sludge and auxiliary fuel flows and open

the bypass dampers should any of the following occur: (1) disruption in building services (e.g., trouble with scrubber water levels, compressed air), (2) disruption in electric services, (3) disruption in communications between the PLC systems, (4) loss of an ID fan, and (5) excess temperature in the system. Each of these events has the potential to cause the incinerator to burn out of control and endanger the plant operators.

4.4 PROCESS OPERATION SUMMARY

At approximately 11:45 a.m. Wednesday, July 21, 1999, the upper hearths (2, 3, 4) of incinerator 6 overheated. This caused premature burning and generation of excess THC emissions. The instantaneous THC concentration hit a peak of 228 ppm. The operator reduced the rate of recycling of shaft cooling air to the lower hearths to remove some heat from the incinerator. This procedure reestablished control within a few minutes. This was the only process disruption of any sort during any of the test runs.

The incinerator operated without upset for the remainder of the test. In fact, the operation was stable for 24-hours per day for the entire test program period. An on-line terminal displayed the levels of the various parameters on a computer monitor for the benefit of the EPA and PES representatives. The terminal could plot trends for any previous time interval specified. The EPA/PES representatives observed that, with few exceptions, no temperatures or concentrations (THC, oxygen) deviated from their set points. All traces remained level. The operators maintained all parameters at constant, steady-state conditions.

During the test the venturi throat operated at approximately 22 inches of water pressure drop and 1,300 gallons per minute flow. Plant operators stated that the pressure drop across the impingement tray scrubber was an additional 6 inches of water.

Table 4-1 presents average values for specific parameters that were monitored during the three successful test runs and for the aborted run (Run Number 1). Appendix M contains Process Data Summary Tables and copies of MSD Daily Data Reports that Mill Creek provided for the four day emissions test program. These reports contain hourly averages of values for approximately 50 operating parameters. The EPA/PES representatives monitored portions of these data operations during the testing to ensure steady-state operation of the incinerator.

Table 4-1. Summary of Data from Mill Creek WWTP Control Room Process Monitors

Date	Run Number	Total Hydrocarbons, ppm		Stack Gas Oxygen, %	Stack Gas Moisture, %	Sewage Sludge Feed Rate, dry tons/hr
		Uncorrected	Corrected, 7% O_2, dry			
July 20, 1999	1*	34.8	57.9	12,1	3.1	1.7
July 20, 1999	2	41.0	70.6	12.5	3.1	1.7
July 20, 1999	Entire Day	35.4	60.6	12.5	3.1	1.7
July 21, 1999	3	31.8	54.2	12.3	3.2	1.4
July 21, 1999	Entire Day	32.5	55.3	12.3	3.1	1.4
July 22, 1999	4	21.5	37.5	12.6	3.3	1.53
July 22, 1999	Entire Day	23.1	40.1	12.5	3.3	1.54

* Run 1 aborted for failure to pass mid-test leak check.

5.0 SAMPLING AND ANALYTICAL PROCEDURES

5.1 AIR EMISSIONS

5.1.1 Air Emission Sampling

All emission sampling followed U.S. EPA published methods located in Title 40, Part 60, Appendix A of the *Code of Federal Regulations* (40 CFR 60); *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (EPA SW-846); or other methods approved for this project by the U.S. EPA. The following specific methods were used:

- EPA Method 1 for determining sampling and traverse points;
- EPA Method 2 for determining flue gas velocity and volumetric flow rate;
- EPA Method 3A for determining flue gas composition and molecular weight (including measurements of flue gas oxygen and carbon dioxide content);
- EPA Method 4 for determining flue gas moisture content;
- EPA Method 10 for determining carbon monoxide emissions;
- EPA Modified Method 5 for determination of PCBs, D/Fs, and PAHs; and
- EPA Method 25A for total hydrocarbon (THC) emissions.

5.1.1.1 Modified Method 5 Sampling

Emission sampling for determination of PCBs, D/Fs, and PAHs was conducted in general accordance with EPA Modified Method 5 (MM5) procedures.

Sampling Train Description. Figure 5-1 illustrates the MM5 sampling train. The train employs a borosilicate glass nozzle and probe for sample withdrawal. The nozzle opening is appropriately sized to maintain isokinetic sampling. Particulate matter and semi-volatile organic compounds are removed from the gas stream and passed through a heated glass filter supported on a Teflon frit.

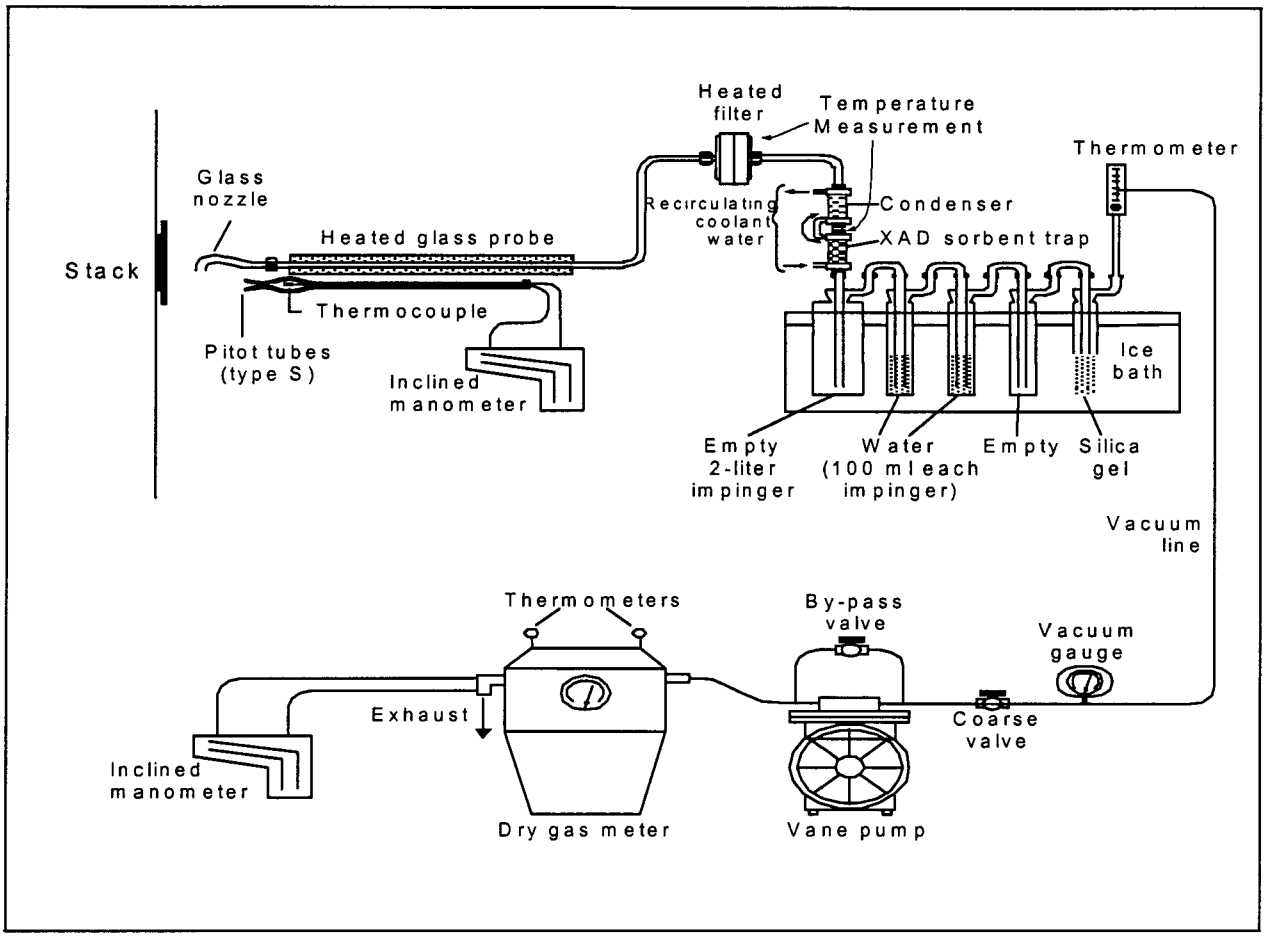


Figure 5-1. Sampling Train for EPA Modified Method 5

After particulate removal, the sample gas passes into a water-cooled glass condenser, then into an XAD-2 sorbent trap. The sorbent trap is packed with precleaned, quality control checked amberlite XAD-2 resin. Coolant water maintained at wet-ice temperature is continuously recirculated through the assembly using a submersible water pump. The condenser cools the sample gases and condenses the stack moisture. The cooled gases and condensate flow down through the XAD-2 resin, which retains the organic compounds.

After passing through the sorbent trap, the sample gases enter a chilled impinger train to remove any remaining moisture. The impinger train consists of five glass impingers packed in ice water. The first modified (short stem) impinger is left empty to facilitate collection of the majority of condensate that passes through the XAD-2 sorbent trap. The second and third impingers each contain 100 mL of high performance liquid chromatography (HPLC) grade water. The fourth impinger is left empty, and the fifth impinger contains approximately 300 g of 6-16 mesh indicating silica gel. Temperature measurements were taken at the probe, filter holder, inlet to XAD-2 sorbent trap, outlet of the silica gel impinger, and inlet and outlet of the dry gas meter.

All components from the nozzle to the fourth impinger were made of glass; excluding the Teflon probe union and filter frit. All connections from the probe to the exit stem of the fourth impinger were sealed with Teflon O-rings. Sealing grease was not used on any connections before the fifth impinger.

Sampling Train Preparation. All glass and Teflon components of the MM5 sampling train were precleaned before use. The following cleaning procedures were used:

(1) Verify that there is no grease on any of the fittings
(2) Wash with hot water and detergent
(3) Rinse with tap water three times
(4) Rinse with deionized, distilled water three times
(5) Rinse with pesticide-grade hexane three times
(6) Rinse with pesticide-grade toluene three times
(7) Rinse with pesticide-grade acetone three times
(8) Bake at 400° F for three hours*
(9) Cap glassware with hexane-rinsed aluminum foil, wrap externally with Teflon tape.

*Note: Probe liner was brushed and rinsed as specified and allowed to air dry.

After cleaning, all glassware was sealed using Teflon tape or hexane-rinsed aluminum foil, and placed in protective containers for transport to the test site.

Precleaned XAD-2 resin was purchased from Supelco. The XAD-2 resin was purified again prior to use in the field. The XAD-2 resin was extracted with methylene chloride for at least 24 hours using Soxhlet apparatus. After extraction, the XAD-2 resin was transferred to a clean Pyrex drying column. The drying column has sufficient space for fluidizing the XAD-2

bed, while generating a minimum resin load at the exit of the column. The resin was dried by passing high-purity nitrogen through the bed. The nitrogen was purified by passing it through a charcoal trap between the nitrogen cylinder and the column. The rate of nitrogen flow through the column was set to agitate the bed gently to remove the residual solvents. After drying, the XAD-2 resin was transferred to a clean jar ready for packing into sorbent traps.

The glass fiber filters were Soxhlet extracted with toluene, followed by extraction with methylene chloride and dried under nitrogen. The clean filters were placed individually in clean glass petri dishes. The clean filters were then labeled and wrapped with precleaned aluminum foil, then in bubble wrap, for shipment to the sampling site.

Prior to packing the XAD-2 resin into the sorbent traps, 40 g of XAD-2 resin and one filter were extracted and analyzed together to determine background levels of PCBs, D/Fs, and PAHs. The PCB, D/F, and PAH concentrations in this background check sample were below the maximum acceptable levels listed in the QAPP. Analytical results from the PCB, D/F, and PAH background check are provided in Appendices E, F, and G, respectively.

The sorbent traps were cleaned by detergent water and rinsed with tap water, distilled water, and methanol. The clean sorbent traps were then placed in an oven at 450°C for 12 hours to remove any trace amounts of organic contaminants. Each clean sorbent trap was packed with approximately 40 g of clean XAD-2 resin. Each packed XAD-2 sorbent trap was spiked with the pre-field surrogate standards listed in Table 5-1. After spiking, both ends of the sorbent trap were sealed with pre-cleaned aluminum foil and Teflon tape. The packed XAD-2 sorbent traps were labeled and wrapped with pre-cleaned aluminum foil, then bubble wrap, and placed in coolers with frozen blue ice for shipment to the sampling site. Once the XAD-traps were spiked with the pre-field surrogate solutions, they were maintained at ≤4°C.

Once in the field and prior to each test run, the MM5 sampling train was assembled and leaked-checked in a clean room at the test site. The sampling train was then separated into at least four sections for transport to the test platform: 1) the probe; 2) the filter and condenser; 3) sorbent trap assembly; and 4) the impinger train. To prevent contamination, the ends of each section was sealed with hexane-rinsed aluminum foil or Teflon tape. At the test platform, the train was reassembled and leak-checked prior to sampling.

Table 5-1. Pre-Field Surrogate Standards

Standard Type	Standard Contents	Spike Amount	Comments
D/F	^{37}Cl4-2,3,7,8-TCDD	8000 pg	XAD samples
	^{13}C12-2,3,4,7,8-PeCDF	8000 pg	
	^{13}C12-1,2,3,4,7,8-HxCDF	8000 pg	Purchased as a mixture (Cambridge Isotope Laboratories EDF-4054)
	^{13}C12-1,2,3,4,7,8-HxCDD	8000 pg	Purchased as a mixture (Cambridge Isotope Laboratories EDF-4054)
	^{13}C12-1,2,3,4,7,8,9-HpCDF	8000 pg	Purchased as a mixture (Cambridge Isotope Laboratories EDF-4054)
PCB	^{13}C12-PCB-81	2000 pg	XAD samples
	^{13}C12-PCB-111	10,000 pg	Purchased as a mixture (Cambridge Isotope Laboratories EC-4062)
PAH	^{13}C-Fluorene	200,000 pg	XAD samples

Preliminary Measurements. Prior to the test program, preliminary measurements were made to facilitate isokinetic sampling. Preliminary measurements included a traverse of the 24 sampling points discussed in Section 3.1.1.1, measuring velocity, temperature, and the cyclonic flow angle at each sampling point. Preliminary measurements of flue gas moisture and flue gas static pressure were conducted at a single point in the gas stream. These measurements were used to determine the nozzle size and K factor used in operation of the MM5 sampling train.

Additionally, independent measurements of stack diameter, port depth, and distances to nearest upstream and downstream disturbances were verified to confirm compliance with Method 1 specifications and to ensure the accuracy of the dimensions supplied in Figure 3-1.

Sampling Train Operation. Sampling was performed in general accordance with Method 5 procedures and specifications, including leak checking, isokinetic sampling rate, and stack traversing.

Prior to each sampling run, the train was assembled at the sampling platform, and the probe heater, filter heating element, and condenser coil recirculating pump switched on. Once the probe and filter housing temperature reached 248 ± 25°F, the train was leak checked. The pre-test leak check was acceptable if a leak rate of less than 0.02 ft^3/min was observed at a vacuum of 15 in. Hg.

Additional leak checks were conducted during each port change and at the completion of each test run. These leak checks were considered acceptable if a leak rate of less than 0.02 ft^3/min was observed at the highest vacuum recorded during the test run. At the port change of the first sampling run, the leak check was deemed to be unacceptable. The WAM was notified, and after discussion, it was determined that the sampling run should be invalidated. Sampling was discontinued, the sampling train was recovered using the stated procedures in the SSTP and the sample archived. Three additional sampling runs were performed, so that three valid sampling runs were available for laboratory analyses.

Sampling was performed for 15 minutes at each of the 24 traverse points, yielding a 360-minute test per run. Pressure and temperature measurements and isokinetic sampling rate adjustments were made at 5-minute intervals. Sample volumes of 206.52, 219.66, and 256.60 dry standard cubic feet were obtained for the respective three test runs.

During sampling, the probe and filter temperatures were maintained at 248 ± 25°F. The temperature of the sorbent trap and the final impinger exit were maintained at or below 68° F. These temperatures were monitored and recorded at five-minute intervals during each sampling run.

Sample Recovery and Cleanup. At the completion of each test, the probe, filter assembly, condenser, and sorbent trap were removed from the train. All open ends of the sampling train components, including the impingers, were sealed with Teflon tape or hexane-rinsed aluminum foil and transported to a clean room for recovery. All wash bottles and funnels were composed of Teflon or glass because polyethylene or other plastics may contain organic contaminants. A flow chart illustrating the MM5 sample recovery procedures is shown in Figure 5-2.

Sample recovery proceeded as follows:

(1) **Filter (Container No. 1):** The filter was removed from the housing with Teflon tweezers and placed into a precleaned glass petri dish. The dish was sealed externally with Teflon wrapping tape.

(2) **Front-Half Rinse (Container Nos. 2 and 3):** The nozzle/probe and front half of the filter holder were rinsed three times with acetone and brushed between rinses using a Teflon-fiber probe brush. The acetone rinse was followed by three rinses with methylene chloride, that were recovered into the same sample container (Container No. 2). Following the methylene chloride rinses, the front-half glassware was rinsed three times with toluene. The toluene rinses were recovered separately (in Container No. 3). All rinses were recovered into precleaned amber glass bottles fitted with Teflon-lined screw caps. The liquid level in each bottle was marked on the bottle and then the cap wrapped with Teflon tape.

(3) **Filter to XAD Sorbent Trap (Container Nos. 4 and 5):** The back half of the filter holder and all connecting glassware from the filter to the sorbent trap were soaked/rinsed, and treated as in (2) above, with the exception that brushing was not done on the acetone rinse. The acetone and methylene chloride soak/rinses were recovered separately from the toluene soak/rinse and recovered into precleaned amber glass bottle with a Teflon-lined screw cap (Container No. 4). The toluene rinse was also recovered into a separate container (Container No. 5). All liquid levels were marked on the bottles and the caps wrapped in Teflon tape.

(4) **Sorbent Cartridge (Container No. 6):** The sorbent trap assembly was weighed to the nearest 0.5 g for determining moisture gain, capped with hexane-rinsed aluminum foil, and sealed externally with Teflon tape. The XAD-2 sorbent material was transported to the laboratory within the glass sorbent trap. The sorbent trap was wrapped with hexane-rinsed aluminum foil, and kept at $\leq 4°C$ to protect it from light and heat.

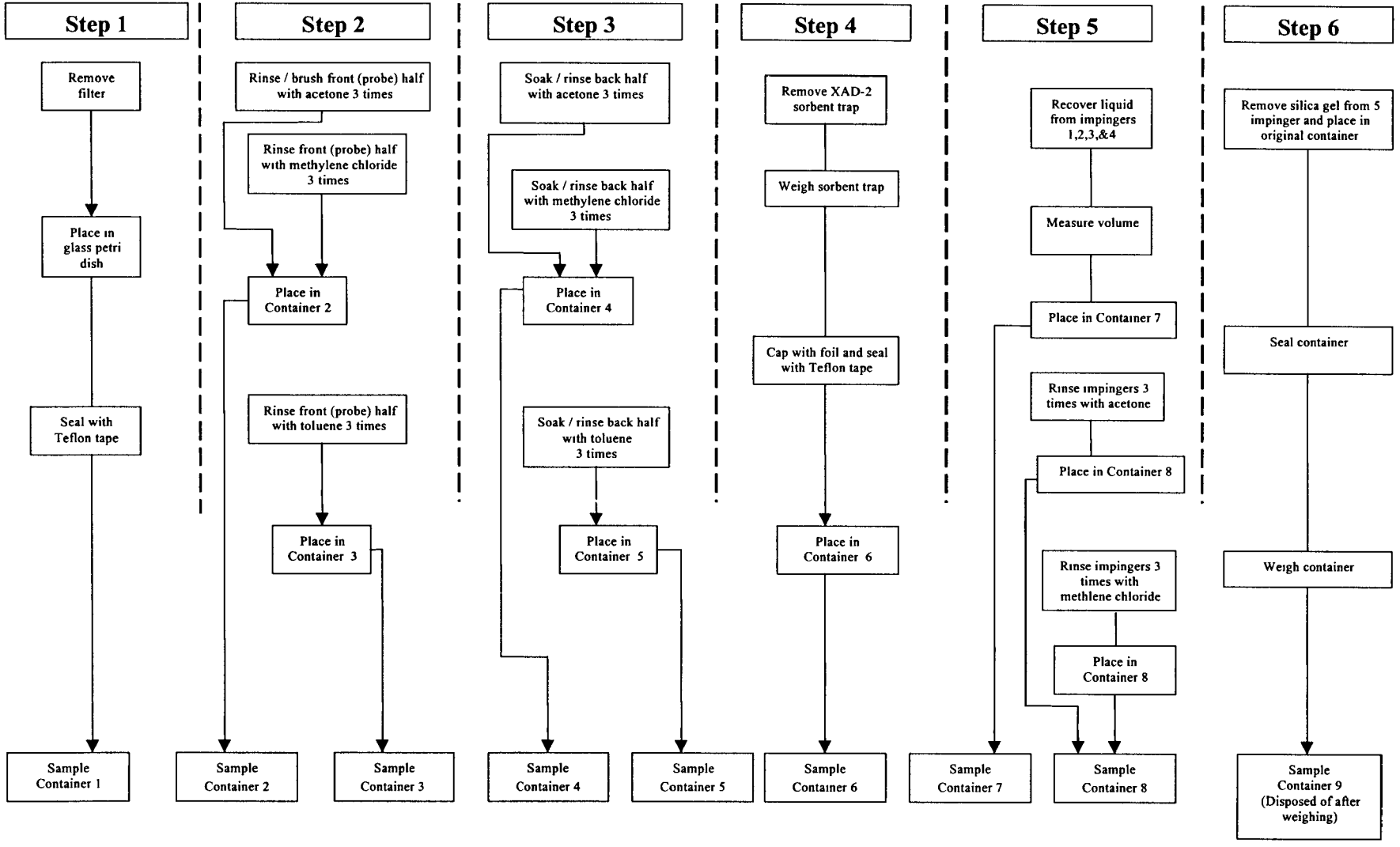


Figure 5-2. Flow Chart for Emission Sample Recovery

(5) **Impingers 1-4 Recovery (Container Nos. 7 and 8):** The condensate collected in impingers 1-4 was transferred to a graduated cylinder and the volume recorded. The liquid was then transferred to a precleaned amber glass bottle fitted with a Teflon-lined screw cap (Container No. 7). The impingers were then rinsed three times with acetone, followed by three rinses with methylene chloride. The rinses were collected into a separate bottle (Container No. 8). All liquid levels were marked on the bottles and the caps wrapped with Teflon tape.

(6) **Silica Gel (Container No. 9):** The silica gel in the fifth impinger was transferred into its original plastic container, sealed, and weighed to the nearest 0.5 g. The percent of silica gel spent, based on the color change, was documented before transferring.

Sample Storage and Transport. Immediately upon recovery, all samples including liquid rinses, filters, and sorbent traps were placed into insulated coolers and packed with frozen blue ice to protect them from light and heat. Battelle transferred all samples requiring off-site analysis at the end of each test day or the following morning. Emission samples were packed separately from sludge and water samples.

While in the custody of field personnel or on-site storage, the temperatures inside the coolers were measured every 8 to 12 hours to ensure that the samples did not exceed 4°C. The samples remained inside the coolers during transport to Battelle. Upon arrival at Battelle's Laboratories, the samples were refrigerated to maintain their temperatures at or below 4°C.

5.1.1.2 Continuous Emission Monitoring for CO, O_2, and CO_2

Instrumental monitoring of the stack gases was performed as follows:

Gas	Reference Method	Instrument Type	Range
CO	Method 10	TECO Model 48 NDIR CO Analyzer	0-6000 ppm_{dv}
O_2	Method 3A	Teledyne Model 320A Chemical Cell Portable O_2 Analyzer	0-25$\%_{dv}$
CO_2	Method 3A	HORIBA Model PIR-2000 NDIR CO_2 Analyzer	0-20$\%_{dv}$

Each of the above analyzers measure gas concentrations on a dry volume basis.

Sampling System Description. An integrated, remote instrumental system housing the CO analyzer, as well as the diluent gas (O_2 and CO_2) monitors, was used. Figure 5-3 outlines the general schematic of the system. The design incorporates a dry extractive system. All of the instruments were housed in a temperature-controlled trailer located at ground level.

The sampling system consisted of a heated stainless steel probe situated at the stack port location. A heated glass fiber filter was attached to the probe for particulate removal. A short section of heated Teflon sample line delivered the sample to an ice-cooled condenser designed to remove the flue gas moisture. The liquid and ice volumes in the condenser were checked every hour. The dry gas sample from the stack port location was transported through an unheated Teflon sample line via the instrumental system sample pump. The gas sample was then pumped through individual Teflon sample lines to the CO, CO_2, and O_2 monitors.

Data Acquisition System. The response outputs of the continuous monitors were recorded digitally by a Campbell Scientific Model CR10WP multi-channel data acquisition system. The system sampled at a rate of 60 Hz, and stored one-minute average values for each gas measured. At the end of each test a hardcopy of the data was printed.

Calibration. At the beginning of each test day, the O_2 and CO_2 monitors were zeroed, using zero nitrogen, and spanned, using EPA traceability protocol gases with a concentration of 80 to 100 percent of the instrument span. Following calibration, a mid-range gas (40 to 60 percent of the instrument span) was introduced to each monitor. The mid-range response error did not exceed two percent of the instrument span, as required by EPA Method 6C.

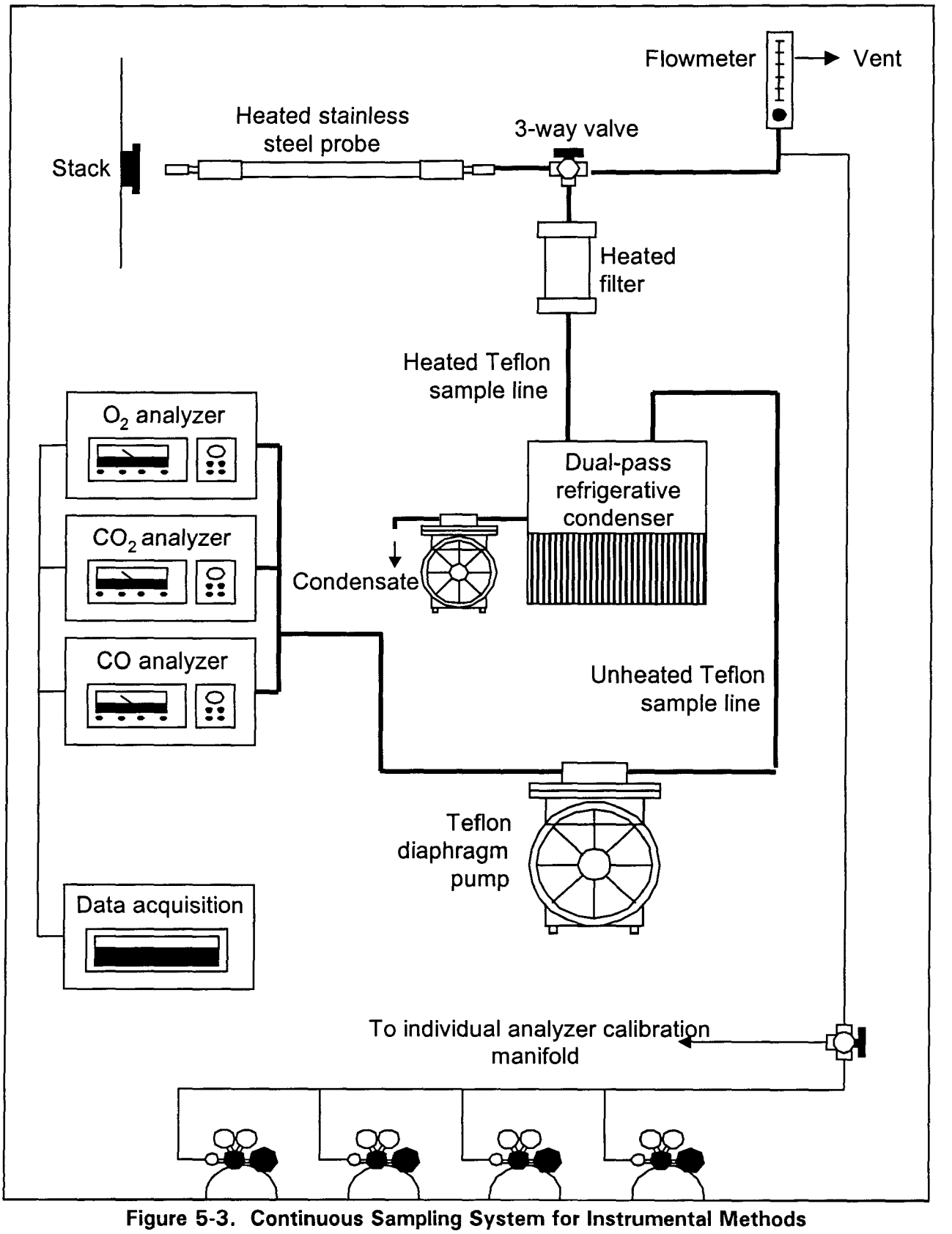


Figure 5-3. Continuous Sampling System for Instrumental Methods (EPA Methods 3A and 10)

The CO monitor was zeroed, using zero nitrogen, and spanned, using a known concentration of CO in nitrogen. Following calibration, the CO monitor was challenged with two additional gas concentrations corresponding to approximately 60 percent and 30 percent of instrument span.

After calibrating the CO, O_2, and CO_2 monitors, a Teflon calibration line was used to introduce calibration gas remotely through the probe to verify the absence of sampling system bias. The bias error did not exceed five percent of the instrument span, as required by EPA Method 6C.

After each test run, zero nitrogen and either a mid- or high-range calibration gas was introduced remotely through the sampling system to each monitor to check for calibration drift error. The calibration drift did not exceed the three percent of the instrument span for valid test runs required by Method 6C.

The following EPA traceability protocol calibration gases (Reference Standard) were used during the test program. Gas certification sheets and data are presented in Appendix L.

Instrument	Allowable Standard	Reference Standard
O_2	10-15% 20-25%	11.5% 21.0%
CO_2	8-12% 16-20%	11.0% 18.0%
CO	~900 ppm ~1800 ppm ~3000 ppm ~6000 ppm	915 ppm 1809 ppm 3000 ppm 5971 ppm
THC	60-90 ppm 150-180 ppm	86.6 ppm 124.6 ppm*
Zero (all instruments)	Nitrogen	

* High calibration gas used was accepted by WAM since analyzer range was low during the test program.

5.1.1.3 Total Hydrocarbon Monitoring

Total hydrocarbon (THC) emissions were monitored during the test program with a Horiba Model MPA-510 total hydrocarbon analyzer. The total hydrocarbon analyzer was operated by MSD staff. Data from the total hydrocarbon analyzer were collected by PES under a separate contract with the U.S. EPA. THC data contained in this report were provided by MSD.

MSD operated the THC analyzer consistent with procedures for operation of continuous emission monitors for continuing compliance demonstration. However, during routine operation, Method 25A data validation procedures were not observed.

To ensure the data validity, a two-point calibration gas audit (CGA) was performed on the hydrocarbon analyzer prior to and following the test program by ETS staff. The CGA was performed in accordance with the procedures of 40 CFR 60, Appendix F, Section 5.1.2. Results of this audit can be found in Section 6.2.1.5.

During the post-test calibration on July 22, 1999 moisture condensation was observed in the flow panel serving the analyzer. The post-test CGA revealed a slower response time and some degraded accuracy. However, the accuracy was still within the allowed 15 percent specification. Condensation within the system could slow the analyzer response time and absorb some of the water-soluble organics in the span gas audit as well as in the emissions being sampled. This moisture problem may account for the observed lower THC results on Run 4.

5.1.2 Air Emission Sample Analysis

5.1.2.1 MM5 Sample Extraction

MM5 emission samples collected in the field and associated quality control samples were extracted and each extract was split into three aliquots as shown in Figure 5-4 for front half extracts and Figure 5-5 for back half extracts. One aliquot (equivalent to 50 percent of the extract) was used for toxic PCB determination, a second aliquot (equivalent to 25 percent of the extract) for D/F analysis, and the final aliquot (equivalent to 25 percent of the extract) for PAH

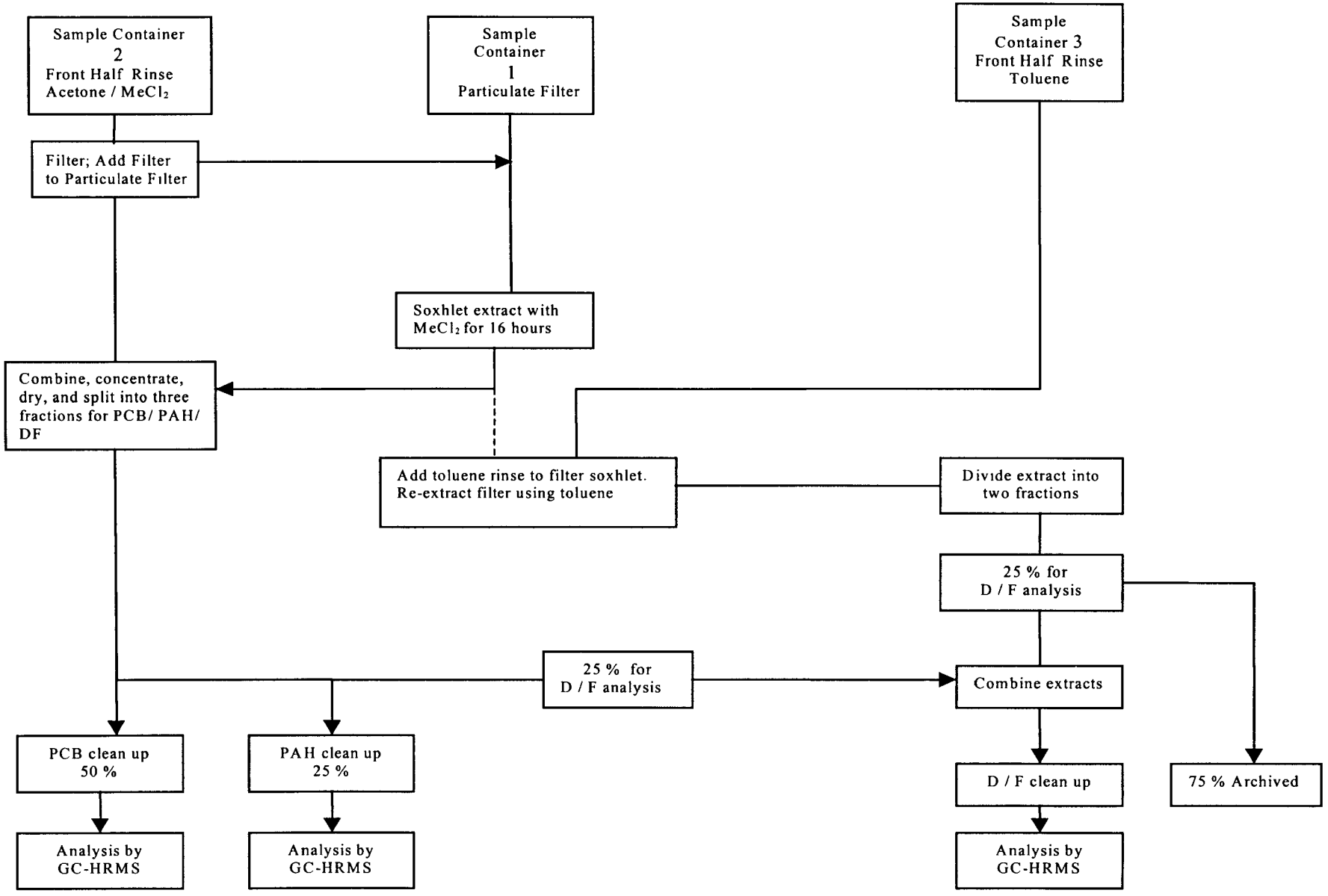


Figure 5-4. Flow Chart for Extraction of MM5 Sampling Train Front Half

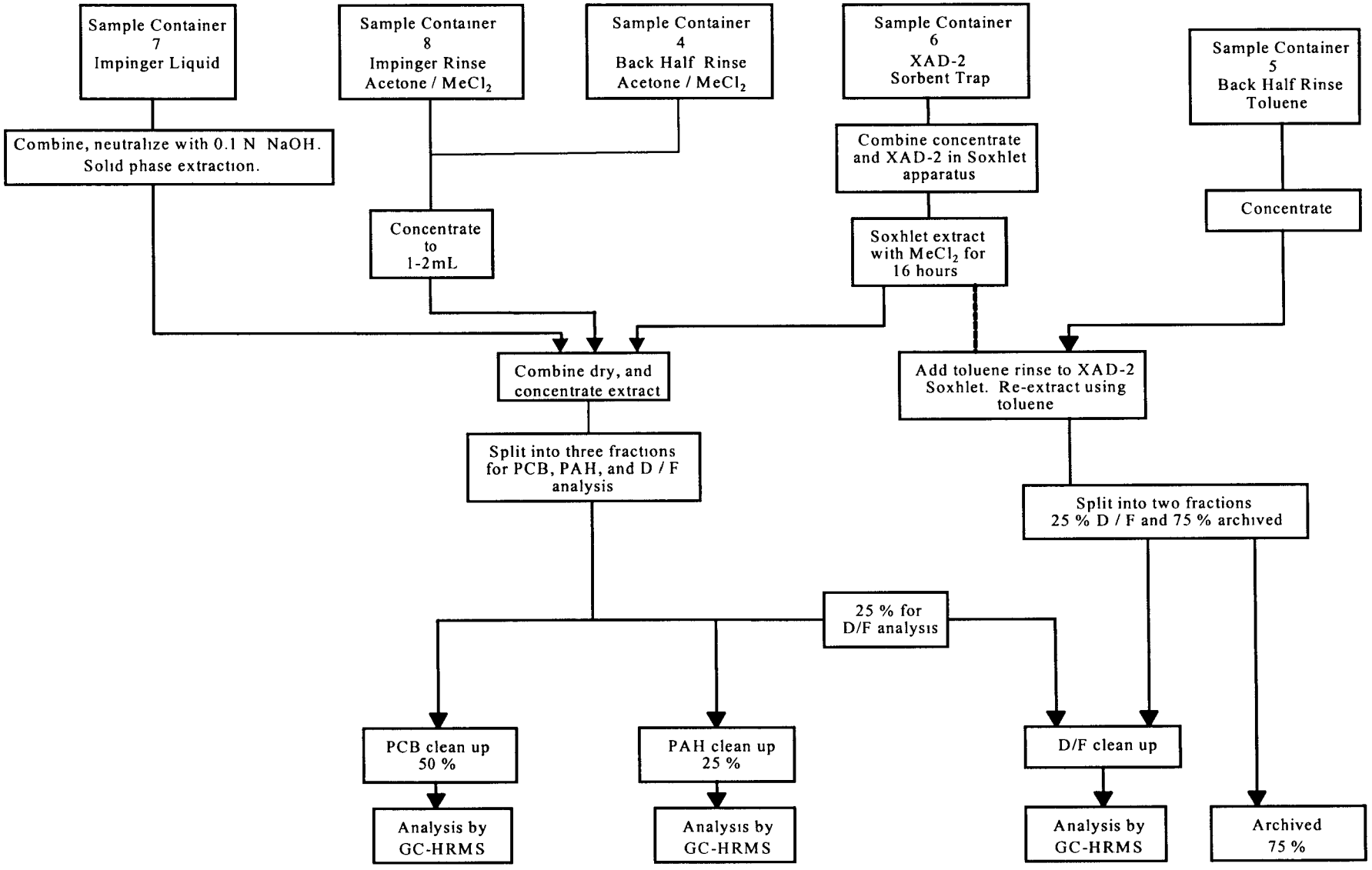


Figure 5-5. Flow Chart for Extraction of MM5 Sampling Train Back Half

5-15

specified in Table 5-2. Laboratory control spike samples were also spiked with native analytes at the levels specified in Table 5-3.

5.1.2.2 PCB Extract Cleanup and Analysis

Cleanup and PCB analysis of emission sample extracts were conducted following procedures given in the draft method, "Determination of Toxic Polychlorinated Biphenyls Emissions from Sewage Incinerator Stationary Sources Using Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry" (July 20, 1999, version). Any revisions to the draft method are detailed in the QAPP Amendment/Corrective Action Records which are included in Appendix P.

5.1.2.3 D/F Extract Cleanup and Analysis

Cleanup of emission sample extracts for D/F analysis were conducted according to EPA Method 8290 with revisions described in Battelle Standard Operating Procedures (SOP) Nos. SOP0802-01-01 and SOP0802-02-01. D/F analysis of emission samples was conducted according to EPA Method 8290 with revisions described in these two Battelle SOPs and modifications from EPA Method 23 "Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans from Stationary Sources" to accommodate the use of Method 23 D/F standards for calibration and spiking. A copy of Battelle SOP Nos. SOP0802-01-01 and SOP0802-02-01 are provided in Appendix N of this report. Cleanup and D/F analysis of emission samples followed EPA Method 8290 with the following modifications:

Sample Cleanup

- Method 23 pre-sampling surrogate standards were spiked into the back-half XAD traps before shipping to the field. Method 23 internal standards were used for all samples rather than the internal standards in Method 8290. Use of the Method 23 standard scheme provided better monitoring of sampling efficiency for source emissions.

Table 5-2. Laboratory Internal Standards

Standard Type	Standard Contents	Spike Amount	Comments
PCDD/PCDF	$^{13}C_{12}$-2,3,7,8-TCDF	8000 pg	All samples
	$^{13}C_{12}$-2,3,7,8-TCDD	8000 pg	
	$^{13}C_{12}$-1,2,3,7,8-PeCDF	8000 pg	Purchased as a mixture
	$^{13}C_{12}$-1,2,3,7,8-PeCDD	8000 pg	(Cambridge Isotope
	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	8000 pg	Laboratories EDF-4053)
	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	8000 pg	
	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	8000 pg	
	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	8000 pg	
	$^{13}C_{12}$-OCDD	16000 pg	
PCB	$^{13}C_{12}$-PCB-77	4000 pg	All samples
	$^{13}C_{12}$-PCB-105	4000 pg	
	$^{13}C_{12}$-PCB-118	4000 pg	Purchased as a mixture
	$^{13}C_{12}$ PCB-126	4000 pg	(Cambridge Isotope
	$^{13}C_{12}$-PCB-156	4000 pg	Laboratories EC-4064)
	$^{13}C_{12}$-PCB-157	4000 pg	
	$^{13}C_{12}$-PCB-167	4000 pg	
	$^{13}C_{12}$ PCB-169	4000 pg	
	$^{13}C_{12}$-PCB-180	4000 pg	
	$^{13}C_{12}$ PCB-189	4000 pg	
	$^{13}C_{12}$-PCB-209	8000 pg	
PAH	d_8-Naphthalene	200 ng	All samples
	d_8-Acenaphthylene	200 ng	
	d_{10}-Acenaphthene	200 ng	
	d_{10}-Fluorene	200 ng	
	d_{10}-Phenanthrene	200 ng	
	d_{10}-Pyrene	200 ng	
	d_{10}-Fluoranthene	200 ng	
	d_{12}-Benzo(a)anthracene	200 ng	
	d_{12}-Chrysene	200 ng	
	d_{12}-Benzo(b)fluoranthene	200 ng	
	d_{12}-Benzo(k)fluoranthene	200 ng	
	d_{12}-Benzo(a)pyrene	200 ng	
	d_{12}-Perylene	200 ng	
	d_{12}-Indeno(1,2,3,-cd)pyrene	200 ng	
	d_{14}-Dibenz(a,h)anthracene	200 ng	
	d_{12}-Benzo(g,h,i)perylene	200 ng	

Table 5-3. Standards for Laboratory Control Spike Samples

Standard Type	Standard Contents	Spike Amount	Comments
PCDD/PCDF	2,3,7,8-TCDD	800 pg	LCS/LCSD samples
	1,2,3,7,8-PeCDD	4000 pg	
	1,2,3,4,7,8-HxCDD	4000 pg	Purchased as a mixture
	1,2,3,6,7,8-HxCDD	4000 pg	(Cambridge Isotope
	1,2,3,7,8,9-HxCDD	4000 pg	Laboratories EDF-7999)
	1,2,3,4,6,7,8-HpCDD	4000 pg	
	OCDD	8000 pg	
	2,3,7,8-TCDF	800 pg	
	1,2,3,7,8-PeCDF	4000 pg	
	2,3,4,7,8-PeCDF	4000 pg	
	1,2,3,4,7,8-HxCDF	4000 pg	
	1,2,3,6,7,8-HxCDF	4000 pg	
	1,2,3,7,8,9-HxCDF	4000 pg	
	2,3,4,6,7,8-HxCDF	4000 pg	
	1,2,3,4,6,7,8-HpCDF	4000 pg	
	1,2,3,4,7,8,9-HpCDF	4000 pg	
	OCDF	8000 pg	
PCB	PCB-77	0.8 ng	LCS/LCSD samples
	PCB-105	40 ng	
	PCB-114	40 ng	Purchased as a mixture
	PCB-118	40 ng	(Cambridge Isotope
	PCB-123	40 ng	Laboratories EDF-7999)
	PCB-126	4 ng	
	PCB-156	40 ng	
	PCB-157	40 ng	
	PCB-167	40 ng	
	PCB-169	8 ng	
	PCB-170	8 ng	
	PCB-180	40 ng	
	PCB-189	8 ng	
PAH	Naphthalene	200 ng	
	Acenaphthylene	200 ng	
	Acenaphthene	200 ng	
	Fluorene	200 ng	
	Phenanthrene	200 ng	
	Pyrene	200 ng	
	Fluoranthene	200 ng	
	Benzo(a)anthracene	200 ng	
	Chrysene	200 ng	
	Benzo(b)fluoranthene	200 ng	
	Benzo(k)fluoranthene	200 ng	
	Benzo(a)pyrene	200 ng	
	Perylene	200 ng	
	Indeno(1,2,3,-cd)pyrene	200 ng	
	Dibenz(a,h)anthracene	200 ng	
	Benzo(g,h,i)perylene	200 ng	

- For the front-half air samples, $^{37}Cl_4$- 2,3,7,8-TCDD was utilized as a cleanup standard following procedures in Method 1613 to monitor analyte recovery through the cleanup process. This was not used in the cleanup of back-half air samples as $^{37}Cl_4$-2,3,7,8-TCDD was added to XAD as a pre-sampling surrogate.

- Reduced volumes of acid, base, and salt solutions were used for the acid/base partitioning cleanup step with no adverse impact upon internal standard recoveries.

- Silica/alumina column cleanup procedures were modified to utilize a stacked column approach which decreases sample preparation time and solvent usage with no adverse impact on internal standard recoveries.

- The carbon column cleanup was modified to use a 20% mixture of Carbopack-C/Celite which is adapted from Method 1613, Section 13.5.

- The final extract concentration procedure was modified to enhance extract transfer to the GC autosampler vials.

Sample Analysis

- GC/HRMS was calibrated using Method 23 calibration solutions since the Method 23 pre-sampling surrogate and internal standard scheme was used for these samples.

- Only 1 μL of solution/extract was injected on-column per run as opposed to 2 μL (split-splitless mode) specified in Method 8290.

- The GC temperature program was modified to permit the solvent peak to elute more slowly so as to not trip the source ion gauge. In addition, the final temperature was reduced to 320°C to accommodate the upper temperature limit of the GC column.

- The ion masses monitored and the quantification relationships used for calculations follow Method 23 since the Method 23 standard scheme was used for these samples.

Any revisions to the procedures stated above are detailed in the QAPP Amendment/Corrective Action Records which are included in Appendix P.

5.1.2.4 PAH Extract Cleanup and Analysis

Extracts from the emission samples designated for PAH analysis were shipped to Quanterra for analysis. Cleanup and PAH analysis of emission samples were conducted following the procedures in California Air Resources Board, Method 429, "Determination of Polycyclic Aromatic Hydrocarbons (PAH) Emissions from Stationary Sources" with the following revisions:

- Spiking volumes and concentrations. The concentration of the internal standard daily spiking solution was 1 ng/μL for all analytes and was spiked at 200 ng per sample. The concentration of the surrogate and alternate standards was 1 ng/μL for all analytes and was spiked at 200 ng per sample. The concentration of the recovery standard was 1 ng/μL for all analytes and was spiked at 50 ng per sample extract. Spike analytes and amounts are listed in Table 5-3.

- Sample extract cleanup. Silica gel was used for extract cleanup. The amount of packing material used and the elution profile differ from that referenced in the method.

- Analysis. Column specifications, split time, and instrument temperatures differed from CARB Method 429 as shown in Table 5-4. Assignment of recovery standards for internal standard calculations and internal standards for native analyte calculations were modified by the substitution and addition of standards provided by the analyzing laboratory.

- Sample reporting. A reporting limit convention was used which includes a limit of detection (LOD) defined as five times the blank result or 10 ng/split, whichever is greater. All detectable levels in emission samples or laboratory QC samples above the reporting limit were reported.

A copy of CARB Method 429 is provided in Appendix N to this report.

Table 5-4. Gas Chromatographic Operating Conditions for PAH Analysis

Operating Parameter	Used for Analyses	CARB Method 429
Column Type	DB-5	DB-5
Length (m)	60	30
ID (mm)	0.32	0.25
Film Thickness (μm)	0.25	0.32
Helium Linear Velocity (cm/sec)	30	30
Injection Mode	Splitless	Splitless
Splitless Time (sec)	60	30
Initial Temperature (°C)	85	45
Initial Time (min)	4	4
Program Rate (°C)	8	8
Final Temperature (°C)	300	300
Final Hold Time (min)	14	Until benzo(g,h,i)perylene has eluted
Injector Temperature (°C)	260	320

5.2 PROCESS SCRUBBER WATER

5.2.1 Scrubber Water Sampling

As shown in Figure 5-6, scrubber water was sampled every 60 minutes using procedures in ASTM Method D3370-95a. Scrubber water was collected in two containers during each sampling period. Scrubber water was first collected in a glass container for immediate temperature determination and on-site pH analysis. The temperature of the scrubber water collected in this container was measured using a glass thermometer. This sample was then taken to the field laboratory for pH measurement. When the pH of this first sample was measured, that sample was discarded.

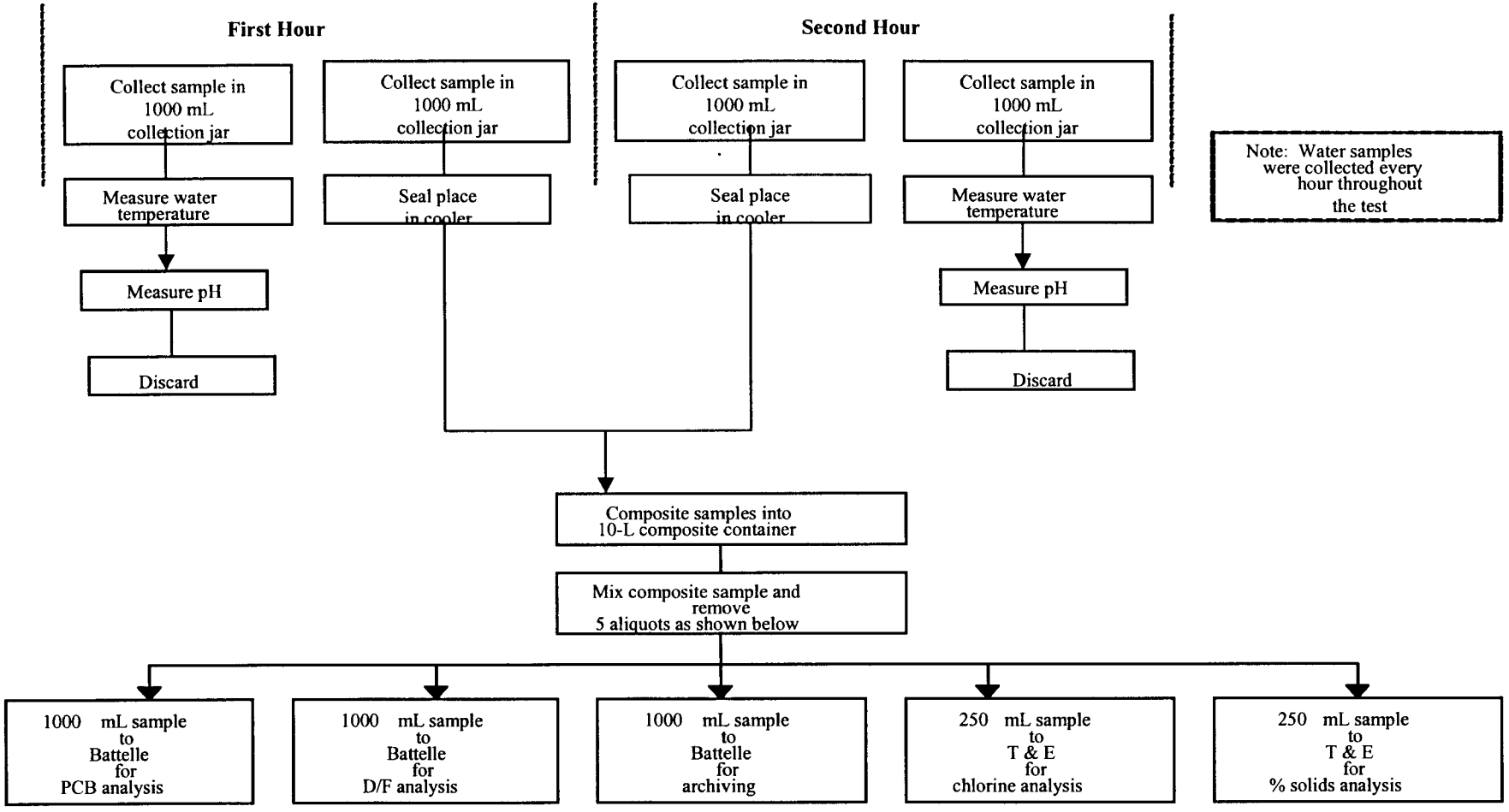


Figure 5-6. Flow Chart for Scrubber Water Sampling

The second container used to collect scrubber water was a 1000 mL collection jar. To collect water into this container, the jar was filled twice with scrubber water and each collection discarded. After the second filling was discarded, the actual sample (third filling) was taken. The jar was filled to the brim to eliminate any head space, sealed, and placed into a cooler and maintained at approximately 4°C until the end of the test. Collection jars were rinsed with distilled water and then acetone before each sample was collected. Any other glassware used, including the composite container, were rinsed with distilled water, followed by acetone between uses. After all six water samples were collected in the collection jars, the contents of each jar was emptied into a precleaned composite container and the combined contents were gently stirred, not shaken. Five aliquots were removed and placed in five separate containers for PCB analysis, D/F analysis, chlorine analysis, percent solids analysis, and archiving as shown below:

PCB analysis:	1000-mL amber glass jar
D/F analysis:	1000-mL amber glass jar
Chlorine analysis:	1000-mL amber glass jar
Percent solids analysis:	1000-mL amber glass jar
Archiving:	1000-mL amber glass jar.

Immediately upon recovery, all samples were placed into insulated coolers packed with frozen blue ice to protect them from light and heat. The samples remained inside the coolers while at the field site and during transport to Battelle. Samples from each test were transported to Battelle at the end of the test day or the following morning. While at Battelle's laboratories, the scrubber water samples were kept refrigerated until analysis.

5.2.2 Scrubber Water Analysis

5.2.2.1 PCB Analysis

Scrubber water samples were analyzed for PCBs according to the draft method, "Determination of Toxic Polychlorinated Biphenyls in Sewage Incinerator Scrubber Water Using Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry" dated July 20, 1999. The extraction procedures are illustrated in Figure 5-7. The complete method is provided in Appendix N to this report.

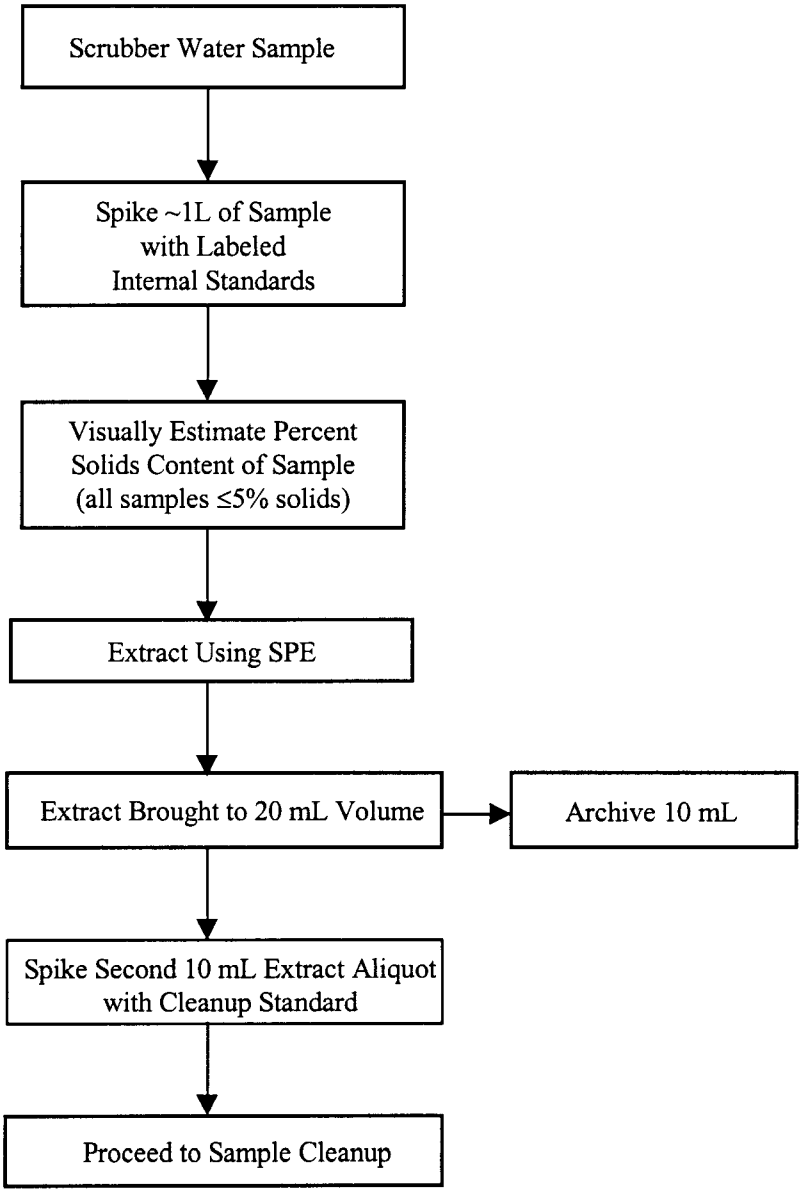


Figure 5-7. Flow Diagram for Preparation and Extraction of Scrubber Water Samples for PCB Analysis

5.2.2.2 D/F Analysis

Scrubber water samples were analyzed for D/F according to EPA Method 8290 "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)" with revisions described in Battelle SOP Nos. SOP0802-01-01 and SOP0802-02-01. The extraction procedures are illustrated in Figure 5-8. SOP Nos. SOP0802-01-01 and SOP0802-02-01 are provided in Appendix N of this report. Cleanup and D/F analysis of scrubber water samples followed EPA Method 8290 with the following modifications:

Sample Cleanup

- 15 labeled compounds were used in the internal standard solution instead of the 9 specified in Method 8290 to provide improved accuracy.
- Cleanup standard $^{37}Cl_4$- 2,3,7,8-TCDD was utilized per Method 1613 to monitor analyte recovery through the cleanup process.
- Reduced volumes of acid, base, and salt solutions were used with no adverse impact upon internal standard recoveries.
- Modified silica/alumina column to utilize a stacked column approach and enhance extract transfer from the carbon column to the GC autosampler vials were made.

Sample Analysis

- GC/HRMS was calibrated at levels specified in Method 1613, Table 4 with an additional standard at concentrations equivalent to one-half the level of Method 1613's lowest calibration point. This provided an expanded calibration concentration range compared with that of Method 8290.
- Only 1 μL of solution/extract was injected on-column per run as opposed to 2 μL (split-splitless mode) specified in Method 8290.
- A combination solution was utilized on DB-5 columns at the beginning and end of each 12-hour run period. This allowed the determination of calibration and column performance in a single run.
- The GC temperature program was modified to permit the solvent peak to elute more slowly so as to not trip the source ion gauge. In addition, the final temperature was reduced to 320°C to accommodate the upper temperature limit of the GC column.

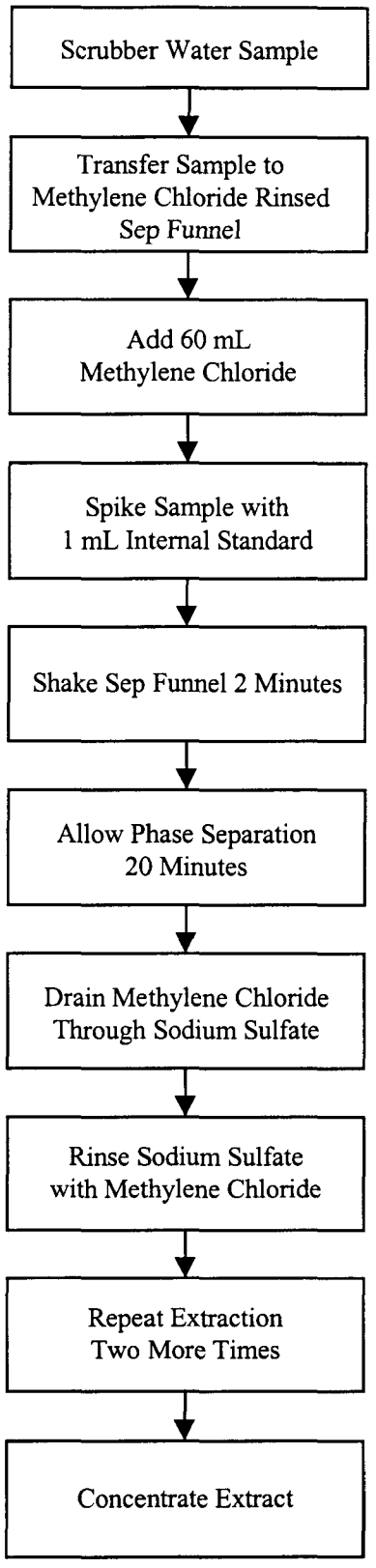


Figure 5-8. Flow Diagram for Preparation and Extraction of Scrubber Water Samples for D/F Analysis

- All five congener groups were monitored separately.
- The mass of the cleanup standard $^{37}Cl_4$-2,3,7,8-TCDD was monitored per Method 1613, Table 8.

Any other revisions are detailed in the QAPP Change Forms/Corrective Action Records which are provided in Appendix P.

5.2.2.3 Chlorine Analysis

Analyses of chlorine in scrubber water samples were conducted using a Hach Model DR2010 photometric analyzer. This unit measures free Cl_2 and total Cl_2 in the 0 to 2 mg/L range. The measurement technique is equivalent to Method 4500 G of Standard Methods for Examination of Water and Wastewater. This method employees a colormetric procedure using a filter photometer. A copy of this method can be found in Appendix N to this report.

5.2.2.4 pH/Temperature Determination

The pH of the scrubber water samples was measured according to Standard Methods for Examination of Water and Wastewater, Method 4500 H, "pH Value." The temperature of the scrubber water was measured immediately after collection in the field using a glass thermometer.

5.3 PROCESS SLUDGE FEED

5.3.1 Sludge Feed Sampling

A sludge feed sample was collected every 60 minutes (Figure 5-9) during the test. The sludge sample was scooped with a precleaned metal scoop and placed in a 750-mL glass container. The full container was then emptied into a precleaned large stainless steel holding container. After all six grab samples were collected, the composite was then mixed using the pan mixing/quartering technique described in ASTM Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities (Designation D6051-96)

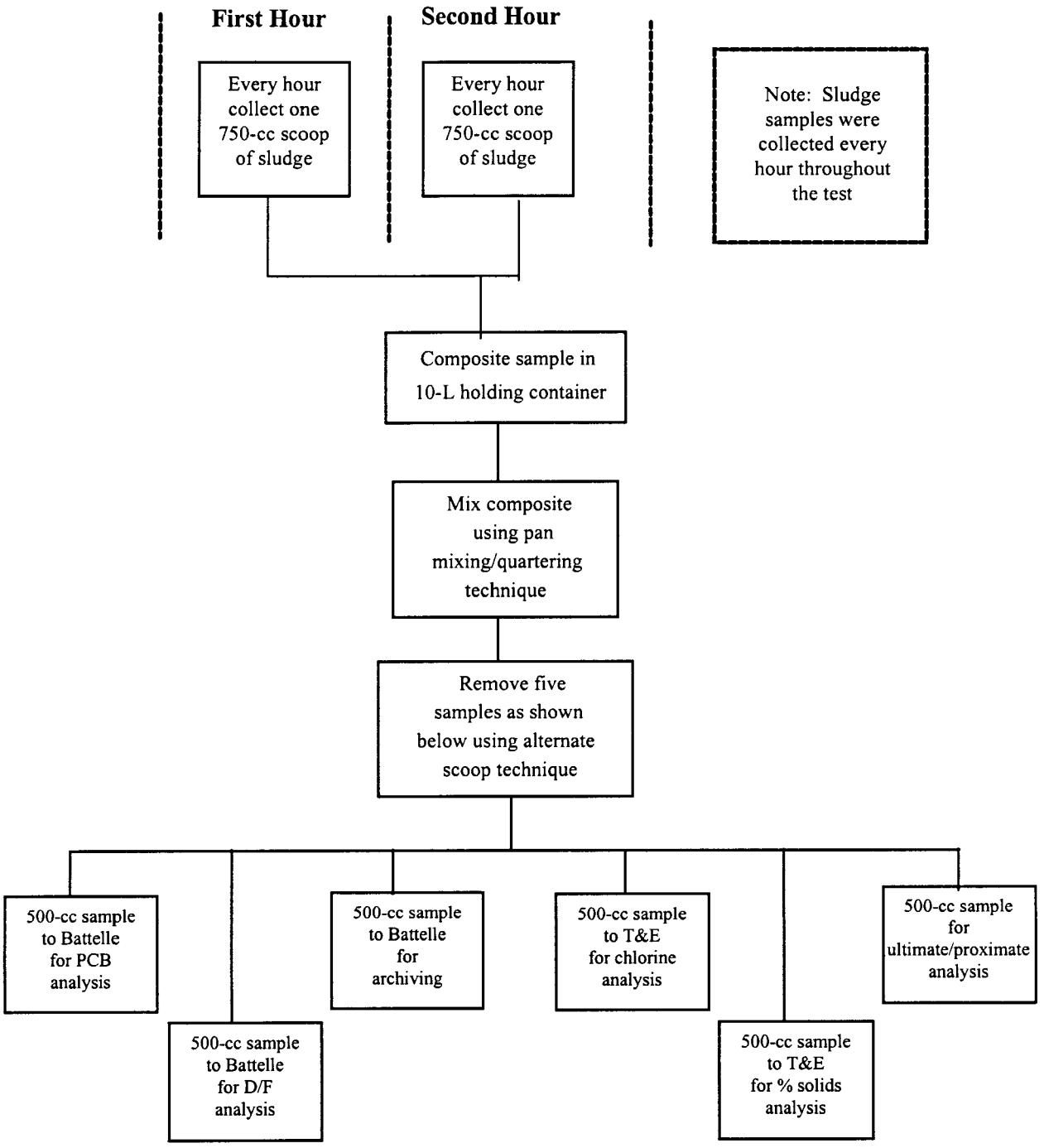


Figure 5-9. Flow Chart for Sewage Sludge Feed Sampling

found within the appendices. Six aliquot samples were removed using an alternate scope technique (also included in ASTM Method D6051-96) and placed into separate containers for PCB analysis, D/F analysis, chlorine analysis, percent solids analysis, ultimate/proximate analysis, and archiving as shown below.

PCB analysis:	1000-mL vented amber glass jar (500 cc sample)
D/F analysis:	1000-mL vented amber glass jar (500 cc sample)
Chlorine analysis:	1000-mL amber glass jar
Percent solids analysis:	1000-mL amber glass jar
Ultimate/proximate analysis:	1000-mL amber glass jar
Archiving	1000-mL amber glass jar.

The sample scoop and all other containers or items that come in contact with the sludge sample were rinsed with distilled water and then acetone before being reused.

Acid was added for sterilization to the jars for PCB analysis, D/F analysis, and archiving (40 mL of 1:1 HNO_3 for every 500 mL of sample). The sewage sludge feed samples were stored at ≤4°C in on-site coolers until transported to Battelle. Containers for chlorine and percent solids analysis were hand carried to the on-site T&E laboratory.

The samples remained inside the coolers while at the field site and during transport to the analytical laboratory. Samples from each test were transported to Battelle at the end of the test day or the following morning. While at Battelle's laboratories, the sewage sludge feed samples were kept refrigerated until analysis.

5.3.2 Sewage Sludge Feed Analysis

5.3.2.1 PCB Analysis

Sewage sludge feed samples were analyzed for PCBs according to the draft method "Determination of Toxic Polychlorinated Biphenyls in Sewage Sludge Using Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry" dated July 20, 1999. The extraction procedures are illustrated in Figure 5-10. This method is provided in

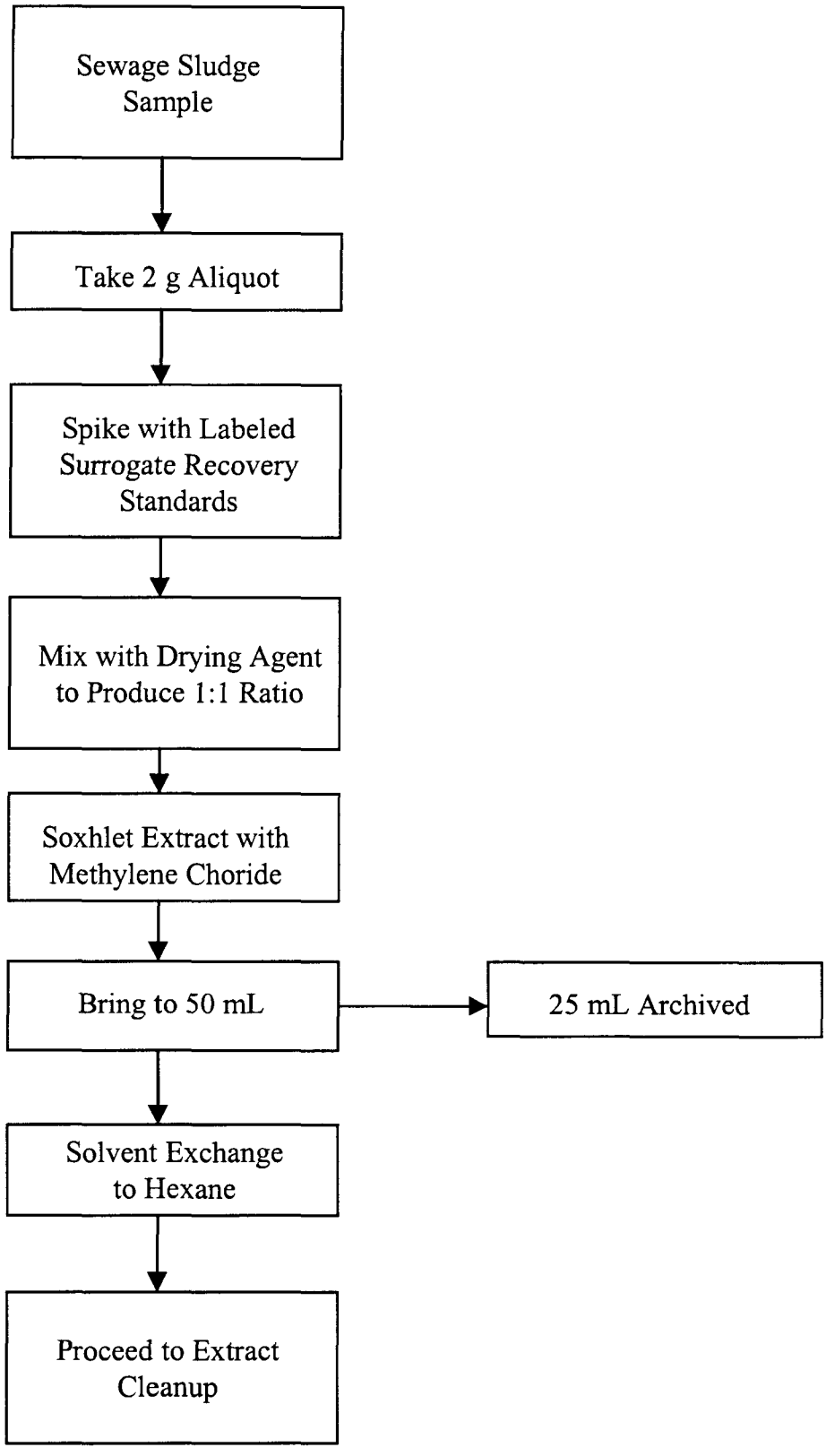


Figure 5-10. Flow Diagram for Sample Preparation and Extraction of Sewage Sludge Feed Samples for PCB Analysis

Appendix N of this report. Any revisions to this draft method are detailed in the QAPP Amendments/Corrective Action Records which are included in Appendix P.

5.3.2.2 D/F Analysis

Sewage sludge feed samples were analyzed for D/Fs according to EPA Method 8290, "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)" with revisions described in Battelle SOP Nos. SOP0802-01-01 and SOP0802-02-01. The extraction procedures are illustrated in Figure 5-11. A copy of SOP Nos. SOP0802-01-01 and SOP0802-02-01 are provided in Appendix N of this report.

Cleanup and D/F analysis of sewage sludge samples followed EPA Method 8290 with the following modifications:

Sample Cleanup

- 15 labeled compounds were used in the internal standard solution instead of the 9 specified in Method 8290 to provide improved accuracy.
- Cleanup standard $^{37}Cl_4$- 2,3,7,8-TCDD was utilized per Method 1613 to monitor analyte recovery through the cleanup process.
- Reduced volumes of acid, base, and salt solutions were used with no adverse impact upon internal standard recoveries.
- Modified silica/alumina column to utilize a stacked column approach and enhance extract transfer from the carbon column to the GC autosampler vials were made.

Sample Analysis

- GC/HRMS was calibrated at levels specified in Method 1613, Table 4 with an additional standard at concentrations equivalent to one-half the level of Method 1613's lowest calibration point. This provided an expanded calibration concentration range compared with that of Method 8290.
- Only 1 μL of solution/extract was injected on-column per run as opposed to 2 μL (split-splitless mode) specified in Method 8290.

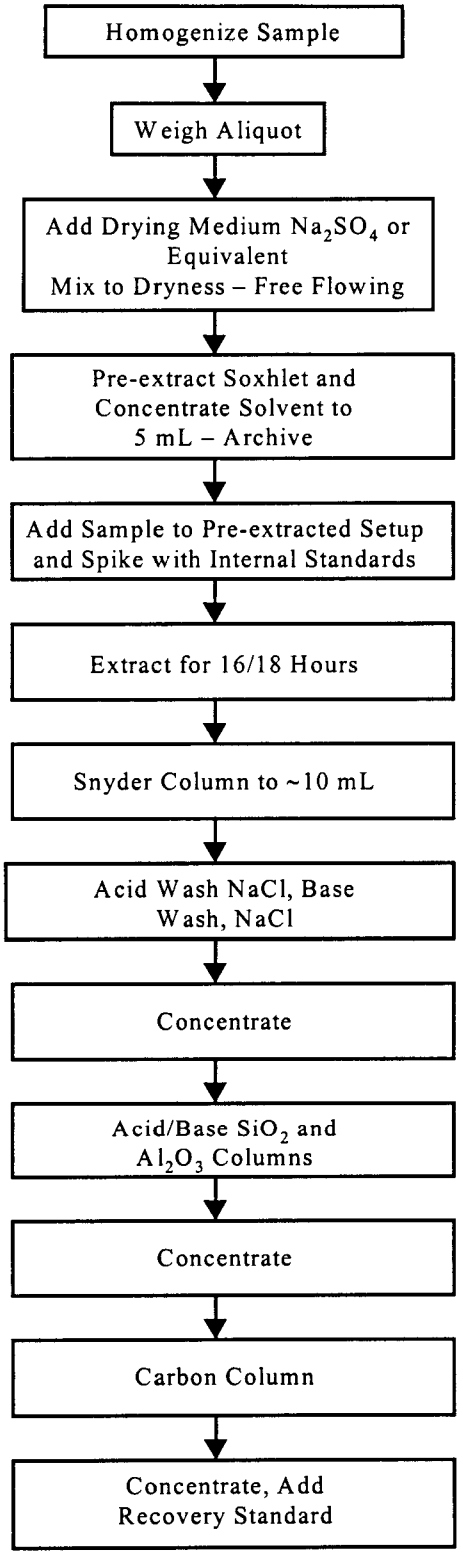


Figure 5-11. Flow Diagram for Preparation and Extraction of Sludge Feed Samples for D/F Analysis

- A combination solution was utilized on DB-5 columns at the beginning and end of each 12-hour run period. This allowed the determination of calibration and column performance in a single run.

- The GC temperature program was modified to permit the solvent peak to elute more slowly so as to not trip the source ion gauge. In addition, the final temperature was reduced to 320°C to accommodate the upper temperature limit of the GC column.

- All five congener groups were monitored separately.

- The mass of the cleanup standard $^{37}Cl_4$-2,3,7,8-TCDD was monitored per Method 1613, Table 8.

Any other revisions are detailed in the QAPP Amendment/Corrective Action Records which are included in Appendix P.

5.3.2.3 Chlorine Analysis

Analysis of chlorine in sewage sludge feed samples was conducted using a Hach Model DR 2010 analyzer. This unit measures free Cl_2 and total Cl_2 in the 0 to 2 mg/L range. The measurement technique is equivalent to Method 4500G of Standard Methods for Examination of Water and Wastewater. The free and total chlorine analyses of sewage sludge were performed on an extract obtained by modified ASTM Method D5233. A copy of the Method 4500G, "DPD Colorimetric Method" and modified Method ASTM D5233 can be found in Appendix N to this report.

5.3.2.4 Percent Solids Analysis

Percent solids in sludge feed samples were determined following Standard Methods for Examination of Water and Wastewater, Method 2540 B, "Total Solids Dried at 103-105°C." Each sample was analyzed in triplicate for percent solids. A copy of this method can be found in Appendix N to this report.

5.3.2.5 Ultimate/Proximate Analysis

Ultimate/proximate analysis of sludge feed samples was performed following ASTM Methods D3176 and D3173, respectively. Copies of these ASTM methods are included in Appendix N to this report.

6.0 INTERNAL QA/QC ACTIVITIES

6.1 QA/QC CHECKS AND ISSUES

6.1.1 Field Sampling

6.1.1.1 MM5 Emission Sampling

Cyclonic Flow. Prior to sampling on the first test day a cyclonic flow check was conducted on the stack for Incinerator No. 6. The cyclonic flow was checked at each of the test points shown in Figure 3-3 following procedures described in 40 CFR 60, Appendix A, Method 1 Section 2. All test points were well below the allowable 20° with a maximum of 8° and an average rotational angle of 4.58°. The cyclonic flow check sheet is provided in Appendix B-1 to this report.

Gas Stratification Check. At the completion of the cyclonic flow check a gas stratification check was conducted using a single CEM system. The CEM system determination indicated that gas stratification was possible in the exhaust stack. The WAM made a decision to traverse the same MM5 traverse point locations, using the CEM sampling probe simultaneous with each MM5 sampling run. The results of the stratification check are shown in Table 6-1. The field data can be found in Appendix B-1 of this report.

Leak Check. The MM5 sampling train was leak checked at the beginning and end of each test run and after moving from one sampling port to another during a run. The leak checks were considered acceptable if a leak rate of less than 0.02 ft^3/min was observed at the highest vacuum recorded during the sampling run.

At the port change of the first sampling run, a leak rate of 0.06 ft^3/min was observed. This leak check was determined not to be acceptable and the sampling run invalidated. Sampling was discontinued, the sampling train was recovered using the stated procedures in the SSTP, and the samples were archived.

Table 6-1. Gas Stratification Check Results

Sampling Point	Time	O_2 (% dv)	% Diff. from Mean	CO_2 (% dv)	% Diff. from Mean	CO (ppm_{dv})	% Diff. from Mean
A1	1545	15.26	4.2	4.31	-5.7	956	-13.5
A2	1549	14.85	1.4	4.27	-6.6	858	-22.4
A3	1553	14.73	0.5	4.35	-4.8	947	-14.3
A4	1557	14.94	2.0	4.16	-9.0	995	-10.0
A5	1601	15.09	3.0	4.29	-6.8	1060	-4.1
A6	1605	14.86	1.4	4.44	-2.8	1156	4.6
B1	1610	15.15	3.4	4.15	-9.2	1216	9.1
B2	1614	14.73	0.5	4.45	-2.6	1203	8.9
B3	1618	14.38	-1.8	4.76	4.2	1209	9.4
B4	1622	13.88	-5.3	5.21	14.0	1294	17.1
B5	1626	13.87	-5.3	5.28	15.5	1218	10.2
B6	1630	14.03	-4.2	5.15	12.7	1152	4.3
Average	--	14.65	--	4.57	--	1105	–

Note: Results obtained with single CEM analyzer on July 19, 1999, 1543-1630.

During the next three sampling runs all pre-test, post-test, and port change leak checks were successfully completed. Leak check results can be found on the isokinetic data sheets in Appendix B-1-2 to this report.

Isokinetic Sampling Check. Isokinetic calculations performed at the end of each test verified that sampling was conducted within the ±10 percent criteria for all tests. These results were:

Run 2	91.7% Isokinetic
Run 3	95.9% Isokinetic
Run 4	97.1% Isokinetic

Although acceptable, the isokinetic sampling rate during MSD-MM5-R2 was lower than expected (91.7 percent), because of an incorrect assumption of flue gas moisture. The K factor was initially calculated for MSD-MM5-R1, based on an assumed moisture of 10 percent, a value measured during previous emissions tests. This value was significantly higher than the actual flue gas moisture content of 4-5 percent measured during the sampling program. Although

preliminary moisture measurements taken on July 19, 1999, supported the lower moisture values, these readings were thought to be anomalous at the time.

The data from the voided MSD-MM5-R1 also indicated 4.7 percent moisture. However, this value was also assumed invalid, because of suspected dilution caused by inleakage indicated by a failed leak check at the midpoint of the sampling run. It is now suspected that the leak originated during removal of the train from the sampling port and had no bearing on sample or moisture collection.

Following recovery of MSD-MM5-R2, the flue gas moisture of 4-5 percent was confirmed and the K factor was recalculated for MSD-MM5-R3 based on this measurement.

Sampling Train Temperatures. Sample probe, filter holder, XAD-2 trap inlet, impinger train exit, and dry gas meter inlet and outlet temperatures were recorded on the isokinetic data sheet every five minutes during each of the three MM5 sampling runs. The XAD-2 trap temperature during all three runs never exceeded the allowable 68°F. Probe and filter holder temperatures were maintained within the acceptable range of 248 ± 25°F. All temperature data can be found on the isokinetic data sheets in Appendix B-1-2 to this report.

6.1.1.2 Continuous Emission Monitoring

Prior to sampling, the CEM sampling system was checked for leaks by turning on the sampling system pump, sealing off the end of the sampling probe, and determining that there was no appreciable flow through the sampling system's flow indicators located in the mobile sampling laboratory.

CEM Calibration and Linearity Check. On each test day and prior to sampling the O_2, CO_2, and CO, the continuous analyzers were calibrated at a low, mid, and high span point to check the instrument calibration and to verify instrument linearity. Results of the daily instrument calibration and linearity checks are shown in Table 6-2. The field data can be found in Appendix L-1 to this report. EPA traceability protocol gas certificates of analysis can be found in Appendix L-5.

Table 6-2. CEM Calibration and Linearity Check Data

Gas	Certified Concentration	Run 2 July 20	Run 3 July 21	Run 4 July 22
	Instrument Reading			
O_2	Zero 21.00% 11.50%	0.00% 21.06% 11.80%	0.00% 20.99% 11.75%	0.00% 20.81% 11.53%
CO_2	Zero 18.00% 11.01%	0.10% 18.05% 10.96%	0.11% 18.46% 11.12%	0.11% 17.96% 10.91%
CO	Zero 5971 ppm 3005 ppm 1809 ppm	0006 ppm 5922 ppm 3105 ppm 1878 ppm	0000 ppm 5676 ppm 3106 ppm 1878 ppm	0004ppm 5925 ppm 3045 ppm 1852 ppm

Response Time Check. At the start of the test program a response time determination was conducted for the O_2, CO_2, and CO CEMs. The instrument response was determined by introducing a span gas into the sampling system and timing the length of time required for the instrument to reach the span concentration. Average response times for each of the instruments are provided in Table 6-3. The field data sheets can be found in Appendix L-1-2 to this report.

Table 6-3. Response Time Check

Instrument	Concentration	Up Scale Average Response Time, sec	Down Scale Average Response Time, sec
O_2	21.0%	126	114
CO_2	18.0%	120	124
CO	3005 ppm	151	144

Bias and Instrument Drift Checks. Bias checks were performed prior to and after each test run. After calibrating the O_2, CO_2, and CO monitors, calibration gas was introduced remotely through the probe and sampling line to verify the sampling system bias. The bias error did not exceed five percent of the instrument span, as required by EPA Method 6C.

A comparison between the pre-test and post-test bias check was then used to determine the CEM drift over the test period. Results of the bias check and instrument drift check are given in Table 6-4. All bias checks and CEM drift checks were within the allowable limits.

6.1.2 Sample Handling

Immediately upon recovery of each MM5 sampling train, all samples including liquid rinses, filters, and sorbent traps were placed into insulated coolers and packed with frozen blue ice to protect them from light and heat. While in the custody of the sampling team, the temperatures inside the coolers were checked every 12 hours to ensure that the samples were maintained at ≤4°C. Scrubber water samples and sewage sludge samples were packed in a cooler separate form the emission samples. Battelle transported all samples requiring off-site analyses to their Columbus Laboratories at the end of each test day or within 24 hours of collection. A schedule of sample recovery and transfer is provided in Table 6-5. Sample temperatures were measured at each point of transfer. All temperatures were maintained at or below 4°C except in the following cases. The temperature of two coolers containing Run 2 emission samples was 13°C and 8°C, and the temperature of one cooler containing some Run 4 scrubber water samples was 5.4°C upon receipt at Battelle as noted in the laboratory sample log-in book. These minor exceedances in storage temperature had no apparent impact on sample results as data for these affected samples were comparable to nonaffected samples. Sample chain-of-custody forms are provided in Appendix D.

Table 6-5. Sample Transfer Schedule

	Run 2	Run 3	Test Run 4
Sample Recovered	2300 (7/20)	1840 (7/21)	1800 (7/22)
Transfer to Battelle Courier	1030 (7/21)	1900 (7/21)	0800 (7/23)
Received at Battelle	1330 (7/21)	2230 (7/21)	1335 (7/23)

Table 6-4. Bias and Instrument Drift Check Data

	Run 2			Run 3			Run 4		
	O_2 ($\%_{dv}$)	CO_2 ($\%_{dv}$)	CO (ppm_{dv})	O_2 ($\%_{dv}$)	CO_2 ($\%_{dv}$)	CO (ppm_{dv})	O_2 ($\%_{dv}$)	CO_2 ($\%_{dv}$)	CO (ppm_{dv})
Span	25	20	6000	25	20	6000	25	20	6000
Zero Drift									
Initial System Response	0.17	0.12	4.13	0.02	0.10	3.61	0.06	0.11	5.42
Final System Response	0.07	0.14	0.77	0.12	0.14	0.26	0.12	0.13	5.71
Zero Drift (%)	-0.40	0.10	-0.06	0.40	0.20	-0.06	0.24	0.10	0.00
Calibration Drift									
Initial System Response	11.80	10.99	1847.30	11.55	10.84	1876.80	11.43	10.85	1831.90
Final System Response	11.62	11.05	1830.90	11.44	10.89	1831.10	11.80	10.85	1853.80
Calibration Drift (%)	-0.72	0.30	-0.27	-0.44	0.25	-0.76	1.48	0.00	0.36
System Bias (Zero)									
Analyzer Cal. Response	0.00	0.10	6.07	0.00	0.11	-0.45	0.00	0.11	3.63
Initial System Response	0.17	0.12	4.13	0.02	0.10	3.61	0.06	0.11	5.42
Initial System Bias (%)	0.68	0.10	-0.03	0.08	-0.05	0.07	0.24	0.00	0.03
Midpoint System Response	0.11	0.15	0.00	0.11	0.15	2.06	0.11	0.13	5.42
Midpoint System Bias (%)	0.44	0.25	-0.10	0.44	0.20	0.04	0.44	0.10	0.03
Final System Response	0.07	0.14	0.77	0.12	0.14	0.26	0.12	0.13	5.71
Final System Bias (%)	0.28	0.20	-0.09	0.48	0.15	0.01	0.48	0.10	0.03
Span	25	20	6000	25	20	6000	25	20	6000
System Bias (Upscale)									
Analyzer Cal. Response	11.80	10.96	1878.00	11.75	11.12	1878.00	11.53	10.91	1852.00

Table 6-4. (Continued)

	Run 2			Run 3			Run 4		
	O_2 ($\%_{dv}$)	CO_2 ($\%_{dv}$)	CO (ppm_{dv})	O_2 ($\%_{dv}$)	CO_2 ($\%_{dv}$)	CO (ppm_{dv})	O_2 ($\%_{dv}$)	CO_2 ($\%_{dv}$)	CO (ppm_{dv})
Initial System Response	11.80	10.99	1847.30	11.55	10.84	1876.80	11.43	10.85	1831.90
Initial System Bias (%)	0.00	0.15	-0.51	-0.80	-1.40	-0.02	-0.40	-0.30	-0.33
Midpoint System Response	11.59	10.93	1831.30	11.70	10.88	1851.50	11.73	10.91	1831.30
Midpoint System Bias (%)	-0.84	-0.15	-0.78	-0.20	-1.20	-0.44	0.80	0.00	-0.35
Final System Response	11.62	11.05	1830.90	11.44	10.89	1831.10	11.80	10.85	1853.80
Final System Bias (%)	-0.72	0.45	-0.78	-1.24	-1.15	-0.78	1.08	-0.30	0.03

6.1.3 Laboratory Analysis

Section 6.3 of this report provides a complete review of laboratory QA/QC performance including listings of method target performance criteria and results achieved. In this section, only potential issues with laboratory analyses that could impact the quality of the data are discussed. In discussing PCB, D/F, and PAH analytical issues, the following items were addressed in order as necessary:

- Calibration (initial and continuing)
- Internal standard recoveries
- Spike recoveries and precision of LCS/LCSD
- Method blank
- Detection limit
- Turnaround time.

6.1.3.1 Emission Samples

PCB Analysis. The initial calibration for both the SPB-Octyl and DB-1 analyses met the requirement for response factors having less than 35% relative standard deviation (RSD) for all analytes (actual range = <17% for SPB-Octyl and DB-1 calibrations). The continuing calibrations met the requirement for response factors being within 35% of the response factors generated in the initial calibration for at least 70% of the analytes.

Field Samples. Summary tables of PCB results and standard recoveries for the front half and back half air samples are shown in Tables 6-6 and 6-7a, respectively. Internal standard recoveries give an indication of how well analytes were extracted from the medium and retained during extract cleanup. For the front half air samples, recoveries of all internal standards were within the method specified limits of 30-150% and ranged from 35-77%. Internal standard recoveries in the back half air samples were also within the 30-150% limits and ranged from 49-84%. These internal standard recoveries indicate analytes were well recovered during laboratory extraction and were retained during the extract cleanup process. $^{13}C_{12}$-PCB-81 and $^{13}C_{12}$-PCB-111 were added as cleanup standards in processing the front half air samples and as pre-sampling surrogate standards spiked into XAD resin before shipping to the field in the back half air samples. Method specified recovery ranges for these standards were from 10-150% for

Table 6-6. Summary of PCB Results and Standard Recoveries for Front Half Air Samples

ANALYTES	RUN 2 48190-11-02 FH-R2-PCB (pg/sample)	DL (pg/sample)	RUN 3 48190-11-03 FH-R3-PCB (pg/sample)	DL (pg/sample)	RUN 4 48190-11-04 FH-R4-PCB (pg/sample)	DL (pg/sample)
PCB-77	750		650		551	
PCB-123	38.4 B		38.7 B		28.5 B	
PCB-118	1300 B		1260 B		1120 B	
PCB-114	91.6 B		72.9 B		62.4 B	
PCB-105	624 B		585 B		524 B	
PCB-126	35.5 B		24.7 B		23.2 B	
PCB-167	65.8 B		58.6 B		51.9 B	
PCB-156	131 #,B		112 #,B		107 #,B	
PCB-157	37.7 #,B		28.5 #,B		28.5 #,B	
PCB-169	61.8 B		30.5 B		38.3 B	
PCB-180	307 B		236 B		257 B	
PCB-170	148 B		106 B		112 B	
PCB-189	17.4 B		12.4 B		20 B	

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	RUN 2 48190-11-02 FH-R2-PCB (pg/sample)	RECOVERY (%)	RUN 3 48190-11-03 FH-R3-PCB (pg/sample)	RECOVERY (%)	RUN 4 48190-11-04 FH-R4-PCB (pg/sample)	RECOVERY (%)
13C-PCB-77	30-150	4000	2660	67	2200	55	2210	55
13C-PCB-118	30-150	4000	1830	46	1390	35	1450	36
13C-PCB-105	30-150	4000	1910	48	1470	37	1440	36
13C-PCB-126	30-150	4000	2280	57	1770	44	1820	45
13C-PCB-167	30-150	4000	1820	46	1450	36	1460	37
13C-PCB-156	30-150	4000	3100 #	77	2400 #	60	2020 #	51
13C-PCB-157	30-150	4000	2760 #	69	2210 #	55	1790 #	45
13C-PCB-169	30-150	4000	2170	54	1750	44	1900	48
13C-PCB-180	30-150	4000	2360	59	1920	48	1860	47
13C-PCB-189	30-150	4000	2350	59	2030	51	1960	49
13C-PCB-209	30-150	8000	2860	36	3140	39	3180	40
CLEANUP STANDARDS								
13C-PCB-81	10-150	2000	1290	65	1050	53	1150	58
13C-PCB-111	20-130	10000	5690	57	4090	41	4850	48

DL = detection limit.
# = Value from second column confirmation.
B = Congener found in lab blank at >20% of the concentration found in the sample.

Table 6-7a. Summary of PCB Results and Standard Recoveries for Back Half Air Samples

ANALYTES	RUN 2 48190-09-02 BH-R2-PCB (pg/sample)	DL (pg/sample)	RUN 3 48190-09-03 BH-R3-PCB (pg/sample)	DL (pg/sample)	RUN 4 48190-09-04 BH-R4-PCB (pg/sample)	DL (pg/sample)
PCB-77	146000 E 90600 *		126000 E 65800 *		47400 E 28100 *	
PCB-123	665		617		229	
PCB-118	32100		29600		13700	
PCB-114	2180		1920		855	
PCB-105	15000		13800		5810	
PCB-126	4060		3390		1390	
PCB-167	2210		1920		858	
PCB-156	3640 #		3400 #		1320 #	
PCB-157	1250 #		1080 #		499 #	
PCB-169	3210		2700		902	
PCB-180	15400		13700		5480	
PCB-170	6170		5550		2820	
PCB-189	538		430		272	

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	RUN 2 48190-09-02 BH-R2-PCB (pg/sample)	RECOVERY (%)	RUN 3 48190-09-03 BH-R3-PCB (pg/sample)	RECOVERY (%)	RUN 4 48190-09-04 BH-R4-PCB (pg/sample)	RECOVERY (%)
13C-PCB-77	30-150	4000	2510	63	2780	70	2080	52
13C-PCB-118	30-150	4000	2360	59	2450	61	2610	65
13C-PCB-105	30-150	4000	2480	62	2650	66	2910	73
13C-PCB-126	30-150	4000	1960	49	2270	57	2200	55
13C-PCB-167	30-150	4000	2540	63	2550	64	2620	66
13C-PCB-156	30-150	4000	3290 #	82	3270 #	82	3350	84
13C-PCB-157	30-150	4000	3360 #	84	3330 #	83	3300	82
13C-PCB-169	30-150	4000	2120	53	2040	51	2660	66
13C-PCB-180	30-150	4000	2170	54	2360	59	2350	59
13C-PCB-189	30-150	4000	2400	60	2580	64	2500	63
13C-PCB-209	30-150	8000	4810	60	5140	64	5380	67
PRE-SAMPLING SURROGATE STANDARDS								
13C-PCB-81	10-150	2000	1070	54	1210	61	414	21
13C-PCB-111	20-130	10000	5240	52	5090	51	2760	28

= Value from second column confirmation.

* = Extract was diluted with additional internal standard and re-analyzed to bring PCB-77 concentration within calibration range. Concentration has been corrected for original extract PCB-77 recovery (shown). Original dilution factor 1:2, PCB-77 result dilution factor 1:25.

E = Original result above calibration range.

DL = Detection limit.

$^{13}C_{12}$-PCB-81 and 20-130% for $^{13}C_{12}$-PCB-111.[1] Recovery of the cleanup standards in the front half air samples were within the limits and ranged from 53-65% for $^{13}C_{12}$-PCB-81 and from 41-57% for $^{13}C_{12}$-PCB-111 indicating that analytes were well retained through the extract cleanup procedures. Recoveries of the pre-sampling surrogates in the back half air samples were also within the limits and ranged from 21-61% for $^{13}C_{12}$-PCB-81 and from 28-52% for $^{13}C_{12}$-PCB-111. The pre-sampling surrogate recoveries indicate how well analytes are retained from field sampling through laboratory analysis. While all the pre-sampling surrogate recoveries were within the target recovery range, the pre-sampling surrogate recoveries in Run 4 (21%, 28%) were approximately half of the recoveries for the surrogates in Runs 2 and 3 (51-61%). Since all internal standard recoveries for Runs 2, 3, and 4 were acceptable and similar between the three runs (indicating that laboratory extraction and cleanup were not a source of analyte loss), the low pre-sampling surrogate recovery in Run 4 suggests loss of this standard during sampling, handling, and/or transport to the laboratory prior to the sample extraction process or as the result of sample matrix interferences which resulted in poor recovery of internal standards. We have witnessed poor standard recoveries for internal standards for PCB, PAH, and D/F (0 to 30% range) in municipal waste combustion and medical waste combustion air matrix samples. The cause is unknown as yet. A discussion of what might have resulted in the lower Run 4 results is provided in Section 2.3. Since PCB concentrations are not corrected for pre-sampling surrogate recoveries, the lower PCB emission concentrations in Run 4 may be attributed to these possible analyte losses. If the concentrations for Runs 2, 3 and 4 are adjusted for the pre-sampling surrogate concentrations as shown in Table 6-7b, then the results

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[1]Method specified limits for the PCB labeled compound recoveries were selected as follows. A very limited method evaluation was undertaken prior to initiation of this test program. No additional body of recovery data for toxic PCBs in incinerator emissions using HRGC/HRMS techniques existed to augment the limited method evaluation results. The closest available data regarding the range of anticipated labeled compound recoveries was EPA Method 1668 for toxics PCBs in other matrices. Since much of the method sample cleanup was based on Method 1668, the estimate of reasonably achieved recovery ranges included in the method were the result of merging recoveries achieved in the method evaluation with the recovery range listed in Method 1668. The recovery ranges for internal standards in the method used (30-150%) are not out of line with recovery ranges for labeled internal standards in other EPA methods utilizing HRGC/HRMS techniques (e.g., Method 1668 = 21-178% and dioxin methods SW846 Method 8290 = 40-135%; EPA Method 1613 = 25-150%; EPA Method 23 = 40-130% for tetra through hexa chlorinated compounds and 25-130% for hepta-octachlorinated compounds). The cleanup and pre-sampling surrogate standard recovery ranges of 10-150% for $^{13}C_{12}$-PCB-81 and 20-130% for $^{13}C_{12}$-PCB-111 may appear on the surface to be significantly worse than pre-sampling surrogates in EPA Method 23 for dioxin (70-130%), however, there is a fundamental difference in that the Method 23 surrogates are quantified against the labeled internal standards added prior to extraction of the sample, and are therefore inherently corrected for any losses in extraction and/or cleanup, whereas the PCB surrogates are quantified against the recovery standard added at the end of cleanup, just before HRGC/HRMS analysis. Because this quantification relationship does not correct for any losses that may be experienced in extraction and cleanup, the recovery range for the PCB surrogate standard can be expected to be more in line with the recovery of other labeled internal standards as listed above.

Table 6-7b. Back Half Air Data Corrected for Pre-sampling Surrogate Recovery

ANALYTES	RUN 2 (pg/sample)	RUN 3 (pg/sample)	RUN 4 (pg/sample)	AVERAGE (pg/sample)	STANDARD DEVIATION	RSD (%)
PCB-77 E	276000	240000	193400	236467	41413	18
PCB-77	171000	117600	114200	134267	31857	24
PCB-123	1256	1172	930	1119	169	15
PCB-118	60800	56400	55800	57667	2730	5
PCB-114	4120	3640	3480	3747	333	9
PCB-105	28200	26200	23600	26000	2307	9
PCB-126	7660	6420	5660	6580	1010	15
PCB-167	4160	3640	3500	3767	348	9
PCB-156	6860	6080	5380	6107	740	12
PCB-157	2360	1932	2040	2111	223	11
PCB-169	6040	5140	3680	4953	1191	24
PCB-180	29000	26000	22400	25800	3305	13
PCB-170	11660	10540	11520	11240	610	5
PCB-189	1016	818	1110	981	149	15

E = Estimated value since calibration range exceeded.

between the three runs agree well within the <50% RSD precision QA/QC requirement for the analytes with actual RSDs ranging from 5-24%.

Because PCB-77 was detected significantly above the highest calibration level for Runs 2, 3 and 4 back half emission extracts (data flagged with "E" in Tables 6-7a and 6-7b), these extracts were diluted with additional internal standard in an effort to bring PCB-77 within the calibration range. These three back-half emission extracts were spiked with an additional 48,000 pg of $^{13}C_{12}$-PCB-77 to bring the total amount of $^{13}C_{12}$-PCB-77 added to the extracts to 50,000 pg. The extracts were then diluted to a final volume of 500 μL in order to bring the concentration of $^{13}C_{12}$-PCB-77 in the diluted extract to 100 pg/μL, which is the same concentration used in the calibration solutions. Diluting the extracts from 20 μL to 500 μL and adjusting the internal standard expands the calibration range for PCB-77 from the original 20 - 8,000 pg/sample to 500 - 200,000 pg/sample. Because the additional $^{13}C_{12}$-PCB-77 was added after-the-fact, the concentration of PCB-77 calculated against this is no longer inherently corrected for recovery of analyte through extraction and cleanup. As a result, the concentrations of PCB-77 detected in the diluted extracts were corrected for the original recovery of $^{13}C_{12}$-PCB-77 in the undiluted extract.

The PCB-77 concentrations determined in the diluted, recovery corrected extracts were within the calibration range and are flagged in Table 6-7a with an "*".

Lab Control Samples. Tables 6-8a and 6-8b present PCB results and standard recoveries for the laboratory control spike and laboratory control spike duplicate (LCS/LCSD) samples for front half and back half air samples. Recovery of all native PCBs spiked into the LCS/LCSD for both front and back half air samples were within the 70-130% target recovery range except for PCB-77 in the LCSD sample for back half airs (134%) and PCB-114 in all LCS/LCSD samples (146-167%). The relative percent difference (RPD) between duplicates were all under the target <30% and ranged from 0.1 to 6.3% for front half air samples and from 0.4 to 8.1% for back half air samples indicating acceptable analytical precision was achieved. Several analytes (PCB-123, PCB-114, and PCB-105) in both the front half and back half LCS/LCSD samples were detected above the calibration range and are flagged with an "E" in Tables 6-8a and 6-8b. These samples were not diluted and reanalyzed since the samples had been spiked intentionally with a level of these standards close to the upper range of the calibration curve, and with the exception of PCB-114, concentrations detected were within 20% of the upper calibration level. Internal standard recoveries for LCS/LCSD samples were all within the target 30-150% and ranged from 33-62% for front half LCD/LCSD samples and from 34-114% for back half air LCS/LCSD samples. Cleanup standard recoveries for front half air LCS/LCSD samples were 61-63% for $^{13}C_{12}$-PCB-81 (target recovery = 10-150%) and 42-45% for $^{13}C_{12}$-PCB-111(target recovery = 20-130%). There were no pre-sampling surrogates added to the LCS/LCSD for the back half air samples as only XAD which was shipped to the field received the pre-sampling surrogate standard.

Blanks. Table 6-9 lists PCB results and standard recoveries for the method blanks and the XAD background check sample used to verify cleanliness of XAD before shipping the sampling trains to the field. Table 6-10 lists PCB results and standard recoveries for field and proof blanks associated with the front half air samples. Table 6-11 lists PCB results and standard recoveries for field and proof blanks associated with the back half air samples. PCBs are quite prevalent in the environment and are commonly found in laboratory solvents and reagents making excessive background concentrations of PCBs common. The levels of PCBs detected in the various blanks are typical of background levels detected using the sensitive high resolution mass spectrometry techniques. Because PCBs are detected in the lab blank and because some samples did not contain high levels of PCBs above the background levels (such as the front half

Table 6-8a. Summary of PCB Results and Standard Recoveries for Front Half Lab Control Spike and Spike Duplicate Samples

ANALYTES	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/sample)	LAB BLANK 48190-11-10 FH-LB-PCB (pg/sample)	LCS 48190-11-08 FH-LCS-PCB (pg/sample)	RECOVERY (%)	LCS DUP 48190-11-09 FH-LCSDUP-PCB (pg/sample)	RECOVERY (%)	RECOVERY RPD (%)
PCB-77	70-130	400	71.6	1070	125	1040	121	3.9
PCB-123	70-130	20000	570	45400 E	112	45000 E	111	1.0
PCB-118	70-130	20000	2010	35100	83	35300	83	0.6
PCB-114	70-130	20000	767	59100 E	146 &	59000 E	146 &	0.1
PCB-105	70-130	20000	1400	46200 E	112	45700 E	111	1.2
PCB-126	70-130	2000	32.0	4700	117	4590	114	2.5
PCB-167	70-130	20000	466	45700	113	45700	113	0.1
PCB-156	70-130	20000	593 #	51200 #	127	51300 #	127	0.2
PCB-157	70-130	20000	489 #	49000 #	121	49100 #	122	0.1
PCB-169	70-130	4000	36.8	7990	99	7960	99	0.4
PCB-180	70-130	20000	37.3	45400	113	44900	111	1.3
PCB-170	70-130	4000	122	10000	123	9390	116	6.3
PCB-189	70-130	4000	53.1	8620	107	8670	108	0.5

INTERNAL STANDARDS	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/sample)	LAB BLANK 48190-11-10 FH-LB-PCB (pg/sample)	LCS 48190-11-08 FH-LCS-PCB (pg/sample)	RECOVERY (%)	LCS DUP 48190-11-09 FH-LCSDUP-PCB (pg/sample)	RECOVERY (%)	RECOVERY RPD (%)
13C-PCB-77	30-150	4000	1160	2360	59	2210	55	
13C-PCB-118	30-150	4000	352	1310	33	1340	33	
13C-PCB-105	30-150	4000	398	1420	36	1410	35	
13C-PCB-126	30-150	4000	910	1740	43	1830	46	
13C-PCB-167	30-150	4000	436	1340	33	1460	37	
13C-PCB-156	30-150	4000	676 #	2120 #	53	2480 #	62	
13C-PCB-157	30-150	4000	682 #	2000 #	50	2380 #	60	
13C-PCB-169	30-150	4000	1010	1710	43	1770	44	
13C-PCB-180	30-150	4000	938	1690	42	1820	46	
13C-PCB-189	30-150	4000	742	1850	46	1920	48	
13C-PCB-209	30-150	8000	1700	2870	36	3050	38	
CLEANUP STANDARDS								
13C-PCB-81	10-150	2000	572	1260	63	1220	61	
13C-PCB-111	20-130	10000	1590	4160	42	4460	45	

% Recovery is calculated as ((concentration found - lab blank sample conc.)/spike conc.) x 100.
& = QC value outside the target recovery goal for Method.
# = Value from second column confirmation.
RPD = Relative percent difference.
E = Result above calibration level.

Table 6-8b. Summary of PCB Results and Standard Recoveries for Back Half Lab Control Spike and Spike Duplicate Samples

ANALYTES	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/sample)	LCS 48190-09-09 BH-LCS-PCB (pg/sample)	RECOVERY (%)	LCS DUP 48190-09-10 BH-LCSDUP-PCB (pg/sample)	RECOVERY (%)	RECOVERY. RPD (%)	LAB BLANK 48190-09-11 BH-LB-PCB (pg/sample)	DL (pg/sample)
PCB-77	70-130	800	1060	127	1120	134 &	5.6	50.0	
PCB-123	70-130	40000	45000 E	112	46900 E	117	4.3	40.3	
PCB-118	70-130	40000	35500	86	36600	88	3.1	1230	
PCB-114	70-130	40000	61800 E	154 &	67000 E	167 &	8.1	81.7 E	
PCB-105	70-130	40000	47100 E	116	48300 E	119	2.3	573 E	
PCB-126	70-130	4000	4680	117	4750	119	1.4	4.25	
PCB-167	70-130	40000	44900	112	47500	119	5.5	50.4	
PCB-156	70-130	40000	47400	118	4970 #	124	4.7	95.6 #	
PCB-157	70-130	40000	45700	114	47100 #	118	3.1	32.3 #	
PCB-169	70-130	8000	7880	98	8100	101	2.8	4.50	
PCB-180	70-130	40000	44000	110	46600	116	5.6	181	
PCB-170	70-130	8000	9640	120	9600	119	0.4	77.1	
PCB-189	70-130	8000	8360	104	8080	108	3.7	6.01	

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	LCS 48190-09-09 BH-LCS-PCB (pg/sample)	RECOVERY (%)	LCS DUP 48190-09-10 BH-LCSDUP-PCB (pg/sample)	RECOVERY (%)	RECOVERY RPD (%)	LAB BLANK 48190-09-11 BH-LB-PCB (pg/sample)	DL (pg/sample)
13C-PCB-77	30-150	4000	1600	40	4550	114		2310	58
13C-PCB-118	30-150	4000	1360	34	1990	50		1470	37
13C-PCB-105	30-150	4000	1520	38	2180	54		1580	39
13C-PCB-126	30-150	4000	1660	42	2410	60		1720	43
13C-PCB-167	30-150	4000	1640	41	1980	50		1680	42
13C-PCB-156	30-150	4000	2490 #	62	2880 #	72		2220 #	55
13C-PCB-157	30-150	4000	2530 #	63	2870 #	72		2160 #	54
13C-PCB-169	30-150	4000	1830	46	2110	53		1680	42
13C-PCB-180	30-150	4000	1520	38	1870	47		1560	39
13C-PCB-189	30-150	4000	1660	41	1930	48		1570	39
13C-PCB-209	30-150	8000	3460	43	3820	48		3920	49
PRE-FIELD SURROGATE STANDARDS									
13C-PCB-81	10-150	2000	NA		NA			NA	
13C-PCB-111	20-130	10000	NA		NA			NA	

% Recovery is calculated as ((concentration found - lab blank sample conc.)/spike conc.) x 100.
& = QC value outside the target recovery goal for Method.
# = Value from second column confirmation.
DL = Detection limit.
NA = Not applicable.
E = Result above calibration range.

Table 6-9. Summary of PCB Results and Standard Recoveries for Background and Method Blank Samples

ANALYTES	METHOD BLANK 48200-51-03 (pg/sample)	DL (pg/sample)	XAD BCKGRD 48200-51-02 (pg/sample)	DL (pg/sample)
PCB-77	4.92		13.0	
PCB-123	ND	16.9	ND	19.7
PCB-118	134		301	
PCB-114	5.09		ND	6.02
PCB-105	67.0		139	
PCB-126	ND	2.32	ND	3.09
PCB-167	4.73		12.1	
PCB-156	11.4		27.6	
PCB-157	2.86		ND	2.77
PCB-169	(0.59)	1.28	ND	1.67
PCB-180	21.6		213	
PCB-170	ND	12.0	115	
PCB-189	ND	11.5	ND	15.0

INTERNAL STANDARDS	SPIKE CONC. (pg/sample)	METHOD BLANK 48200-51-03 (pg/sample)	RECOVERY (%)	XAD BCKGRD 48200-51-01 (pg/sample)	RECOVERY (%)
13C-PCB-77	4000	3560	189 &	2810	70
13C-PCB-118	4000	1780	44	1780	45
13C-PCB-105	4000	1600	40	1540	38
13C-PCB-126	4000	2210	55	2000	50
13C-PCB-167	4000	1960	49	1930	48
13C-PCB-156	4000	1720	43	1750	44
13C-PCB-157	4000	1620	40	1610	40
13C-PCB-169	4000	1720	43	1720	43
13C-PCB-180	4000	1290	32	1470	37
13C-PCB-189	4000	1380	35	1540	38
13C-PCB-209	8000	3610	42	3760	47
CLEANUP STANDARDS					
13C-PCB-81	2000	NA		NA	
13C-PCB-111	10000	NA		NA	

DL = Detection Limits; {EMPC}; (Below Detection Limit).
ND = Not detected, detection limit value provided in adjacent righthand side column.
NA = Not applicable.

Table 6-10. Summary of PCB Results and Standard Recoveries for Field Blank and Proof Blank Front Half Air Samples

ANALYTES	FIELD BLANK 1 48190-11-07 FH-FB1-PCB (pg/sample)	DL (pg/sample	PROOF BLANK 1 48190-11-05 FH-PB1-PCB (pg/sample)	DL (pg/sample)	PROOF BLANK 2 48190-11-06 FH-PB2-PCB (pg/sample)	DL (pg/sample)
PCB-77	67.8		77.6		186	
PCB-123	33.5		30.4		206	
PCB-118	1460		1310		5450	
PCB-114	61.3		53.6		321	
PCB-105	655		589		2630	
PCB-126	4.50		6.19		13.1	
PCB-167	37.6		40.9		208	
PCB-156	85.7 #		85.6		393 #	
PCB-157	17.8 #		17.5		154 #	
PCB-169	ND	2.13	ND	2.31	13.3	
PCB-180	163		186		555	
PCB-170	67.2		72.8		204	
PCB-189	ND	3.44	4.88		18.3	

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	FIELD BLANK 1 48190-11-07 FH-FB1-PCB (pg/sample)	RECOVERY (%)	PROOF BLANK 1 48190-11-05 FH-PB1-PCB (pg/sammple)	RECOVERY (%)	PROOF BLANK 2 48190-11-06 FH-PB2-PCB (pg/sample)	RECOVERY (%)
13C-PCB-77	30-150	4000	2580	64	2440	61	1820	46
13C-PCB-118	30-150	4000	1920	48	1680	42	1090	27 &
13C-PCB-105	30-150	4000	2060	51	1820	46	1180	29 &
13C-PCB-126	30-150	4000	2270	57	20650	51	1570	39
13C-PCB-167	30-150	4000	1920	48	1690	42	1140	29 &
13C-PCB-156	30-150	4000	2860 #	71	2640 #	66	2050 #	51
13C-PCB-157	30-150	4000	2690 #	67	2580 #	64	1830 #	46
13C-PCB-169	30-150	4000	2060	52	1930	48	1530	38
13C-PCB-180	30-150	4000	2210	55	1970	49	1510	38
13C-PCB-189	30-150	4000	2030	51	1930	48	1530	38
13C-PCB-209	30-150	8000	1930	24 &	2930	37	2020	25 &
CLEANUP STANDARDS								
13C-PCB-81	10-150	2000	1370	68	1240	62	930	46
13C-PCB-111	20-130	10000	5880	59	5160	52	3470	35

DL = Detection Limit, value specified only when analyte is not detected or else flagged {EMPC}; (Below Detection Limit).
ND = Not detected, detection limit value provided in adjacent righthand side column.
# = Value from second column confirmation.
& = QC value outside the target recovery goal for Method.

Table 6-11. Summary of PCB Results and Standard Recoveries for Field and Proof Blank Back Half Air Samples

ANALYTES	FIELD BLANK 1 48190-09-07 BH-FB1-PCB (pg/sample)	DL (pg/sample	PROOF BLANK 1 48190-09-05 BH-PB1-PCB (pg/sample)	DL (pg/sample	PROOF BLANK 2 48190-09-06 BH-PB2-PCB (pg/sample)	DL (pg/sample)
PCB-77	178		182		463	
PCB-123	72.0		32.1		167	
PCB-118	1780		1740		8010	
PCB-114	130		77.3		324	
PCB-105	873		835		3370	
PCB-126	16.2		11.9		12.4	
PCB-167	92.7		55.4		135	
PCB-156	155		123 #		316 #	
PCB-157	61.6		28.7		59.4 #	
PCB-169	7.51		{1.14}		3.07	
PCB-180	285		258		536	
PCB-170	116		121		200	
PCB-189	10.1		6.94		6.13	

INTERNAL STANDARDS	TARGET RANGE (%)	SPIKE CONC. (pg/sample)	FIELD BLANK 1 48190-09-07 BH-FB1-PCB (pg/sample)	RECOVERY (%) (pg/sample)	PROOF BLANK 1 48190-09-05 BH-PB1-PCB (pg/sample)	RECOVERY (%) (pg/sample)	PROOF BLANK 2 48190-09-06 BH-PB1-PCB (pg/sample)	RECOVERY (%) (pg/sample)
13C-PCB-77	30 - 150	4000	3140	79	2740	68	2150	54
13C-PCB-118	30 - 150	4000	1980	50	1860	47	1770	44
13C-PCB-105	30 - 150	4000	2120	53	1940	49	1960	49
13C-PCB-126	30 - 150	4000	2370	59	2430	61	2310	58
13C-PCB-167	30 - 150	4000	2280	57	2060	52	1900	48
13C-PCB-156	30 - 150	4000	2900 #	73	2760 #	69	2740 #	68
13C-PCB-157	30 - 150	4000	2820 #	70	2730 #	68	2810 #	70
13C-PCB-169	30 - 150	4000	2510	63	2500	62	2300	57
13C-PCB-180	30 - 150	4000	2200	55	2020	51	2090	52
13C-PCB-189	30 - 150	4000	2250	56	2200	55	2390	60
13C-PCB-209	30 - 150	8000	5170	65	4790	60	4950	62
PRE-SAMPLING SURROGATE STANDARDS								
13C-PCB-81	10 - 150	2000	1440	72	1310	65	964	48
13C-PCB-111	20 - 130	10000	4920	49	4610	46	4190	42

DL = Detection Limits; {EMPC}; (Below Detection Limit).
& = QC value outside the accuracy or precision goal for the Method (10% - 150% recovery of standard spiked samples).
# = Value from second column confirmation.

air samples), there are several instances where the lab blanks exceeded the method performance criteria of being at less than 20% of the sample concentration. Results for Runs 2, 3 and 4 are flagged with a "B" in Tables 6-6 and 6-7a and in result tables presented in Section 2 of this report if the lab blank concentration was greater than 20% of the reported sample concentration.

The target detection limit for these samples was 100 pg/sample. Actual detection limits achieved were significantly lower than this.

The target turnaround time for the PCB analysis of emission samples was to extract within 30 days of collection and analyze within 45 days of extraction. These turnaround times were met with extractions occurring within 11 days of collection and analysis occurring within 30 days of extraction.

D/F Analysis. The initial calibration met the requirement for response factors having less than 20% relative standard deviation (RSD) for native analytes and less than 35% RSD for labeled analytes (actual range = <13% for native analytes and <19% for labeled analytes). The continuing calibrations met the requirement for response factors being within 20% of the initial calibration response factors for native analytes and being within 30% of the initial calibration response factors for labeled analytes for all D/F but native OCDF. Native OCDF response factors in the middle and last of the three continuing calibrations which bracketed the emission sample analyses were higher than the 20% criteria which is derived from Method 8290. The emission samples were analyzed using Method 23 calibration and internal standard solutions (see QAPP amendment 9) and all but the OCDF response factor from the middle continuing calibration met the Method 23 objective of being within 30% of the initial calibration.

Field Samples. Summary tables of D/F results and standard recoveries for the front half and back half air samples are shown in Tables 6-12 and 6-13, respectively. Internal standard recoveries give an indication of how well analytes were extracted from the medium and retained during extract cleanup. For the front half air samples, recoveries of all internal standards were within the method specified limits of 40-135% and ranged from 54-98%. Internal standard recoveries in the back half air samples were also within the 40-135% limits and ranged from 47-95%. These internal standard recoveries indicate analytes were well recovered during laboratory extraction and were retained during the extract cleanup process. $^{37}Cl_4$-2,3,7,8- TCDD was added as a cleanup standard (after extraction, but before any cleanup procedures) in processing the front

Table 6-12. Summary of Dioxin/Furan Results and Standard Recoveries for Front Half Air Samples

ANALYTES	RUN 2 48190-11-02 FH-R2-DIOX (pg/sample)	DL (pg/sample)	RUN 3 48190-11-03 FH-R3-DIOX (pg/sample)	DL (pg/sample)	RUN 4 48190-11-04 FH-R4-DIOX (pg/sample)	DL (pg/sample)
2378-TCDD	6.08 #		ND #	2.98	ND #	4.49
12378-PeCDD	ND	15.0	ND	15.2	ND	17.8
123478-HxCDD	5.44		ND	5.10	ND	5.49
123678-HxCDD	13.7		9.58		10.3	
123789-HxCDD	24.9		ND	3.11	16.0	
1234678-HpCDD	205		138		127	
OCDD	689		429		404	
2378-TCDF	108 #		55.6 #		85.8 #	
12378-PeCDF	23.4		14.2		16.7	
23478-PeCDF	79.1		34.1		47.2	
123478-HxCDF	76.1		32.3		41	
123678-HxCDF	29.6		16.0		18.0	
123789-HxCDF	ND	6.32	ND	6.28	ND	7.17
234678-HxCDF	75.4		37.1		40.3	
1234678-HpCDF	156		83.9		90.4	
1234789-HpCDF	20.6		(7.65) J	9.77	(10.4) J	11.0
OCDF	129		102		82.1	
Total Tetra-Furans	698		395		594	
Total Tetra-Dioxins	324		181		142	
Total Penta-Furan	624		263		380	
Total Penta-Dioxins	69.9		21.3		15.9	20.2
Total Hexa-Furans	360		136		194	
Total Hexa-Dioxins	239		140		182	
Total Hepta-Furans	244		91.5		98.5	
Total Hepta-Dioxins	431		314		296	
Total TEQ	98.4		38.6		52.5	

Table 6-12. (Continued)

ANALYTES	TARGET RANGE (%)	SPIKE CONC. (pg/sample)	RUN 2 48190-11-02 FH-R2-DIOX (pg/sample)	RECOVERY (%)	RUN 3 48190-11-03 FH-R3-DIOX (pg/sample)	RECOVERY (%)	RUN 4 48190-11-04 FH-R4-DIOX (pg/sample)	RECOVERY (%)
INTERNAL STANDARDS								
13C-2378-TCDD	40 - 135	8000	5580 #	70	6010 #	75	4510 #	56
13C-12378-PeCDD	40 - 135	8000	7640	95	7870	98	6780	85
13C-123678-HxCDD	40 - 135	8000	5980	75	6060	76	6060	76
13C-1234678-HpCDD	40 - 135	8000	6660	83	6850	86	7160	90
13C-OCDD	40 - 135	16000	13000	81	14600	91	15500	97
13C-2378-TCDF	40 - 135	8000	5170 #	65	5540 #	69	4300 #	54
12378-PeCDF	40 - 135	8000	6760	84	6920	87	5710	71
123678-HxCDF	40 - 135	8000	5240	65	5400	67	5070	63
1234678-HpCDF	40 - 135	8000	6340	79	6484	81	6370	80
CLEANUP STANDARDS								
37Cl-2378-TCDD	40 - 135	800	820 #	103	800 #	100	796 #	100

DL = Detection Limits; {EMPC}; (Below Detection Limit).
# = Value from second column confirmation.
ND = Not detected.
J = Value found below the detection limit

Table 6-13. Summary of Dioxin/Furan Results and Standard Recoveries for Back Half Air Samples

ANALYTES	RUN 2 48190-09-02 BH-R2-DIOX (pg/sample)	DL (pg/sample)	RUN 3 48190-09-03 BH-R3-DIOX (pg/sample)	DL (pg/sample)	RUN 4 48190-09-04 BH-R4-DIOX (pg/sample)	DL (pg/sample)
2378-TCDD	565 #		418 #		225 #	
12378-PeCDD	97.9		78.4		33.2	
123478-HxCDD	81.5		90.1		38.4	
123678-HxCDD	209		235		76.1	
123789-HxCDD	{203}		217		106	
1234678-HpCDD	991		1120		496	
OCDD	1160		1460		534	
2378-TCDF	9150 #		7630 #		3490 #	
12378-PeCDF	1110		920		433	
23478-PeCDF	2190		1720		775	
123478-HxCDF	1240		1070		575	
123678-HxCDF	447		396		213	
123789-HxCDF	ND	12.2	ND	14.1	ND	15.7
234678-HxCDF	661		563		303	
1234678-HpCDF	1150		1060		593	
1234789-HpCDF	109		99.8		46.4	
OCDF	397		412		197	
Total Tetra-Furans	32100		30000		16600	
Total Tetra-Dioxins	17400		21800		4680	
Total Penta-Furan	28200		22500		9910	
Total Penta-Dioxins	4060		4070		1240	
Total Hexa-Furans	6220		5330		2550	
Total Hexa-Dioxins	3300		3490		1990	
Total Hepta-Furans	1680		1530		815	
Total Hepta-Dioxins	2250		2500		1290	
Total TEQ	2530		2080		983	

Table 6-13. (Continued)

ANALYTES	TARGET RANGE (%)	SPIKE CONC. (pg/sample)	RUN 2 48190-09-02 BH-R2-DIOX (pg/sample)	RECOVERY %	RUN 3 48190-09-03 BH-R3-DIOX (pg/sample)	RECOVERY (%)	RUN 4 48190-09-04 BH-R4-DIOX (pg/sample)	RECOVERY (%)
INTERNAL STANDARDS								
13C-2378-TCDD	40 - 135	8000	4960 #	62	4420 #	55	4460 #	56
13C-12378-PeCDD	40 - 135	8000	6190	77	6220	78	5880	73
13C-123678-HxCDD	40 - 135	8000	5510	69	5820	73	5490	69
13C-1234678-HpCDD	40 - 135	8000	5970	75	6580	82	6160	77
13C-OCDD	40 - 135	16000	14100	88	15100	95	14300	90
13C-2378-TCDF	40 - 135	8000	3840 #	48	3740 #	47	3970 #	50
12378-PeCDF	40 - 135	8000	5600	70	5730	72	5340	67
123678-HxCDF	40 - 135	8000	4880	61	5380	67	4960	62
1234678-HpCDF	40 - 135	8000	5730	72	6250	78	5640	70
PRE-SAMPLING SURROGATE STANDARDS								
37Cl-2378-TCDD	70 - 130	8000	8440 #	106	8740 #	109	3840 #	48 &
13C-123478-HxCDD	70 - 130	8000	8320	104	9730	122	4280	53 &
13C-23478-PeCDF	70 - 130	8000	7760	97	8490	106	3620	45 &
13C-123478-HxCDF	70 - 130	8000	7840	98	8530	107	4110	51 &
13C-1234789-HpCDF	70 - 130	8000	6060	76	7610	95	3460	43 &

DL = Detection Limits; {EMPC}; (Below Detection Limit).
ND = Not detected.
# = Value from second column confirmation.

half air samples. Recovery of this standard in the front half air samples ranged from 100-103% indicating analytes were well retained in the cleanup process. $^{37}Cl_4$-2,3,7,8- TCDD, $^{13}C_{12}$-1,2,3,4,7,8-HxCDD, $^{13}C_{12}$ -2,3,4,7,8-PeCDF, $^{13}C_{12}$ -1,2,3,4,7,8-HxCDF, and $^{13}C_{12}$ -1,2,3,4,7,8,9-HpCDF were added as pre-sampling surrogate standards spiked into XAD resin before shipping to the field in the back half air samples. The specified recovery ranges for these standards were from 70-130%. Recoveries of the pre-sampling surrogates in the back half air samples were within the 70-130% limits for Run 2 and Run 3 (76-122%), but were uniformly low for Run 4 (43-53%). Since all internal standard recoveries for Runs 2, 3, and 4 were acceptable and similar between the three runs (indicating that laboratory extraction and cleanup were not a source of analyte loss), the low pre-sampling surrogate recovery for D/F in Run 4 suggests loss of this standard during sampling, handling, and/or transport to the laboratory prior to the sample extraction process. In addition, the same pattern was seen in the back half PCB samples with Runs 2 and 3 being comparable and Run 4 showing a drop in both analyte concentrations and pre-sampling surrogate recoveries, while PCB internal standard recoveries were comparable among all three runs. Since the D/F and PCB pre-sampling surrogate standard additions to XAD were separate standard spikes, this eliminates the possibility of an error in spiking the XAD before going to the field. Since the D/F and PCB sample fractions went through separate cleanup procedures, cleanup activities can also be eliminated as causing the loss of analytes. As with the PCB pre-sampling surrogate recoveries for Run 4, D/F pre-sampling surrogate recoveries for Run 4 were approximately half that obtained in Runs 2 and 3. It is anticipated that if back half D/F analyte concentrations were corrected for the recovery of the pre-sampling surrogates (as in the example for PCBs in Table 6-7b) that acceptable precision among the replicate runs would be obtained.

Lab Control Samples. Tables 6-14a and 6-14b present a summary of D/F results and standard recoveries for LCS/LCSD front half and back half air samples, respectively. The recovery objective for native analytes in the LCS/LCSD samples was 40-135%. The actual range for the front and back half control spikes was 79-135% except for 2,3,4,6,7,8-HxCDF which was recovered high in both the LCS and LCSD for the front half (138% in both), OCDF which was recovered high in the LCSD for both the front half and back half (136% and 143%, respectively), and 1,2,3,7,8,9-HxCDF which was recovered high on both the front half and back half LCSD (141% and 147%, respectively). The RPD between analyte concentrations found in

Table 6-14a. Summary of Dioxin/Furan Results and Standard Recoveries for Front-Half Lab Control Spike and Spike Duplicate Samples

ANALYTES	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/sample)	LAB BLANK 48190-11-10 FH-LB-DIOX (pg/sample)	DL (pg/sample)	LCS 48190-11-08 FH-LCS-DIOX (pg/sample)	RECOVERY (%)	LCS DUP 48190-11-09 FH-LCSDUP-DIOX (pg/sample)	RECOVERY (%)	RECOVERY (RPD)
2378-TCDD	40 - 135	800	ND #	3.75	930	116	796	99	16
12378-PeCDD	40 - 135	4000	ND	17.6	4140	104	3940	99	5
123478-HxCDD	40 - 135	4000	ND	5.94	3860	97	3830	96	0.7
123678-HxCDD	40 - 135	4000	ND	5.51	4100	103	4110	103	0.3
123789-HxCDD	40 - 135	4000	ND	3.63	4270	107	3960	99	7
1234678-HpCDD	40 - 135	4000	ND	4.94	4060	102	3900	97	4
OCDD	40 - 135	8000	31.9		8290	103	7870	98	5
2378-TCDF	40 - 135	800	ND #	6.13	933	117	851	106	9
12378-PeCDF	40 - 135	4000	ND	8.70	4450	111	4300	108	3
23478-PeCDF	40 - 135	4000	ND	7.26	4760	119	3170	79	40
123478-HxCDF	40 - 135	4000	ND	7.38	4690	117	4710	118	0.4
123678-HxCDF	40 - 135	4000	ND	6.44	4530	113	4480	112	1
123789-HxCDF	40 - 135	4000	ND	7.44	5420	135	5650	141 &	4
234678-HxCDF	40 - 135	4000	ND	7.13	5540	138 &	5520	138 &	0.3
1234678-HpCDF	40 - 135	4000	ND	2.63	4210	105	4160	104	1
1234789-HpCDF	40 - 135	4000	ND	11.7	4600	115	5120	128	11
OCDF	40 - 135	8000	ND	20.2	10300	128	10800	136 &	5.6
Total Tetra-Furans			ND	10.7	933		851		
Total Tetra-Dioxins			ND	14.9	930		796		
Total Penta-Furan			ND	14.8	9210		7470		
Total Penta-Dioxins			ND	20.1	4140		3940		
Total Hexa-Furans			ND	14.1	20200		20400		
Total Hexa-Dioxins			ND	8.95	12200		11900		
Total Hepta-Furans			ND	21.5	8810		9280		
Total Hepta-Dioxins			ND	22.5	4060		3900		
Total TEQ			0.032		9080		8030		

Table 6-14a. (Continued)

INTERNAL STANDARDS	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/sample)	LAB BLANK 48190-11-10 FH-LB-DIOX (pg/sample)	DL (pg/sample)	LCS 48190-11-08 FH-LCS-DIOX (pg/sample)	RECOVERY (%)	LCS DUP 48190-11-09 FH-LCSDUP-DIOX (pg/sample)	RECOVERY (%)	RECOVERY RPD (%)
13C-2378-TCDD	40 - 135	8000	5110 #	64	4270	53	5910	74	
13C-12378-PeCDD	40 - 135	8000	6970	87	7240	91	7070	88	
13C-123678-HxCDD	40 - 135	8000	5820	73	5830	73	5980	75	
13C-1234678-HpCDD	40 - 135	8000	6500	81	7050	88	7570	95	
13C-OCDD	40 - 135	16000	13800	86	15300	95	16900	106	
13C-2378-TCDF	40 - 135	8000	4930 #	62	3980	50	5040	63	
13C-12378-PeCDF	40 - 135	8000	6010	75	5510	69	5900	74	
13C-123678-HxCDF	40 - 135	8000	5140	64	4850	61	4940	62	
13C-1234678-HpCDF	40 - 135	8000	5990	75	6380	80	6710	84	
CLEANUP STANDARDS									
37Cl-2378-TCDD	40 - 135	800	810 #	101	820	102	660	83	

DL = Detectionlimit.
ND = Not detected.
% Recovery is calculated as ((concentration found - blank field sample concen.)/spike conc.) x 100.
& = QC value outside the accuracy or precision goal for Method (40-135% Recovery of native compounds in spike samples).
# = Value from second column confirmation.

Table 6-14b. Summary of Dioxin/Furan Results and Standard Recoveries for Back-Half Lab Control Spike and Spike Duplicate Samples

ANALYTES	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/sample)	LAB BLANK 48190-09-11 BH-LB-DIOX (pg/sample)	DL (pg/sample	LCS 48190-09-09 BH-LCS-DIOX (pg/sample)	RECOVERY (%)	LCS DUP 48190-09-09 BH-LCSDUP-DIOX (pg/sample)	RECOVERY (%)	RECOVERY (RPD)
2378-TCDD	40 - 135	800	(1.50) #,J	1.81	839	105	869	109	4
12378-PeCDD	40 - 135	4000	ND	10.1	4040	101	4180	104	3
123478-HxCDD	40 - 135	4000	ND	3.19	3850	96	3920	98	2
123678-HxCDD	40 - 135	4000	ND	2.94	3870	97	4190	105	8
123789-HxCDD	40 - 135	4000	ND	1.94	4070	102	4100	102	0
1234678-HpCDD	40 - 135	4000	{0.319}		3910	98	4100	102	4
OCDD	40 - 135	8000	24.5		7900	99	8260	103	4
2378-TCDF	40 - 135	800	5.13 #		864	105	906	110	5
12378-PeCDF	40 - 135	4000	ND	5.00	4170	104	4430	111	7
23478-PeCDF	40 - 135	4000	ND	4.19	4140	104	4530	113	9
123478-HxCDF	40 - 135	4000	ND	4.07	4550	114	4840	121	7
123678-HxCDF	40 - 135	4000	ND	3.50	4340	109	4540	114	5
123789-HxCDF	40 - 135	4000	ND	4.07	5200	130	5870	147 &	17
234678-HxCDF	40 - 135	4000	ND	3.94	1510	128	5350	134	6
1234678-HpCDF	40 - 135	4000	3.38		3920	98	4190	105	7
1234789-HpCDF	40 - 135	4000	ND	5.76	4590	115	4610	115	0
OCDF	40 - 135	8000	(7.63) J	9.95	9610	120	11500	143 &	18
Total Tetra-Furans			4.44		884		906		
Total Tetra-Dioxins			12.39		839		869		
Total Penta-Furan			ND	8.45	8320		8960		
Total Penta-Dioxins			ND	11.4	4040		4180		
Total Hexa-Furans			3.00		19200		20600		
Total Hexa-Dioxins			ND	4.82	11800		12200		
Total Hepta-Furans			8.51		8510		8800		
Total Hepta-Dioxins			{22.2}		3910		4090		
Total TEQ			0.544		8470		8960		

Table 6-14b. (Continued)

INTERNAL STANDARDS	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/sample)	LAB BLANK 48190-09-11 BH-LB-DIOX (pg/sample)	RECOVERY (%)	LCS 48190-09-09 BH-LCS-DIOX (pg/sample)	RECOVERY (%)	LCS DUP 48190-09-09 BH-LCSDUP-DIOX (pg/sample)	RECOVERY (%)	RECOVERY (RPD)
13C-2378-TCDD	40 - 135	8000	4910 #	61	5720	72	5360	67	
13C-12378-PeCDD	40 - 135	8000	6280	78	7550	94	6820	85	
13C-123678-HxCDD	40 - 135	8000	5120	64	5720	71	5590	70	
13C-1234678-HpCDD	40 - 135	8000	6040	76	7100	89	7120	89	
13C-OCDD	40 - 135	16000	14100	88	17000	106	16800	105	
13C-2378-TCDF	40 - 135	8000	4660 #	58	5260	66	4960	62	
13C-12378-PeCDF	40 - 135	8000	5560	70	6320	79	5600	70	
13C-123678-HxCDF	40 - 135	8000	4400	55	4880	61	4720	59	
13C-1234678-HpCDF	40 - 135	8000	5800	73	6510	81	6400	80	
PRE-SAMPLING SURROGATE STANDARDS									
37Cl-2378-TCDD	40 - 135	800	4	0.5	NA		NA		

DL = Detection limit.
% Recovery is calculated as ((concentration found - blank field sample concen.)/spike conc.) x 100.
J = Estimated value, below detection limit.
& = QC value outside the accuracy or precision goal for Method (40-135% Recovery of native compounds in spike samples).
# = Value from second column confirmation.
ND = Not detected.

the LCS and LCSD in the front half samples met the data quality objective of <20% (actual values ranged from 0.3 to 16%) except for 2,3,4,7,8-PeCDF with an RPD of 40%. The RPD between the LCS and LCSD for back half samples met the data quality objective of <20% for all analytes and ranged from 0 -18%. Recoveries of internal standards and the cleanup standard associated with the front half LCS/LCSD samples were all within the 40-135% target and ranged from 50-106%. Recoveries of internal standards added to the back half LCS/LCSD samples were also within the 40-135% range and ranged from 59-106%. Pre-sampling surrogate standards were not added to the back half LCS/LCSD samples as only XAD which went to the field received this standard. The internal and cleanup standard recoveries indicate that analytes were efficiently extracted from the air sampling media and were retained during the laboratory cleanup process.

Blanks. Table 6-15 lists D/F results and standard recoveries for all lab blanks and the XAD background check sample used to verify cleanliness of XAD before shipping the sampling trains to the field. Table 6-16 lists D/F results and standard recoveries for field and proof blanks associated with the front half air samples. Table 6-17 lists D/F results and standard recoveries for field and proof blanks associated with the back half air samples. Very few D/F congeners were found in the blanks and those which were detected were at levels below the lowest calibration level (40 pg/sample for tetra compounds, 200 pg/sample for penta-hepta compounds, and 400 pg/sample for octa compounds). Recoveries of internal standards and the cleanup standard in the lab blanks were within 40-135% and ranged from 55-101%. Recoveries of internal standards and the cleanup standard in the front half field and proof blanks were within 40-135% and ranged from 40-101%. Recoveries of internal standards in the back half field and proof blanks were within 40-135% and ranged from 61-89%. Pre-sampling surrogate recoveries in the back half field and proof blank samples were all within the 70-130% limit and ranged from 91-127%.

The target detection limit for these samples, based on the lowest calibration level was 40 pg/sample for tetra compounds, 200 pg/sample for penta-hepta compounds and 400 pg/sample for octa compounds. Actual detection limits achieved were significantly lower than this.

The target turnaround time for the D/F analysis of emission samples was to extract within 30 days of collection and analyze within 45 days of extraction. These turnaround times were met

Table 6-15. Summary of Dioxin/Furan Results and Standard Recoveries for Lab and Method Blank Samples

ANALYTES	LAB BLANK 48190-11-10 FH-LB-DIOX (pg/sample)	DL (pg/sample)	LAB BLANK 48190-09-11 BH-LB-DIOX (pg/sample)	DL (pg/sample)	METHOD BLANK 48200-51-03 (pg/sample)	DL (pg/sample)	XAD BCKGRD 48200-51-02 (pg/sample)	DL (pg/sample)
2378-TCDD	ND #	3.75	(1.50) #,J	1.81	ND	1.74	ND	2.46
12378-PeCDD	ND	17.6	ND	10.1	ND	4.60	ND	7.80
123478-HxCDD	ND	5.94	ND	3.19	ND	2.35	ND	2.93
123678-HxCDD	ND	5.51	ND	2.94	ND	2.71	ND	3.30
123789-HxCDD	ND	3.63	ND	1.94	ND	3.25	ND	4.01
1234678-HpCDD	ND	4.94	{0.319}		ND	1.68	(1.42) J	1.91
OCDD	31.9		24.5		3.38		(3.09) J	3.66
2378-TCDF	ND #	6.13	5.13 #		ND	2.58	ND	3.49
12378-PeCDF	ND	8.70	ND	5.00	ND	1.53	ND	2.08
23478-PeCDF	ND	7.26	ND	4.19	ND	2.62	ND	3.61
123478-HxCDF	ND	7.38	ND	4.07	ND	1.80	ND	2.25
123678-HxCDF	ND	6.44	ND	3.50	ND	1.77	ND	2.20
123789-HxCDF	ND	7.44	ND	4.09	ND	1.55	ND	1.79
234678-HxCDF	ND	7.13	ND	3.94	ND	1.96	ND	2.43
1234678-HpCDF	ND	2.63	3.38		ND	1.53	ND	1.77
1234789-HpCDF	ND	11.7	ND	5.76	ND	2.74	ND	3.05
OCDF	ND	20.21	(7.63) J	9.95	ND	7.56	ND	8.62
Total Tetra-Furans	ND	10.7	4.44		(5.22) J	11.0	(0.95) J	14.9
Total Tetra-Dioxins	ND	14.9	12.39		(0.13) J	4.38	(0.31) J	6.20
Total Penta-Furan	ND	14.8	ND	8.45	ND	3.24	(0.24) J	4.46
Total Penta-Dioxins	ND	20.1	ND	11.4	ND	47.1	ND	79.9
Total Hexa-Furans	ND	14.1	3.00		(0.60) J	3.98	(0.17) J	4.87
Total Hexa-Dioxins	ND	8.75	ND	4.82	ND	3.84	ND	4.73
Total Hepta-Furans	ND	21.5	8.51		ND	4.94	(0.49) J	5.60
Total Hepta-Dioxins	ND	22.5	{22.2}		(0.64) J	5.68	(1.52) J	4.98
Total TEQ	0.032		0.544		NA			

Table 6-15. (Continued)

INTERNAL STANDARDS	SPIKE CONC. (pg/sample)	LAB BLANK 48190-11-10 FH-LB-DIOX (pg/sample)	RECOVERY (%)	LAB BLANK 48190-09-11 BH-LB-DIOX (pg/sample)	RECOVERY (%)	METHOD BLANK 48200-51-09 (pg/sample)	RECOVERY (%)	XADBCKGRD 48200-51-02 (pg/sample)	RECOVERY (%)
13C-2378-TCDD	8000	5110 #	64	4910 #	61	6720	84	6920	86
13C-12378-PeCDD	8000	6940	87	6270	78	8480	106	8090	101
13C-123478-HxCDD	8000	NA	NA	NA	NA	6370	80	6630	83
13C-123678-HxCDD	8000	5820	73	5110	64	6400	80	6500	81
13C-1234678-HpCDD	8000	6500	81	6050	76	6180	77	6870	86
13C-OCDD	16000	3800	86	14100	88	10300	64	2000	75
13C-2378-TCDF	8000	4930 #	62	4660 #	58	7080	88	7280	91
13C-12378-PeCDF	8000	6010	75	5570	70	7680	96	8320	104
13C-23478-PeCDF	8000	NA	NA	NA	NA	9160	115	9190	115
13C-123478-HxCDF	8000	NA	NA	NA	NA	6960	87	7070	88
13C-123678-HxCDF	8000	5140	64	4410	55	6680	84	6940	87
13C-123789-HxCDF	8000	NA	NA	NA	NA	6180	77	6830	85
13C-234678-HxCDF	8000	NA	NA	NA	NA	7230	90	7460	93
13C-1234678-HpCDF	8000	5990	75	5810	73	6520	81	7300	91
13C-1234789-HpCDF	8000	NA	NA	NA	NA	4990	62	5600	70
CLEANUP STANDARDS/PRESAMPLING SURROGATE STANDARDS									
37Cl-2378-TCDD	800	808 #	101	8	0	NA	NA	NA	NA
13C-123478-HxCDD	800	NA	NA	NA	NA	NA	NA	NA	NA
13C-23478-PeCDF	800	NA	NA	NA	NA	NA	NA	NA	NA
13C-123478-HxCDF	800	NA	NA	NA	NA	NA	NA	NA	NA
13C-1234789-HpCDF	800	NA	NA	NA	NA	NA	NA	NA	NA

DL = Detection Limits; {EMPC}; (Below Detection Limit).
# = Value from second column confirmation.
J = Value found below detection limit.
ND = Not detected.
NA = Not applicable.

Table 6-16. Summary of Dioxin/Furan Results and Standard Recoveries for Field Blank and Proof Blank Front Half Air Samples

ANALYTES	FIELD BLANK 1 48190-11-07 FH-FB1-DIOX (pg/sample)	DL (pg/sample)	PROOF BLANK 1 48190-11-05 FH-PB1-DIOX (pg/sample)	DL (pg/sample)	PROOF BLANK 2 48190-11-06 FH-PB2-DIOX (pg/sample)	DL (pg/sample)
2378-TCDD	ND	10.6	ND	6.38	ND	12.6
12378-PeCDD	ND	19.5	ND	10.95	ND	18.9
123478-HxCDD	ND	6.07	ND	3.94	ND	5.32
123678-HxCDD	ND	5.69	ND	3.69	ND	4.94
123789-HxCDD	ND	3.75	ND	2.44	ND	3.25
1234678-HpCDD	ND	4.57	ND	3.00	ND	4.07
OCDD	23.3		21.5		20.1	
2378-TCDF	ND	6.63	ND	3.94	ND	7.38
12378-PeCDF	ND	10.9	ND	6.26	ND	10.2
23478-PeCDF	ND	9.20	ND	5.26	ND	8.51
123478-HxCDF	ND	7.82	ND	5.00	ND	7.13
123678-HxCDF	ND	6.82	ND	4.32	ND	6.19
123789-HxCDF	ND	7.88	ND	5.00	ND	7.19
234678-HxCDF	ND	7.57	ND	4.82	ND	6.94
1234678-HpCDF	ND	2.56	3.75		ND	2.31
1234789-HpCDF	ND	11.2	ND	7.57	ND	10.1
OCDF	ND	19.3	ND	13.1	ND	17.4
Total Tetra-Furans	ND	13.5	14.8		ND	15.0
Total Tetra-Dioxins	ND	18.4	ND	11.1	ND	21.9
Total Penta-Furan	ND	18.6	ND	10.6	ND	17.3
Total Penta-Dioxins	ND	22.2	ND	12.5	ND	21.5
Total Hexa-Furans	ND	14.9	ND	9.51	ND	13.6
Total Hexa-Dioxins	ND	9.2	ND	5.94	ND	8.01
Total Hepta-Furans	ND	3.13	(3.75) J	13.9	ND	18.5
Total Hepta-Dioxins	ND	20.8	(5.07) J	13.8	ND	18.6
Total TEQ	0.023		0.059		0.020	

Table 6-16. (Continued)

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	FIELD BLANK 1 48190-11-07 FH-FB1-DIOX (pg/sample)	RECOVERY (%)	PROOF BLANK 1 48190-11-05 FH-PB1-DIOX (pg/sample)	RECOVERY (%)	PROOF BLANK 2 48190-11-06 FH-PB2-DIOX (pg/sample)	RECOVERY (%)
13C-2378-TCDD	40 - 135	8000	4380	55	5380	67	3190	40
13C-12378-PeCDD	40 - 135	8000	6440	81	7620	95	5450	68
13C-123678-HxCDD	40 - 135	8000	5480	69	6150	77	5150	64
13C-1234678-HpCDD	40 - 135	8000	6690	84	7360	92	6350	79
13C-OCDD	40 - 135	16000	14200	89	15800	99	13600	85
13C-2378-TCDF	40 - 135	8000	4280	53	5120	64	3170	40
13C-12378-PeCDF	40 - 135	8000	5190	65	6330	79	4450	56
13C-123678-HxCDF	40 - 135	8000	4560	57	5290	66	4270	53
13C-1234678-HpCDF	40 - 135	8000	6030	75	6690	84	5700	71
CLEANUP STANDARDS								
37Cl-2378-TCDD	40 - 135	800	804	101	768	96	792	99

ND = Not detected.
DL = Detection Limit.
1J = Below detection limit.

Table 6-17. Summary of Dioxin/Furan Results and Standard Recoveries for Field Blank and Proof Blank Back Half Air Samples

ANALYTES	FIELD BLANK 1 48190-09-07 BH-FB1-DIOX (pg/sample)	DL (pg/sample)	PROOF BLANK 1 48190-09-05 BH-PB1-DIOX (pg/sample)	DL (pg/sample)	PROOF BLANK 2 48190-09-06 BH-PB2-DIOX (pg/sample)	DL (pg/sample)
2378-TCDD	ND	6.76	ND #	2.96	ND #	2.75
12378-PeCDD	ND	16.0	ND	16.4		16.2
123478-HxCDD	ND	5.52	ND	5.32		5.20
123678-HxCDD	ND	5.12	ND	4.96		4.88
123789-HxCDD	ND	3.36	ND	3.24		3.20
1234678-HpCDD	{11.9}		14.3		{6.80}	
OCDD	54.0		73.6		{34.0}	
2378-TCDF	4.68		(4.08) #	4.88	14.3 #	
12378-PeCDF	ND	8.08	ND	8.12		8.12
23478-PeCDF	ND	6.76	ND	6.80		6.80
123478-HxCDF	ND	6.20	ND	6.64		6.64
123678-HxCDF	ND	5.40	ND	5.76		5.80
123789-HxCDF	ND	6.24	ND	6.64		6.68
234678-HxCDF	ND	6.00	ND	6.40		6.44
1234678-HpCDF	9.76		11.1		5.76	
1234789-HpCDF	ND	11.0	ND	10.5		10.8
OCDF	ND	19.0	14.0 J	18.1	9.56 J	18.6
Total Tetra-Furans	(4.68) J	9.00	6.64 J	9.40	73.2	
Total Tetra-Dioxins	ND	11.8		12.1	41.2	
Total Penta-Furan	ND	13.6		13.8	14.7	
Total Penta-Dioxins	ND	18.2		18.6	4.80 J	18.4
Total Hexa-Furans	(5.44) J	11.8		12.6	6.40 J	12.7
Total Hexa-Dioxins	ND	8.32	5.96 J	8.00		7.88
Total Hepta-Furans	19.7		19.6		14.3	
Total Hepta-Dioxins	(11.9) J	21.2	24.8		14.8	
Total TEQ	0.74		1.00		2.2	

Table 6-17. (Continued)

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	FIELD BLANK 1 48190-09-07 BH-FB1-DIOX (pg/sample)	RECOVERY (%)	PROOF BLANK 1 48190-09-05 BH-PB1-DIOX (pg/sample)	RECOVERY (%)	PROOF BLANK 2 48190-09-06 BH-PB2-DIOX (pg/sample)	RECOVERY (%)
13C-2378-TCDD	40 - 135	8000	6000	75	5160 #	65	5280 #	66
13C-12378-PeCDD	40 - 135	8000	7080	89	6320	79	6680	83
13C-123678-HxCDD	40 - 135	8000	6080	76	5400	67	6000	75
13C-1234678-HpCDD	40 - 135	8000	6200	78	6040	75	6240	78
13C-OCDD	40 - 135	16000	13600	85	13400	84	13900	87
13C-2378-TCDF	40 - 135	8000	5560	70	4840 #	61	4880 #	61
13C-12378-PeCDF	40 - 135	8000	6200	78	5640	71	5760	72
13C-123678-HxCDF	40 - 135	8000	5680	71	4880	61	5120	64
13C-1234678-HpCDF	40 - 135	8000	5800	73	5440	68	5600	70
PRE-SAMPLING SURROGATE STANDARDS								
37Cl-2378-TCDD	70 - 130	8000	8240	103	9040 #	113	8920 #	112
13C-123478-HxCDD	70 - 130	8000	8960	112	10200	127	9040	113
13C-23478-PeCDF	70 - 130	8000	9360	117	10200	127	10000	125
13C-123478-HxCDF	70 - 130	8000	8080	101	9440	118	9240	115
13C-1234789-HpCDF	70 - 130	8000	7280	91	10000	126	9640	121

DL = Detection Limits; {EMPC}.
ND = No data.
# = Value from second column confirmation.
J = Below detection limit.

with extractions occurring within 13 days of collection and analysis occurring within 15 days of extraction.

PAH Analysis.

Field Samples. The measured native PAH data and recovery data of internal standards, surrogate standards, and recovery standards in the front half (FH) and back half (BH) air samples are summarized in Tables 6-18 and 6-19. The recovery standards (acenaphthene-d_{10} and pyrene-d_{10}) were added to the sample extracts after completing the sample preparation of cleanup steps. Acceptable recoveries (64-124%) for the recovery standards were established in the FH and BH samples. These data suggest that there were no significant sample matrix effects on the native acenaphthene and pyrene in the resulting sample extracts for GC/MS analysis. However, the possible sample matrix effect on other relatively more reactive target PAH compounds cannot be discounted.

Quantitative recoveries (>70%) of all the internal standards except benzo[a]pyrene-d_{12} and perylene-d_{12} were obtained in the FH samples. Recoveries of benzo[a]pyrene-d_{12} and perylene-d_{12} ranged from 17 to 22% and from 18-32%, respectively. The low recoveries of these two internal standards could be explained by either loss through the sample preparation process and/or through sample matrix effects. The internal standards benzo[a]pyrene-d_{12} and perylene-d_{12} are relatively more reactive PAH compounds as compared to other internal standards. Thus, it is assumed that the sample matrix effects could contribute significantly to the loss experienced by these two internal standards. It should be noted that the loss of benzo[a]pyrene-d_{12} does not directly apply to the relatively more stable benzo[e]pyrene. Originally, the internal standard for the benzo[e]pyrene was benzo[a]pyrene-d_{12}. The measured values of benzo[e]pyrene could be overestimated because of the low recovery of the Method 429 specified internal standard. Ideally, benzo[e]pyrene-d_{12} could have been used as the internal standard for benzo[e]pyrene; however, this standard was not included in the specified method. The benzo[e]pyrene was re-quantified using benzo[k]fluoranthene-d_{12}, an internal standard because the benzo[e]pyrene and benzo[k]fluoranthene have similar reactivity. Surrogate standards were not used in the FH samples, thus no recovery data were reported.

Table 6-18. Summary of PAH Results and Standard Recoveries for Front Half Air Samples

ANALYTES	RUN 2 48190-11-02 FH-R2-PAH (ng/sample)	DL (ng/sample)	RUN 3 48190-11-03 FH-R3-PAH (ng/sample)	DL (ng/sample)	RUN 4 48190-11-04 FH-R4-PAH (ng/sample)	DL (ng/sample)
Acenaphthene	ND	20	ND	20	ND	20
Acenaphthylene	ND	20	ND	20	ND	20
Anthracene	ND	20	ND	20	ND	20
Benzo(a)anthracene	53		ND	20	ND	20
Benzo(b)fluoranthene	350		120		96	
Benzo(k)fluoranthene	270		92		37	
Benzo(g,h,i)perylene	210		120		ND	20
Benzo(a)pyrene	110		53		ND	20
Benzo(e)pyrene	2000 E		640		270	
Chrysene	200		77		43	
Dibenzo(a,h)anthracene	85		51		21	
Fluoranthene	53		33		23	
Fluorene	ND	20	ND	20	ND	20
Indeno(1,2,3-c,d)pyrene	260		170		29	
2-Methylnaphthalene	40		37		27	
Naphthalene	1700 E,B		530 B		440 B	
Perylene	ND	20	ND	20	ND	20
Pyrene	43		27		ND	20
Phenanthrene	130	64	ND	64	ND	64

Table 6-18. (Continued)

INTERNAL STANDARDS	SPIKE CONC. (ng/sample)	RUN 2 48190-11-02 FH-R2-PAH (ng/sample)	RECOVERY (%)	RUN 3 48190-11-03 FH-R3-PAH (ng/sample)	RECOVERY (%)	RUN 4 48190-11-04 FH-R4-PAH (ng/sample)	RECOVERY (%)
Acenaphthylene-d8	200	112	56	100	50	128	64
Benzo(a)anthracene-d12	200	142	71	148	74	124	62
Benzo(a)pyrene-d12	200	34	17 &	44	22 &	42	21 &
Benzo(b)fluoranthene-d12	200	204	102	214	107	178	89
Benzo(g,h,i)perylene-d12	200	148	74	166	83	162	81
Benzo(k)fluoranthene-d12	200	172	86	194	97	206	103
Chrysene-d12	200	194	97	204	102	218	109
Dibenzo(a,h)anthracene-d14	200	170	85	192	96	156	78
Fluoranthene-d10	200	188	94	204	102	266	133
Fluorene-d10	200	174	87	194	97	158	79
Indeno(1,2,3-c,d)pyrene-d12	200	168	84	186	93	174	87
Naphthalene-d8	200	174	87	172	86	176	88
Perylene-d12	200	64	32 &	36	18 &	58	29 &
Phenanthrene-d10	200	164	82	194	92	138	69
SURROGATE STANDARDS							
13C-Fluorene-d10	200		NA		NA		NA
RECOVERY STANDARDS							
Acenaphthene-d10	200	148	74	158	79	150	75
Pyrene-d10	200	180	90	192	96	248	124

DL = Detection limit.
& = QC value outside the accuracy goal for Method (10-150% Recovery of Standard Spiked samples).
ND = Not detected, value noted is reporting limit.
E = Estimated result, reported concentration exceeds the calibration range.
NA = Not applicable.
B = Lab method blank contamination of target analytes at level above DL.

Table 6-19. Summary of PAH Results and Standard Recoveries for Back Half Air Samples

ANALYTES	RUN 2 48190-09-02 BH-R2-PAH (ng/sample)	DL (ng/sample)	RUN 3 48190-09-03 BH-R3-PAH (ng/sample)	DL (ng/sample)	RUN 4 48190-09-04 BH-R4-PAH (ng/sample)	DL (ng/sample)
Acenaphthene	160		130		26	
Acenaphthylene	1700 D,JA		2000 E,JA		260 JA	
Anthracene	210		82JA		93	
Benzo(a)anthracene	83		390		82	
Benzo(b)fluoranthene	860		910		170	
Benzo(k)fluoranthene	840		470		120	
Benzo(g,h,i)perylene	130		89		85	
Benzo(a)pyrene	ND	20	NQ	20	NQ	20
Benzo(e)pyrene	530 JA		540 JA		120 JA	20
Chrysene	3300 E		2000 E		440	
Dibenzo(a,h)anthracene	20		25		ND	20
Fluoranthene	3900 E		3600 E		910	
Fluorene	1500 E		1700 E		130	
Indeno(1,2,3-c,d)pyrene	97		88		45	
2-Methylnaphthalene	32000 D,E,B		30000 D,E,B		1300 D,B	
Naphthalene	450000 D,E,B		380000 D,E,B		210000 D,E,B	
Perylene	370 JA		NQ	20	57 JA	
Pyrene	2600 E		2400 E		490	
Phenanthrene	33000 D,E,B		33000 D,E,B		8000 E,B	

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Table 6-19. (Continued)

INTERNAL STANDARDS	SPIKE CONC. (ng/sample)	RUN 2 48190-09-02 BH-R2-PAH (ngsample)	RECOVERY (%)	RUN 3 48190-09-03 BH-R3-PAH (ng/sample)	RECOVERY (%)	RUN 4 48190-09-04 BH-R4-PAH (ng/sample)	RECOVERY (%)
Acenaphthylene-d8	200	3.0	1.5 &	1.4	0.7 &	2.4	1.2 &
Benzo(a)anthracene-d12	200	84	42 &	72	36 &	76	38 &
Benzo(a)pyrene-d12	200	12.2	6.1 &	9.8	4.9 &		NA
Benzo(b)fluoranthene-d12	200	204	102	176	88	172	86
Benzo(g,h,i)perylene-d12	200	94	47 &	94	47 &	134	67
Benzo(k)fluoranthene-d12	200	136	68	172	86	148	74
Chrysene-d12	200	132	66	176	88	146	73
Dibenzo(a,h)anthracene-d14	200	116	58	126	63	126	63
Fluoranthene-d10	200	146	73	164	82	164	82
Fluorene-d10	200	186	93	180	90	216	108
Indeno(1,2,3-c,d)pyrene	200	120	60	136	68	136	68
Naphthalene-d8	200	182	91	178	89	154	77
Perylene-d12	200	3.8	1.9 &		NA	3.6	1.8 &
Phenanthrene-d10	200	248	124	256	128	250	125
SURROGATE STANDARDS							
13C6-Fluorene	200	48	24 &	62	31 &	26	13 &
RECOVERY STANDARDS							
Acenaphthene-d10	200	152	76	174	87	146	73
Pyrene-d10	200	128	64	140	70	148	74

DL = Detection limit.
& = QC value outside the accuracy goal for Method (50-150% Recovery of Standard Spiked samples).
ND = Not detected, value noted is reporting limit.
NA = Not applicable.
D = Result reported is the analysis of a dilution.
E = Estimated result, reported concentration exceeds the calibration range.
B = Lab method blank contamination of target analyte at the level above DL.
JA = The analyte was positively identified , but the quantification was an estimate.
NQ = Not quantifiable due to low standard recovery of target analyte.

Acceptable recoveries (>50%) of 10 out of 14 internal standards were obtained in the BH samples. The four internal standards with low recoveries in the BH samples were acenaphthylene-d_8, benz[a]anthracene-d_{12}, benzo[a]pyrene-d_{12}, and perylene-d_{12}. These four internal standards are relatively more reactive as compared to other remaining internal standards. As described above, the loss of the internal standards was possibly due to the combination of sample preparation loss and sample matrix effects. Matrix effects between the FH and BH samples could have resulted in more internal standards being located in the BH samples with lower than 50% recovery. For the same reason discussed before, the internal standard, benzo[k]fluoranthene-d_{12}, was used for the quantification of benzo[e]pyrene level. Low recoveries (13-24%) were obtained for the surrogate standard (field spike) in the BH samples. The loss of the surrogate standards are believed to be from field handling and sample matrix effects. Acceptable recovery standard results were obtained in the BH samples; ranging from 64-87%.

Lab Control Samples. This assumption that sample matrix effects influenced the low recovery of benzo[a]pyrene-d_{12} and perylene-d_{12} is further supported with the recovery data obtained from lab control spike (LCS) and lab control spike duplicate (LCSD) samples. Tables 6-20 (a and b) summarize the recovery data of the FH and BH lab control spikes (LCS) and lab control spike duplicates (LCSD), respectively. As shown in Table 6-20 (a and b), acceptable (50-150%) recoveries of all native target PAHs except naphthalene were obtained in the LCS and LCSD samples. Quantitative recoveries (120-125%) of naphthalene were obtained in the LCS and LCSD FH samples but were not recovered in the BH samples because of the high reporting level limit (470 ng/sample) of naphthalene resulting from the background levels present in the field blank. Note that acceptable recoveries (>50%) of all internal standards were obtained in these LCS and LCSD samples. Unlike in the field real samples discussed before, quantitative recoveries of the internal standard, benzo[a]pyrene-d_{12}, were obtained and ranged from 86-93%. This finding confirmed that sample matrix effects notably contributed to the loss of the internal standards of benzo[a]pyrene-d_{12}, perylene-d_{12} in the field FH and BH samples and acenaphthylene-d_8 and benz[a]anthracene-d_{12} in the field BH samples. As expected, satisfactory recoveries (>90%) of the recovery standards were established in these LCS and LCSD samples.

Blanks - Lab and Method. Table 6-21 summarizes the results and recovery data for the FH and BH lab and method blank samples. As shown in Table 6-21, all the target native PAH

Table 6-20a. Summary of PAH Results and Standard Recoveries for Front Half Lab Control Spike and Spike Duplicate Samples

ANALYTES	TARGET RANGE (%)	LCS SPIKE CONC. (ng/sample)	LCS 48190-11-08 FH-LCS-PAH (ng/sample)	RECOV. (%)	LCS DUP 48190-11-09 FH-LCSDUP-PAH (ng/sample)	RECOV. (%)	RECOV. RPD (%)	FIELD BLANK 48190-11-07 FH-FB1-PAH (ng/sample)	DL (ng/sample)
Acenaphthene	50-150	200	150	75	170	85	10	ND	20
Acenaphthylene	50-150	200	180	90	180	90	0	ND	20
Anthracene	50-150	200	260	130	250	125	5	ND	20
Benzo(a)anthracene	50-150	200	200	100	190	95	5	ND	20
Benzo(b)fluoranthene	50-150	200	200	100	210	105	5	ND	20
Benzo(k)fluoranthene	50-150	200	190	95	200	100	5	ND	20
Benzo(g,h,i)perylene	50-150	200	200	100	210	105	5	ND	20
Benzo(a)pyrene	50-150	200	190	95	200	100	5	ND	20
Benzo(e)pyrene	50-150	200	220	110	220	110	0	ND	20
Chrysene	50-150	200	200	100	210	105	5	ND	20
Dibenzo(a,h)anthracene	50-150	200	200	100	200	100	0	ND	20
Fluoranthene	50-150	200	220	92	210	87	5	36	
Fluorene	50-150	200	210	105	250	125	20	ND	20
Indeno(1,2,3-c,d)dipyrene	50-150	200	170	85	200	100	15	ND	20
2-Methylnaphthalene	50-150	200	180	90	190	95	5	ND	20
Naphthalene	50-150	200	250 B	125	240 B	120	1	ND	100
Perylene	50-150	200	200	100	200	100	0	ND	20
Pyrene	50-150	200	210	94	210	94	0	21	
Phenanthrene	50-150	200	210	105	210	105	0	68 B	64

Table 6-20a. (Continued)

INTERNAL STANDARDS	TARGET RANGE (%)	LCS SPIKE CONC. (ng/sample)	LCS 48190-11-08 FH-LCS-PAH (ng/sample)	RECOV. (%)	LCS DUP 48190-11-09 FH-LCSDUP-PAH (ng/sample)	RECOV. (%)	RECOV. RPD (%)	FIELD BLANK 48190-11-07 FH-FB1-PAH (ng/sample)	RECOV. (%)
Acenaphthylene-d8	50-150	200	198	99	190	95	4	162	81
Benzo(a)anthracene-d12	50-150	200	162	81	178	89	8	156	78
Benzo(a)pyrene-d12	50-150	200	178	89	172	86	3	142	71
Benzo(b)fluoranthene-d12	50-150	200	194	97	194	97	0	220	110
Benzo(g,h,i)perylene-d12	50-150	200	220	110	168	84	26	184	92
Benzo(k)fluoranthene-d12	50-150	200	214	107	196	98	9	206	103
Chrysene-d12	50-150	200	226	113	204	102	11	206	103
Dibenzo(a,h)anthracene-d14	50-150	200	204	102	158	79	23	174	87
Fluoranthene-d10	50-150	200	200	100	196	98	2	188	94
Fluorene-d10	50-150	200	132	66	140	70	4	150	75
Indeno(1,2,3-c,d)pyrene-d12	50-150	200	224	112	170	85	27	190	95
Naphthalene-d8	50-150	200	198	99	172	86	13	174	87
Perylene-d12	50-150	200	178	89	174	87	2	100	50
Phenanthrene-d10	50-150	200	130	65	134	67	2	152	76
SURROGATE STANDARDS									
13C6-Fluorene	50-150	200		NA		NA			NA
RECOVERY STANDARDS									
Acenaphthene-d10	50-150	200	216	108	194	97	11	192	96
Pyrene-d10	50-150	200	204	102	190	95	7	184	92

DL = Detection limit.
& = QC value outside the accuracy of precision goal for Method (50-150% Recovery of Standard Spiked samples).
ND = Not detected.
NA = Not applicable.
B = Lab blank contamination of target analyte at concentration above the reporting level.

Table 6-20b. Summary of PAH Results and Standard Recoveries for Back Half Lab Control Spike and Spike Duplicate Samples

ANALYTES	TARGET RANGE (%)	LCS SPIKE CONC. (ng/sample)	LCS 48190-09-09 BH-LCS-PAH (ng/sample)	RECOV. (%)	LCS DUP 48190-09-10 BH-LCSDUP-PAH (ng/sample)	RECOV. (%)	RECOV. RPD (%)	FIELD BLANK 48190-09-07 BH-FB1-PAH (ng/sample)	DL
Acenaphthene	50-150	200	170	85	150	75	10	ND	20
Acenaphthylene	50-150	200	180	90	170	85	5	ND	20
Anthracene	50-150	200	250	125	200	100	25	ND	20
Benzo(a)anthracene	50-150	200	200	100	190	95	5	ND	20
Benzo(b)fluoranthene	50-150	200	200	100	170	85	15	ND	20
Benzo(k)fluoranthene	50-150	200	200	100	200	100	0	ND	20
Benzo(g,h,i)perylene	50-150	200	190	62	190	62	0	67	
Benzo(a)pyrene	50-150	200	200	100	190	95	5	ND	20
Benzo(e)pyrene	50-150	200	210	93	230	103	10	25	
Chrysene	50-150	200	190	95	180	90	5	ND	20
Dibenzo(a,h)anthracene	50-150	200	210	105	190	95	10	ND	20
Fluoranthene	50-150	200	220	85	200	75	10	50	
Fluorene	50-150	200	230	115	230	115	0	ND	20
Indeno(1,2,3-c,d)dipyrene	50-150	200	190	95	170	85	10	ND	20
2-Methylnaphthalene	50-150	200	210	105	200	100	5	ND	120
Naphthalene	50-150	200	NQ*	NQ*	NQ*	NQ*	NA	ND	470
Perylene	50-150	200	200	100	190	95	5	ND	20
Pyrene	50-150	200	210	78	190	68	10	55	20
Phenanthrene	50-150	200	210	105	210	105	0	ND	120

Table 6-20b. (Continued)

INTERNAL STANDARDS	TARGET RANGE (%)	LCS SPIKE CONC. (ng/sample)	LCS 48190-09-09 BH-LCS-PAH (ng/sample)	RECOV. (%)	LCS DUP 48190-09-10 BH-LCSDUP-PAH (ng/sample)	RECOV. (%)	RECOV. RPD (%)	FIELD BLANK 48190-09-07 BH-FB1-PCB (ng/sample)	RECOV. (%)
Acenaphthylene-d8	50-150	200	196	98	178	89		172	86
Benzo(a)anthracene-d12	50-150	200	182	91	172	86		166	83
Benzo(a)pyrene-d12	50-150	200	186	93	162	81		176	88
Benzo(b)fluoranthene-d12	50-150	200	198	99	200	100		208	104
Benzo(g,h,i)perylene-d12	50-150	200	196	98	174	87		220	110
Benzo(k)fluoranthene-d12	50-150	200	212	106	196	98		208	104
Chrysene-d12	50-150	200	228	114	222	111		222	111
Dibenzo(a,h)anthracene-d14	50-150	200	198	99	172	86		238	119
Fluoranthene-d10	50-150	200	212	106	204	102		198	99
Fluorene-d10	50-150	200	176	88	186	93		154	77
Indeno(1,2,3-c,d)pyrene-d12	50-150	200	198	99	178	89		228	114
Naphthalene-d8	50-150	200	184	92	178	89		156	78
Perylene-d12	50-150	200	182	91	152	76		182	91
Phenanthrene-d10	50-150	200	152	76	166	83		142	71
SURROGATE STANDARDS									
13C6-Fluorene	50-150	200		NA		NA		122	62
RECOVERY STANDARDS									
Acenaphthene-d10	50-150	100	218	109	200	100		190	95
Pyrene-d10	50-150	100	204	102	196	98		190	95

DL = Detection limit.
& = QC value outside the accuracy goal for Method.
ND = Not detected.
NQ* = Not determined because of high detection limit of analyte resulting from BH lab blank contamination.
NA = Not applicable.

Table 6-21. Summary of PAH Results and Standard Recoveries for Lab and Method Blank Samples

ANALYTES	LAB BLANK 48190-11-10 FH-LB-PAH (ng/sample)	DL (ng/sample)	LAB BLANK 48190-09-11 BH-LB-PAH (ng/sample)	DL (ng/sample)	METHOD BLANK 48200-51-03 (ng/sample)	DL (ng/sample)	XAD BACKGROUND 48200-51-02 (ng/sample)	DL (ng/sample)
Acenaphthene	ND	20	ND	20	D	20	ND	20
Acenaphthylene	ND	20	ND	20	ND	20	ND	20
Anthracene	ND	20	ND	20	ND	20	ND	20
Benzo(a)anthracene	ND	20	ND	20	ND	20	ND	20
Benzo(b)fluoranthene	ND	20	ND	20	ND	20	ND	20
Benzo(k)fluoranthene	ND	20	ND	20	ND	20	ND	20
Benzo(g,h,i)perylene	ND	20	ND	20	ND	20	ND	20
Benzo(a)pyrene	ND	20	ND	20	ND	20	ND	20
Benzo(e)pyrene	ND	20	ND	20	ND	20	ND	20
Chrysene	ND	20	ND	20	ND	20	ND	20
Dibenzo(a,h)anthracene	ND	20	ND	20	ND	20	ND	20
Fluoranthene	ND	20	ND	20	ND	20	ND	20
Fluorene	ND	20	ND	20	ND	20	ND	20
Indeno(1,2,3-c,d)pyrene	ND	20	ND	20	ND	20	ND	20
2-Methylnaphthalene	ND	20	25	20	ND	20	ND	20
Naphthalene	21		94		59 J,B	20	150	20
Perylene	ND	20	ND	20	ND	20	ND	20
Pyrene	ND	20	ND	20	ND	20	ND	20
Phenanthrene	ND	20	25		ND	20	20	

Table 6-21. (Continued)

INTERNAL STANDARDS	TARGET RANGE (%)	SPIKE CONC. (ng/sample)	LAB BLANK 48190-11-10 FH-LB-PAH (ng/sample)	RECOV. (%)	LAB BLANK 48190-09-11 BH-LB-PAH (ng/sample)	RECOV. (%)	METHOD BLANK 48200-51-03 (ng/sample)	RECOV. (%)	XAD BACKGROUND 48200-51-02 (ng/sample)	RECOV. (%)
Acenaphthylene-d8	50 - 150	200	182	91	194	97	186	93	192	96
Benzo(a)anthracene-d12	50 - 150	200	158	79	130	65	144	72	170	85
Benzo(a)pyrene-d12	50 - 150	200	152	76	158	79	172	86	202	101
Benzo(b)fluoranthene-d12	50 - 150	200	182	91	162	81	182	91	200	100
Benzo(g,h,i)perylene-d12	50 - 150	200	166	83	172	86	172	86	196	98
Benzo(k)fluoranthene-d12	50 - 150	200	186	93	218	109	204	102	222	111
Chrysene-d12	50 - 150	200	218	109	270	135	238	119	234	117
Dibenzo(a,h)anthracene-d14	50 - 150	200	148	74	134	67	146	73	174	87
Fluoranthene-d10	50 - 150	200	188	94	260	130	208	104	222	111
Fluorene-d10	50 - 150	200	152	76	156	78	154	77	176	88
Indeno(1,2,3-c,d)pyrene	50 - 150	200	160	80	158	79	156	78	190	95
Naphthalene-d8	50 - 150	200	184	92	200	100	208	104	218	109
Perylene-d12	50 - 150	200	156	78	172	86	146	73	172	86
Phenanthrene-d10	50 - 150	200	124	62	132	66	132	66	164	82
SURROGATE STANDARDS										
13C6-Fluorene		200		NA		NA		NA		NA
RECOVERY STANDARDS										
Acenaphthene-d10	50-150	200	192	96	246	123	214	107	214	107
Pyrene-d10	50-150	200	188	94	194	97	232	116	228	114

DL = Detection limit.
ND = Not detected.
NA = Not applicable.
B = Lab method blank contamination of target analyte at concentration above the reporting level.
J = Estimated value, below detection limit.

compounds except naphthalene were not detected in the method blank. Naphthalene and phenanthrene were detected in the clean XAD-2 trap and the BH lab blank. This result is typical of background levels for these compounds (per M-429 PQL for HRMS analyses) in XAD which has not been stringently cleaned to remove these manufacturing by-product contaminants. In the BH lab blank, 2-methyl naphthalene was also detected. Per the WAM's instructions and the method, five times the detected concentration in the blank was noted as the reporting limit for the compounds detected. As expected, acceptable recoveries (50-150%) were obtained for all internal standards and recovery standards in the lab blank, method blank, and XAD-2 blank samples.

Blanks - Field and Proof. The measured native PAH data and recovery data of internal standards, surrogate standards, and recovery standards in the FH and BH field and proof blank samples are summarized in Tables 6-22 and 6-23, respectively. As shown in Table 6-22, only trace amounts of phenanthrene, fluoranthene, and pyrene were detected in the field and proof blank FH samples. These compounds are likely oxygenated artifacts which are sometimes generated when air is induced to XAD during field blank collection. Acceptable recoveries (50-150%) were obtained for all the internal standards and recovery standards in these samples.

As shown in Table 6-23, trace amounts of pyrene, fluoranthene, benzo[e]pyrene, and benzo[g,h,i]perylene were detected in the field and proof blank BH samples. Acceptable recoveries (50-150%) were established for all the internal standards and recovery standards in these blank samples. Acceptable recoveries of the surrogate standards were also established and ranged from 61 to 69%.

6.1.3.2 Scrubber Water Samples

PCB Analysis. The initial calibration for both the SPB-Octyl and DB-1 analyses met the requirement for response factors having less than 35% relative standard deviation (RSD) for all analytes (actual range = <17% for SPB-Octyl and DB-1 calibrations). The continuing calibrations met the requirement for response factors being within 35% of the response factors generated in the initial calibration for at least 70% of the analytes. In fact, all analytes in all continuing calibrations were within 35% of the initial calibration.

Table 6-22. Summary of PAH Results and Standard Recoveries for Field and Proof Blank Front Half Air Samples

ANALYTES	FIELD BLANK 48190-11-07 FH-FB1-PAH (ng/sample)	DL (ng/sample)	PROOF BLANK 1 48190-11-05 FH-PB1-PAH (ng/sample)	DL (ng/sample)	PROOF BLANK 2 48190-11-06 FH-PB2-PAH (ng/sample)	DL (ng/sample)
Acenaphthene	ND	20	ND	20	ND	20
Acenaphthylene	ND	20	ND	20	ND	20
Anthracene	ND	20	ND	20	ND	20
Benzo(a)anthracene	ND	20	ND	20	ND	20
Benzo(b)fluoranthene	ND	20	ND	20	ND	20
Benzo(k)fluoranthene	ND	20	ND	20	ND	20
Benzo(g,h,i)perylene	ND	20	ND	20	ND	20
Benzo(a)pyrene	ND	20	ND	20	ND	20
Benzo(e)pyrene	ND	20	ND	20	ND	20
Chrysene	ND	20	ND	20	ND	20
Dibenzo(a,h)anthracene	ND	20	ND	20	ND	20
Fluoranthene	36		ND	20	ND	20
Fluorene	ND	20	ND	20	ND	20
Indeno(1,2,3-c,d)pyrene	ND	20	ND	20	ND	20
2-Methylnaphthalene	ND	20	53		58	
Naphthalene	ND	100	440 B	100	350 B	100
Perylene	ND	20	ND	20	ND	20
Pyrene	21		ND	20	ND	20
Phenanthrene	68 B	64	ND	64	ND	64

Table 6-22. (Continued)

INTERNAL STANDARDS	SPIKE CONC. (ng/sample)	FIELD BLANK 48190-11-07 FH-FB1-PAH (ng/sample)	RECOVERY (%)	PROOF BLANK 1 48190-11-05 FH-PB1-PAH (ng/sample)	RECOVERY (%)	PROOF BLANK 2 48190-11-06 FH-PB2-PAH (ng/sample)	RECOVERY (%)
Acenaphthylene-d8	200	162	81	146	73	136	68
Benzo(a)anthracene-d12	200	156	78	162	81	144	72
Benzo(a)pyrene-d12	200	142	71	176	88	152	76
Benzo(b)fluoranthene-d12	200	220	110	200	100	194	97
Benzo(g,h,i)perylene-d12	200	184	92	194	97	164	82
Benzo(k)fluoranthene-d12	200	206	103	212	106	194	97
Chrysene-d12	200	206	103	220	110	204	102
Dibenzo(a,h)anthracene-d14	200	174	87	194	97	168	84
Fluoranthene-d10	200	188	94	188	94	172	86
Fluorene-d10	200	150	75	154	77	146	73
Indeno(1,2,3-c,d)pyrene-d12	200	190	95	198	99	166	83
Naphthalene-d8	200	174	87	152	76	148	74
Perylene-d12	200	100	50	178	89	160	80
Phenanthrene-d10	200	152	76	158	79	136	68
SURROGATE STANDARDS							
13C6-Fluorene	200		NA		NA		NA
RECOVERY STANDARDS							
Acenaphthene-d10	200	192	96	166	83	160	80
Pyrene-d10	200	184	92	174	87	168	84

DL = Detection limit.
ND = Not detected.
NA = Not applicable.
B = Lab method blank contamination of target analyte at concentration above the reporting level.

Table 6-23. Summary of PAH Standard Recoveries for Field and Proof Blank Back Half Air Samples

ANALYTES	FIELD BLANK 48190-09-07 BH-FB1-PAH (ng/sample)	DL (ng/sample)	PROOF BLANK 1 48190-09-05 BH-PB1-PAH (ng/sample)	DL (ng/sample)	PROOF BLANK 2 48190-09-06 BH-PB2-PAH (ng/sample)	DL (ng/sample)
Acenaphthene	ND	20	ND	20	ND	20
Acenaphthylene	ND	20	ND	20	ND	20
Anthracene	ND	20	ND	20	ND	20
Benzo(a)anthracene	ND	20	ND	20	ND	20
Benzo(b)fluoranthene	ND	20	ND	20	ND	20
Benzo(k)fluoranthene	ND	20	ND	20	ND	20
Benzo(g,h,i)perylene	67		29		69	
Benzo(a)pyrene	ND	20	ND	20	ND	20
Benzo(e)pyrene	25		ND	20	ND	20
Chrysene	ND	20	ND	20	ND	20
Dibenzo(a,h)anthracene	ND	20	ND	20	ND	20
Fluoranthene	50		21		37	
Fluorene	ND	20	ND	20	ND	20
Indeno(1,2,3-c,d)pyrene	ND	20	ND	20	ND	20
2-Methylnaphthalene	ND	120	ND	120	ND	120
Naphthalene	ND	470	ND	470	ND	470
Perylene	ND	20	ND	20	ND	20
Pyrene	55		28		58	
Phenanthrene	ND	120	ND	120	ND	120

Table 6-23. (Continued)

INTERNAL STANDARDS	SPIKE CONC. (ng/sample)	FIELD BLANK 48190-09-07 BH-FB1-PAH (ng/sample)	RECOVERY (%)	PROOF BLANK 1 48190-09-05 BH-PB1-PAH (ng/sample)	RECOVERY (%)	PROOF BLANK 2 48190-09-06 BH-PB2-PAH (ng/sample)	RECOVERY (%)
Acenaphthylene-d8	200	172	86	192	96	178	89
Benzo(a)anthracene-d12	200	166	83	194	97	190	95
Benzo(a)pyrene-d12	200	176	88	194	97	196	98
Benzo(b)fluoranthene-d12	200	208	104	204	102	210	105
Benzo(g,h,i)perylene-d12	200	220	110	184	92	170	85
Benzo(k)fluoranthene-d12	200	208	104	218	109	204	102
Chrysene-d12	200	222	111	242	121	232	116
Dibenzo(a,h)anthracene-d14	200	238	119	180	90	170	85
Fluoranthene-d10	200	198	99	206	103	172	86
Fluorene-d10	200	154	77	168	84	166	83
Indeno(1,2,3-c,d)pyrene-d12	200	228	114	188	94	178	89
Naphthalene-d8	200	156	78	182	91	154	77
Perylene-d12	200	182	91	200	100	190	95
Phenanthrene-d10	200	142	71	156	78	140	70
SURROGATE STANDARDS							
13C6-Fluorene	200	124	62	122	61	138	69
RECOVERY STANDARDS							
Acenaphthene-d10	200	190	95	222	111	188	94
Pyrene-d10	200	190	95	206	103	172	86

DL = Detection limit.
ND = Not detected.

Field Samples. Summary tables of PCB results and standard recoveries for the water inlet samples are shown in Table 6-24. Water outlet sample results are in Table 6-25. Internal standard recoveries were within the method specified limits of 20-160% and ranged from 42-83% for inlet samples and from 38-76% for outlet samples. These internal standard recoveries indicate analytes were well recovered during laboratory extraction and were retained during the extract cleanup process. $^{13}C_{12}$-PCB-81 and $^{13}C_{12}$-PCB-111 were added to the water samples as cleanup standards after extraction but before any cleanup procedures. Method specified recovery ranges for these standards were from 20-160% for $^{13}C_{12}$-PCB-81 and 40-140% for $^{13}C_{12}$-PCB-111. Recovery of the cleanup standards in the inlet and outlet samples were within the limits and ranged from 58-107% for $^{13}C_{12}$-PCB-81 and from 53-85% for $^{13}C_{12}$-PCB-111 indicating that analytes were well retained through the extract cleanup procedures. Results for water inlet Run 4 and water outlet Run 3 are from the archive portion of these sample extracts. Internal standard recoveries from analysis of the initial portion of these two samples were indicative of a laboratory error resulting in the combining of WO-R3-312B and WI-R4-411B during the final carbon column cleanup step. In order to verify this assumption and provide results for these two samples, the archive portions of these two samples along with the archive portion of the lab blank were then analyzed.

Lab Control Samples. Table 6-26 presents PCB results and standard recoveries for the laboratory control spike and laboratory control spike duplicate (LCS/LCSD) scrubber water samples. Recovery of all native PCBs spiked into the LCS/LCSD were within the 40-160% target recovery range except for PCB-77 in the LCS sample (166%). The relative percent difference (RPD) between duplicates were all under the target <50% and ranged from 1-48% indicating acceptable analytical precision was achieved. Several analytes (PCB-118 in the LCS and PCB-114 in both the LCS and LCSD) were detected slightly above the upper calibration range of 40,000 pg/L and are flagged with an "E". Internal standard recoveries for the LCS were below the acceptable range of 20-160%. Recoveries of cleanup standards for the LCS indicate that the internal standard losses did not occur in the cleanup process and were most likely a result of inefficient extraction. The low recoveries for the LCS internal standards did not affect accuracy as evidenced by good recovery of analytes in the LCS sample (only PCB-77 was

Table 6-24. Summary of PCB Results and Standard Recoveries for Scrubber Water Inlet Samples

ANALYTES	RUN 2 48200-56-03 WI-R2-211B (pg/L)	DL (pg/L)	RUN 3 48200-56-05 WI-R3-311B (pg/L)	DL (pg/L)	RUN 4-ARCHIVE 48200-56-07 WI-R4-411B (pg/L)	DL (pg/L)
PCB-77	158 B		166 B		138	
PCB-123	177 B		31.6 B		17.8 B	
PCB-118	1200 B		1060 B		1060 B	
PCB-114	233 B		68.0 B		45.1 B	
PCB-105	690 B		491 B		504 B	
PCB-126	24.0 B		6.91		3.66 B	
PCB-167	186 B		44.4 B		36.1 B	
PCB-156	243 #,B		85.2 #,B		80.5 #,B	
PCB-157	166 #,B		28.5 #,B		19.6 #,B	
PCB-169	26.8 B		5.81 B		1.97 B	
PCB-180	338 B		190 B		177 B	
PCB-170	105 B		79.9 B		78.2 B	
PCB-189	29.0 B		9.14 B		{5.12} B	

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	RUN 2 48200-56-03 WI-R2-211B (pg/sample)	RECOVERY (%)	RUN 3 48200-56-05 WI-R3-311B (pg/sample)	RECOVERY (%)	RUN 4-ARCHIVE 48200-56-07 WI-R4-411B (pg/sample)	RECOVERY (%)
13C-PCB-77	20 - 160	2000	952	48	1230	62	1660	83
13C-PCB-118	20 - 160	2000	910	45	1170	59	1190	59
13C-PCB-105	20 - 160	2000	996	50	1180	59	1171	59
13C-PCB-126	20 - 160	2000	1150	57	1430	71	1350	67
13C-PCB-167	20 - 160	2000	1060	53	1250	63	1150	57
13C-PCB-156	20 - 160	2000	1310 #	65	1570 #	79	1330 #	67
13C-PCB-157	20 - 160	2000	1320 #	66	1460 #	73	1300 #	65
13C-PCB-169	20 - 160	2000	1210	60	1400	70	1180	59
13C-PCB-180	20 - 160	2000	1180	59	1510	76	1100	55
13C-PCB-189	20 - 160	2000	1270	63	1510	76	1370	54
13C-PCB-209	20 - 160	4000	2530	63	3010	75	1670	42
CLEANUP STANDARDS								
13C-PCB-81	20 - 160	1000	585	58	735	74	1070	107
13C-PCB-111	40 - 140	5000	2720	54	3380	68	4240	85

# = Value from second column confirmation.
DL = Detection Limit.
B = Congener found in lab blank at >20% of the concentration found in the sample.

Table 6-25. Summary of PCB Results and Standard Recoveries for Scrubber Water Outlet Samples

ANALYTES	RUN 2 48200-56-04 WO-R2-212B (pg/L)	DL (pg/L)	RUN 3-ARCHIVE 48200-56-06 WO-R3-312B (pg/L)	DL (pg/L)	RUN 4 48200-56-08 WO-R4-412B (pg/L)	DL (pg/L)
PCB-77	4180		3630		2520	
PCB-123	85.8 B		65.1 B		50.2 B	
PCB-118	1680 B		3520 B		1620 B	
PCB-114	157 B		155 B		124 B	
PCB-105	946 B		1870 B		854 B	
PCB-126	135		118		98.3	
PCB-167	158 B		164 B		125 B	
PCB-156	249 #,B		413 #		236 #,B	
PCB-157	119 #,B		110 #,B		83.0 #,B	
PCB-169	113		80.7		97.6	
PCB-180	686 B		1000		653 B	
PCB-170	264 B		414		298 B	
PCB-189	30.1 B		23.8 B		44.4 B	

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	RUN 2 48200-56-04 WO-R2-212B (pg/sample)	RECOVERY (%)	RUN 3-ARCHIVE 48200-56-06 WO-R3-312B (pg/sample)	RECOVERY (%)	RUN 4 48200-56-08 WO-R4-412B (pg/sample)	RECOVERY (%)
13C-PCB-77	20 - 160	2000	1340	67	1440	72	1110	55
13C-PCB-118	20 - 160	2000	1140	57	1130	57	1010	51
13C-PCB-105	20 - 160	2000	1220	61	1170	59	1110	55
13C-PCB-126	20 - 160	2000	1420	71	1250	63	1300	65
13C-PCB-167	20 - 160	2000	1280	64	1080	54	1100	55
13C-PCB-156	20 - 160	2000	1510 #	76	1360 #	68	1390 #	69
13C-PCB-157	20 - 160	2000	1520 #	76	1300 #	65	1370 #	68
13C-PCB-169	29 - 160	2000	1430	71	1100	55	1260	63
13C-PCB-180	20 - 160	2000	1360	68	1020	61	1250	63
13C-PCB-189	20 - 160	2000	1500	75	1000	50	1300	65
13C-PCB-209	20 - 160	4000	2930	73	1520	38	2280	57
CLEANUP STANDARDS								
13C-PCB-81	20 - 160	1000	662	66	840	85	684	68
13C-PCB-111	40 - 140	5000	2630	53	3670	73	3080	62

DL = Detection limit.
# = Value from second column confirmation.
B = Congener found in lab blank at >20% of the concentration found in the sample.

Table 6-26. Summary of PCB Results and Standard Recoveries for Lab Control Spike and Spike Duplicate Scrubber Water Samples

ANALYTES	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/L)	LCS 48200-56-09 LCS-PCB (pg/L)	RECOVERY (%)	LCS DUP 48200-56-10 LCSDUP-PCB (pg/L)	RECOVERY (%)	RECOVERY RPD (%)
PCB-77	40 - 160	800	1360	166 &	853	102	48
PCB-123	40 - 160	40000	31800	79	31440	78	1
PCB-118	40 - 160	40000	40740 E	99	25970	62	46
PCB-114	40 - 160	40000	56630 E	141	48740 E	121	15
PCB-105	40 - 160	40000	36460	90	33080	82	10
PCB-126	40 - 160	4000	2770	69	3270	81	16
PCB-167	40 - 160	40000	27630	69	28320	71	2
PCB-156	40 - 160	40000	30300 #	75	30500 #	76	1
PCB-157	40 - 160	40000	27740 #	69	28600 #	71	3
PCB-169	40 - 160	8000	4420	55	4690	59	6
PCB-180	40 - 160	40000	27900	69	24660	61	12
PCB-170	40 - 160	8000	6380	79	4780	59	29
PCB-189	40 - 160	8000	4680	58	4300	54	8

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	LCS 48200-56-09 LCS-PCB (pg/sample)	RECOVERY (%)	LCS DUP 48200-56-10 LCSDUP-PCB (pg/sample)	RECOVERY (%)
13C-PCB-77	20 - 160	2000	53	3 &	1060	53
13C-PCB-118	20 - 160	2000	32	2 &	657	33
13C-PCB-105	20 - 160	2000	35	2 &	650	33
13C-PCB-126	20 - 160	2000	50	3 &	809	40
13C-PCB-167	20 - 160	2000	29	1 &	572	29
13C-PCB-156	20 - 160	2000	41 #	2 &	835	42
13C-PCB-157	20 - 160	2000	37 #	2 &	752	38
13C-PCB-169	20 - 160	2000	46	2 &	677	34
13C-PCB-180	20 - 160	2000	39	2 &	727	36
13C-PCB-189	20 - 160	2000	31	2 &	726	36
13C-PCB-209	20 - 160	4000	58	1 &	1240	31
CLEANUP STANDARDS						
13C-PCB-81	20 - 160	100	475	48	743	79
13C-PCB-111	40 - 140	5000	1930	39	2820	56

Lab Control Spike % Recovery is calculated as ((concentration found - lab blank conc.)/spike conc.) x 100.
& = QC value outside the target recovery goal for Method.
# = *Value from second column confirmation.*
E = Estimated value reported is outside of calibration range.

recovered slightly above the data quality objective of 40-160%). Precision also was not affected.

Blanks. Table 6-27 lists PCB results and standard recoveries for the lab blanks. All water sample lab blanks were made using 1 L of HPLC grade reagent water in the laboratory. The levels of PCBs detected in the various blanks are typical of background levels detected using the sensitive high resolution mass spectrometry techniques. Because PCBs are detected in the lab blank and because some samples did not contain high levels of PCBs above the background levels there are several instances where the lab blanks exceeded the method performance criteria of being at less than 20% of the sample concentration. Results for Runs 2, 3 and 4 are flagged with a "B" in Tables 6-24 and 6-25 if the lab blank concentration was greater than 20% of the reported sample concentration. Internal standard recoveries for the blanks were within the 20-160% limits and ranged from 29-65%. Cleanup standard recoveries ranged from 54-84% for $^{13}C_{12}$-PCB-81 (target recovery = 20-160%) and from 45-67% for $^{13}C_{12}$-PCB-111(target recovery = 40-140%).

The target detection limit for these samples was 25 pg/L. Actual detection limits achieved were significantly lower than this.

The target turnaround time for the PCB analysis of scrubber water samples was to extract within 30 days of collection and analyze within 45 days of extraction. These turnaround times were met with extractions occurring within 7 days of collection and analysis occurring within 29 days of extraction. The only exception is for the two archive samples (inlet Run 4 and outlet Run 3) which were analyzed within 56 days of extraction.

D/F Analysis. The initial calibration met the requirement for response factors having less than 20% relative standard deviation (RSD) for native analytes and less than 35% RSD for labeled analytes (actual range = <10% for native analytes and < 8% for labeled analytes). All analytes in all continuing calibrations met the requirement for response factors being within 20% of the initial calibration response factors for native analytes and being within 30% of the initial calibration response factors for labeled analytes.

Field Samples. Summary tables of D/F results and standard recoveries for the water inlet samples are shown in Table 6-28. Water outlet sample results and standard recoveries are shown in Table 6-29. Several internal standard recoveries were below the acceptability range for Method 8290 of 40-135%, but most were within the range allowed by Method 1613 of 25-150%.

Table 6-27. Summary of PCB Results and Standard Recoveries for Lab Blank Scrubber Water Samples

ANALYTES	LAB BLANK 48200-56-11 LB-PCB (pg/L)	DL (pg/L)	LAB BLANK-ARCHIVE 48200-56-11 (pg/L)	DL (pg/L)
PCB-77	38.7		26.8	
PCB-123	83.0		41.0	
PCB-118	985		858	
PCB-114	152		91.3	
PCB-105	480		396	
PCB-126	8.25		(4.19) J	4.82
PCB-167	93.0		45.4	
PCB-156	124 #		73.7 #	
PCB-157	70.6 #		33.8 #	
PCB-169	8.97		5.14	
PCB-180	173		131	
PCB-170	60.5		46.1	
PCB-189	13.7		10.4	

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	LAB BLANK 48200-56-11 LB-PCB (pg/sample)	RECOVERY (%)	LAB BLANK-ARCHIVE 48200-56-11 BH-LB-PCB (pg/sample)	RECOVERY (%)
13C-PCB-77	20 - 160	2000	821	41	1290	65
13C-PCB-118	20 - 160	2000	628	31	729	36
13C-PCB-105	20 - 160	2000	617	31	659	33
13C-PCB-126	20 - 160	2000	878	44	1080	54
13C-PCB-167	20 - 160	2000	712	36	749	37
13C-PCB-156	20 - 160	2000	919 #	46	890 #	44
13C-PCB-157	20 - 160	2000	800 #	40	720 #	36
13C-PCB-169	20 - 160	2000	972	49	912	46
13C-PCB-180	20 - 160	2000	940	47	859	43
13C-PCB-189	20 - 160	2000	973	49	684	34
13C-PCB-209	20 - 160	4000	2220	56	1140	29
CLEANUP STANDARDS						
13C-PCB-81	20 - 160	1000	540	54	843	84
13C-PCB-111	40 - 140	5000	2240	45	3360	67

DL = Detection limits.
# = Value from second column confirmation.
J = Estimated value, below detection limit.

Table 6-28. Summary of Dioxin/Furan Results and Standard Recoveries for Scrubber Water Inlet Samples

ANALYTES	RUN 2 48200-57-02 WI-R2-211A (pg/L)	DL (pg/L)	RUN 3 48200-57-04 WI-R3-311A (pg/L)	DL (pg/L)	RUN 4 48200-57-06 WI-R4-411A (pg/L)	DL (pg/L)
2378-TCDD	ND	12.6	ND	2.59	ND	7.66
12378-PeCDD	ND	27.4	ND	7.06	ND	23.2
123478-HxCDD	ND	12.7	ND	2.06	ND	6.10
123678-HxCDD	ND	13.5	ND	2.22	ND	6.49
123789-HxCDD	ND	12.9	ND	2.11	ND	6.19
1234678-HpCDD	ND	32.8	ND	4.32	ND	12.2
OCDD	40.1		11.7		15.7	
2378-TCDF	ND	9.50	ND	2.07	ND	6.33
12378-PeCDF	ND	17.7	ND	3.80	ND	11.4
23478-PeCDF	ND	11.1	ND	1.58	ND	5.02
123478-HxCDF	ND	14.4	ND	2.29	ND	6.42
123678-HxCDF	ND	12.7	ND	2.25	ND	6.17
123789-HxCDF	ND	17.5	ND	2.50	ND	7.70
234678-HxCDF	ND	14.1	ND	2.26	ND	6.77
1234678-HpCDF	ND	9.31	1.37		ND	3.35
1234789-HpCDF	ND	35.0	ND	4.72	ND	13.5
OCDF	ND	46.7	ND	5.87	ND	15.8
Total Tetra-Furans	91.8		(1.19) J	4.88	ND	14.9
Total Tetra-Dioxins	ND	28.7	(1.72) J	5.89	ND	17.4
Total Penta-Furan	ND	35.5	ND	6.27	ND	19.4
Total Penta-Dioxins	ND	45.9	ND	11.8	ND	38.8
Total Hexa-Furans	ND	53.4	ND	2.26	ND	24.8
Total Hexa-Dioxins	ND	29.2	ND	4.77	ND	14.0
Total Hepta-Furans	ND	19.1	(3.71) J	10.5	(2.93) J	29.4
Total Hepta-Dioxins	ND	98.7	ND	13.0	ND	36.7
Total TEQ	0.04		0.025		0.02	

Table 6-28. (Continued)

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	RUN 2 48190-57-02 WI-R2-211A (pg/sample)	RECOVERY (%)	RUN 3 48190-57-04 WI-R3-311A (pg/sample)	RECOVERY (%)	RUN 4 48190-57-06 WI-R4-411A (pg/sample)	RECOVERY (%)
13C-2378-TCDD	40 - 135	2000	1180	59	670	33 &	714	36 &
13C-12378-PeCDD	40 - 135	2000	1480	74	792	40	802	40
13C-123478-HxCDD	40 - 135	2000	1070	53	698	35 &	773	39 &
13C-123678-HxCDD	40 - 135	2000	1160	58	797	40	902	45
13C-1234678-HpCDD	40 - 135	2000	860	43	726	36 &	830	41
13C-1234678-HpCDD	40 - 135	2000	1540	38 &	1310	33 &	1580	40
13C-OCDD	40 - 135	2000	1220	61	633	32 &	666	33 &
13C-2378-TCDF	40 - 135	4000	1200	60	720	36 &	761	38 &
13C-12378-PeCDF	40 - 135	2000	959	48	756	38 &	753	38 &
13C-23478-PeCDF	40 - 135	2000	1010	50	690	35 &	788	39 &
13C-123478-HxCDF	40 - 135	2000	1095	55	717	36 &	862	43
13C-123678-HxCDF	40 - 135	2000	920	46	703	35 &	765	38&
13C-123789-HXCDF	40 - 135	2000	1030	51	713	36 &	793	40
13C-1234678-HpCDF	40 - 135	2000	820	41	680	34 &	798	40
13C-1234789-HpCDF	40 - 135	2000	760	38 &	623	31 &	725	36 &
CLEANUP STANDARDS								
37Cl-2378-TCDD	40 - 135	200	121	60	132	66	140	70

DL = Detection Limits; {EMPC}.
& = QC value outside the target recovery for Method.
ND = Not detected.
J = Estimated value, below detection limit.

Table 6-29. Summary of Dioxin/Furan Results and Standard Recoveries for Scrubber Water Outlet Samples

ANALYTES	RUN 2 48200-57-03 WO-R2-212A (pg/L)	DL (pg/L)	RUN 3 48200-57-05 WO-R3-312A (pg/L)	DL (pg/L)	RUN 4 48200-57-07 WO-R4-412A (pg/L)	DL (pg/L)
2378-TCDD	9.06 #		(13.3) #,J	4.38	13.5 #	
12378-PeCDD	ND	4.94	ND	13.1	ND	4.12
123478-HxCDD	1.48		ND	3.90	2.61	
123678-HxCDD	2.82		ND	4.02	6.07	
123789-HxCDD	2.78		ND	3.89	5.73	
1234678-HpCDD	11.0		24.6		31.9	
OCDD	29.5		58.7		47.0	
2378-TCDF	181 #		222 #		256 #	
12378-PeCDF	14.3		24.5		25.4	
23478-PeCDF	29.4		45.4		53.1	
123478-HxCDF	11.6		16.4		25.0	
123678-HxCDF	4.56		6.66		10.7	
123789-HxCDF	ND	1.77	ND	4.83	ND	1.68
234678-HxCDF	6.23		12.8		16.5	
1234678-HpCDF	12.4		19.6		25.0	
1234789-HpCDF	ND	3.24	ND	8.33	ND	3.34
OCDF	6.19		10.4		8.16	
Total Tetra-Furans	824		1120		866	
Total Tetra-Dioxins	475		821		277	
Total Penta-Furan	318		493		495	
Total Penta-Dioxins	46.4		57.3		65.6	
Total Hexa-Furans	48.1		80.2		113	
Total Hexa-Dioxins	29.5		53.5		102	
Total Hepta-Furans	17.3		28.9		35.3	
Total Hepta-Dioxins	27.4		52.9		73.6	
Total TEQ	39.6		55.3		61.7	

Table 6-29. (Continued)

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/L)	RUN 2 48190-57-03 WO-R2-212A (pg/L)	RECOVERY (%)	RUN 3 48190-57-05 WO-R3-312A (pg/L)	RECOVERY (%)	RUN 4 48190-57-07 WO-R4-412A (pg/L)	RECOVERY (%)
13C-2378-TCDD	40 - 135	2000	1010 #	50	384 #	19 &	1320 #	66
13C-12378-PeCDD	40 - 135	2000	1220	61	457	23 &	1420	71
13C-123478-HxCDD	40 - 135	2000	1100	55	404	20 &	1220	61
13C-123678-HxCDD	40 - 135	2000	1110	55	466	23 &	1340	67
13C-1234678-HpCDD	40 - 135	2000	1060	53	449	22 &	1130	56
13C-OCDD	40 - 135	4000	2040	51	813	20 &	1960	49
13C-2378-TCDF	40 - 135	2000	1020 #	51	406 #	20 &	1310 #	65
13C-12378-PeCDF	40 - 135	2000	1100	55	408	20 &	1300	65
13C-23478-PeCDF	40 - 135	2000	1120	56	423	21 &	1300	63
13C-123478-HxCDF	40 - 135	2000	1020	51	408	20 &	1200	60
13C-123678-HxCDF	40 - 135	2000	1060	53	425	21 &	1210	61
13C-123789-HXCDF	40 - 135	2000	1070	54	412	21&	1160	58
13C-234678-HpCDF	40 - 135	2000	1050	53	418	21 &	1250	62
13C-1234678-HpCDF	40 - 135	2000	1010	51	403	20 &	1080	54
13C-1234789-HpCDF	40 - 135	2000	976	49	382	19 &	989	49
CLEANUP STANDARDS								
37Cl-2378-TCDD	40 - 135	200	127	63	131	65	139	69

& = QC value outside the target recovery goal for Method.
ND = Not detected, detection limit value provided in adjacent righthand side column.
# = Value from second column configuration.
J = Estimated value, below detection limit.

All of the internal standard recoveries for Run 3 outlet water sample (WO-R3-312A) were below Method 1613 limits. Because the cleanup standard recovery for this sample (65%) is within acceptable limits and similar to recovery of cleanup standard in other outlet samples, the low internal standard recoveries are attributed to inefficient extraction rather than losses during cleanup procedures. The particulate matter found in the scrubber water samples may have contributed to the lower recoveries as the samples required processing through several solid phase extraction (SPE) columns (see Corrective Action Report in Appendix P-3). Low internal standard recoveries can increase the degree of uncertainty about the accuracy of concentrations measured in a sample. However, because the internal standard recoveries for WO-R3-312A (19-23%) did not diverge greatly from the acceptable limit for Method 1613 (25%) and because there was good precision in the concentrations of analytes found between Runs 2, 3 and 4, it is not expected that the low internal standard recoveries for this sample had any significant impact on reported results.

Lab Control Samples and Blanks. Table 6-30 presents D/F results and standard recoveries for the laboratory control spike, laboratory control spike duplicate (LCS/LCSD), and lab blank scrubber water samples. Recovery of all native D/F spiked into the LCS/LCSD were within the 40-135% target range (actual range = 54-111%). The relative percent difference (RPD) between analytes in the duplicates were all under the target <20% except for OCDF (RPD = 57%). Internal standard and cleanup standard recoveries for the LCS/LCSD were all within the 40-135% target and ranged from 47-74%. Only a trace of OCDD was detected in the lab blank. All water sample lab blanks were made using 1 L of HPLC grade reagent water in the laboratory. Low levels of OCDD in blanks are not uncommon due to the prevalence of this analyte in the environment. Internal and cleanup standard recoveries in the lab blank ranged from 53-70% and were all within the target range of 40-135%.

The target detection limit for these samples, based on the lowest calibration level using the Method 1613 calibration curve was 10 pg/L for tetra compounds, 50 pg/L for penta-hepta compounds and 100 pg/L for octa compounds. These detection limits were achieved.

The target turnaround time for the D/F analysis of scrubber water samples was to extract within 30 days of collection and analyze within 45 days of extraction. These turnaround times were met with extractions occurring within 8 days of collection and analysis occurring within 21 days of extraction.

Table 6-30. Summary of Dioxin/Furan Results and Standard Recoveries for Lab Control Spike, Spike Duplicate, and Lab Blank Scrubber Water Samples

ANALYTES	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/L)	LCS 48200-57-08 (pg/L)	RECOVERY (%)	LCS DUP 48200-57-09 (pg/L)	RECOVERY (%)	RECOVERY RPD (%)	LAB BLANK 48200-57-28 (pg/L/)	DL (pg/L)
2378-TCDD	40 - 135	200	223	111	210	105	6	ND	1.52
12378-PeCDD	40 - 135	1000	1050	105	1030	103	1	ND	3.96
123478-HxCDD	40 - 135	1000	964	96	890	89	8	ND	1.11
123678-HxCDD	40 - 135	1000	1010	102	996	100	2	ND	1.21
123789-HxCDD	40 - 135	1000	985	98	974	97	1	ND	1.15
1234678-HpCDD	40 - 135	1000	1020	102	981	98	4	ND	2.08
OCDD	40 - 135	2000	2030	101	1930	97	5	5.30	
2378-TCDF	40 - 135	200	207	101	197	96	5	ND	1.09
12378-PeCDF	40 - 135	1000	1090	109	1060	106	3	ND	2.06
23478-PeCDF	40 - 135	1000	1090	109	1010	101	8	ND	0.830
123478-HxCDF	40 - 135	1000	1040	104	1010	101	3	ND	1.23
123678-HxCDF	40 - 135	1000	1050	105	1030	103	2	ND	1.21
123789-HxCDF	40 - 135	1000	1020	102	961	96	6	ND	1.25
234678-HxCDF	40 - 135	1000	1110	111	1050	106	5	ND	1.15
1234678-HpCDF	40 - 135	1000	1080	108	1040	104	3	ND	0.62
1234789-HpCDF	40 - 135	1000	1060	106	1010	101	5	ND	2.12
OCDF	40 - 135	2000	1940	97	1080	54	57 &	ND	2.66
Total Tetra-Furans			210		197			ND	2.58
Total Tetra-Dioxins			223		210			ND	3.46
Total Penta-Furan			2190		2080			ND	3.34
Total Penta-Dioxins			1050		1030			ND	6.63
Total Hexa-Furans			4220		4060			ND	4.50
Total Hexa-Dioxins			2960		2860			ND	2.60
Total Hepta-Furans			2140		2050			ND	5.05
Total Hepta-Dioxins			1020		981			ND	6.26
Total TEQ			2120		2030			0.005	

Table 6-30. (Continued)

INTERNAL STANDARDS	TARGET RECOVERY (%)	LCS SPIKE CONC. (pg/sample)	LCS 48200-57-08 (pg/sample)	RECOVERY (%)	LCS DUP 48200-57-09 (pg/sample)	RECOVERY (%)	LAB BLANK 48200-57-28 (pg/sample)	DL (pg/sample)
13-2378-TCDD	40 - 135	2000	1190	59	1090	54	1060	53
13C-12378-PeCDD	40 - 135	2000	1480	74	1360	68	1360	68
13C-123478-HxCDD	40 - 135	2000	1360	68	1330	67	1280	64
13C-123678-HxCDD	40 - 135	2000	1440	72	1380	69	1380	69
13C-1234678-HpCDD	40 - 135	2000	1360	68	1310	66	1390	70
13C-OCDD	40 - 135	4000	2560	64	2170	54	2770	69
13C-2378-TCDF	40 - 135	2000	1270	63	1170	59	1140	57
13C-12378-PeCDF	40 - 135	2000	1340	67	1260	63	1260	63
13C-23478-PeCDF	40 - 135	2000	930	47	1430	71	1390	70
13C-123478-HxCDF	40 - 135	2000	1310	65	1270	63	1220	61
13C-123678-HxCDF	40 - 135	2000	1280	64	1280	64	1230	61
13C-123789-HxCDF	40 - 135	2000	1370	69	1260	63	1340	67
13C-234678-HpCDF	40 - 135	2000	1420	71	1370	69	1320	66
13C-1234678-HpCDF	40 - 135	2000	1260	63	1240	62	1260	63
13C-1234789-HpCDF	40 - 135	2000	1260	63	1050	53	1290	65
CLEANUP STANDARDS								
37Cl-2378-TCDD	40 - 135	200	127	64	125	62	117	59

Lab Control Spike % Recovery is calculated as ((concentration found - lab blank conc.)/spike conc.) x 100.
ND = Not detected.
DL = Detection limit.
& = QC value outside the target recovery for Method.

Chlorine Analysis. Samples for chlorine analysis were collected from both inlet and outlet scrubber water systems. Chlorine is metered into the final effluent at a known and constant rate; however, there is no control on the Cl concentration in the effluent utilized by the scrubber water system. MSD expected the chlorine concentration in the effluent water to range from 3 to 4 mg/L. Samples from the first day of sampling were analyzed by the USEPA T&E laboratory and found to contain negligible Cl. MSD analyzed duplicate samples using the amperometric method (APHI Method 4500-Cl-d). Table 6-31 shows that the MSD results verified the results obtained by the T&E Laboratory.

Table 6-31. Intercomparison of Total Chlorine Analyses

Date	Scrubber Water	T&E, mg/L	MSD, mg/L
July 21	In	0.09	0.04
	Out	0.00	0.00
July 22	In	0.08	0.00
	Out	0.03	0.00

6.1.3.3 Sewage Sludge Feed Samples

PCB Analysis. The initial calibration for both the SPB-Octyl and DB-1 analyses met the requirement for response factors having less than 35% relative standard deviation (RSD) for all analytes (actual range = <17% for SPB-Octyl and DB-1 calibrations). The continuing calibrations met the requirement for response factors being within 35% of the response factors generated in the initial calibration for at least 70% of the analytes. In fact, all analytes in all continuing calibrations were within 35% of the initial calibration.

Field Samples. PCB results and standard recoveries for the sewage sludge feed samples are shown in Table 6-32. Internal standard recoveries were within the method specified limits of 20-160% and ranged from 34-62%. These internal standard recoveries indicate analytes were well recovered during laboratory extraction and were retained during the extract cleanup process. $^{13}C_{12}$-PCB-81 and $^{13}C_{12}$-PCB-111 were added to the sewage sludge samples as cleanup standards after extraction but before any cleanup procedures. Method specified recovery ranges for these standards were from 20-160% for $^{13}C_{12}$-PCB-81 and 40-140% for $^{13}C_{12}$-PCB-111. Recovery of

Table 6-32. Summary of PCB Results and Standard Recoveries for Sewage Sludge Samples

ANALYTES	RUN 2 48290-15-08 S-R2-213B (pg/g dry)	DL (pg/g dry)	RUN 3 48190-15-09 S-R3-313B (pg/g dry)	DL (pg/g dry)	RUN 4 48190-15-10 S-R4-413B (pg/g dry)	DL (pg/g dry)
PCB-77	40900		41090		45380	
PCB-123	231 B		276 B		241 B	
PCB-118	12250 B		13500 B		12860 B	
PCB-114	691 B		674 B		738 B	
PCB-105	7010 B		7390 B		7290 B	
PCB-126	1120		1210		1480	
PCB-167	878 B		968 B		959 B	
PCB-156	1770 #		1880 #		1880 #	
PCB-157	472 #		565 #		536 #	
PCB-169	453		601		656	
PCB-180	6000		6780		6710	
PCB-170	2530		2570		2780	
PCB-189	181 B		198 B		218 B	

INTERNAL STANDARDS	TARGET RECOVERY (%)	SPIKE CONC. (pg/sample)	RUN 3 48190-15-08 S-R2-213B (pg/sample)	RECOVERY (%)	RUN 3 48190-15-09 S-R3-313B (pg/sample)	RECOVERY (%)	RUN 4 48190-15-10 S-R4-413B (pg/sample)	RECOVERY (%)
13C-PCB-77	20 - 160	15000	7330	49	6910	46	7300	49
13C-PCB-118	20 - 160	15000	6920	46	7230	48	7380	49
13C-PCB-105	20 - 160	15000	7080	47	7370	49	7540	50
13C-PCB-126	20 - 160	15000	5990	40	6120	41	6250	42
13C-PCB-167	20 - 160	15000	6620	44	6610	44	7220	48
13C-PCB-156	20 - 160	15000	9330 #	62	8860 #	59	9000 #	60
13C-PCB-157	20 - 160	15000	9190 #	61	8540 #	57	8800 #	59
13C-PCB-169	20 - 160	15000	5260	35	5050	34	5490	37
13C-PCB-180	20 - 160	15000	7420	49	7460	50	7960	53
13C-PCB-189	20 - 160	15000	7180	48	7230	48	7680	51
13C-PCB-209	20 - 160	30000	12650	42	13400	45	14600	49
CLEANUP STANDARDS								
13C-PCB-81	20 - 160	6000	2370	40	2180	36	2410	40
13C-PCB-111	40 - 140	30000	13940	46	13300	44	13900	46

DL = Detection limit.
# = Value from second column confirmation.
B = Lab method blank contamination of target analytes at the level above DL.

the cleanup standards were within the limits and ranged from 36-40% for $^{13}C_{12}$-PCB-81 and from 44-46% for $^{13}C_{12}$-PCB-111.

Lab Control and Blank Samples. Table 6-33 presents PCB results and standard recoveries for the matrix spike and matrix spike duplicate (MS/MSD) sewage sludge samples and the lab blank. Recovery of all native PCBs spiked into the MS/MSD were within the 40-160% target and ranged from 83-160%. The relative percent difference (RPD) between duplicates were all under the target <50% and ranged from 1-20 % indicating acceptable analytical precision was achieved. Internal standard recoveries for the MS and MSD were within the acceptable range of 20-160% and ranged from 37-65%. Cleanup standard recoveries for the MS/MSD samples were 40-42% for $^{13}C_{12}$-PCB-81 (target recovery = 20-160%) and 50-53% for $^{13}C_{12}$-PCB-111(target recovery = 40-140%). As with the emission and water samples, the lab blank for the sewage sludge samples contained background levels of PCBs that are not unexpected due to the ubiquitous nature of PCBs. The levels of PCBs detected in the lab blank are typical of background levels detected using the sensitive high resolution mass spectrometry techniques. Because PCBs are detected in the lab blank and because some samples did not contain high levels of PCBs above the background levels there are several instances where the lab blanks did not meet the method performance criteria of being less than 20% of the sample concentration. Results for Runs 2, 3 and 4 are flagged with a "B" in Table 6-32 if the lab blank concentration was greater than 20% of the reported sample concentration. Several internal standard recoveries for the lab blank ($^{13}C_{12}$-PCB-77, $^{13}C_{12}$-PCB-118 and $^{13}C_{12}$-PCB-105) were lower than the acceptable limit of 20-160%. Cleanup standard recoveries were also slightly lower than the acceptable limit with $^{13}C_{12}$-PCB-81 recovered at 15% (target recovery = 20-160%) and $^{13}C_{12}$-PCB-111 recovered at 13% (target recovery = 40-140%). The low cleanup standard recoveries indicate that analyte losses may have occurred during the cleanup process and are not representative of inefficient extraction of analytes in the lab blank. Low internal standard recoveries increase the uncertainty about the accuracy of reported native analyte concentrations; however, the concentrations of PCB-77, PCB-118, and PCB-105 found in the sewage sludge lab blanks when compared to the concentrations of these analytes in the lab blanks for emission and water samples (comparing all on a pg per 20μL extract basis) are very similar. This increases the

Table 6-33. Summary of PCB Standard Recoveries for Matrix Spike, Spike Duplicate, and Lab Blank Sewage Sludge Samples

ANALYTES	TARGET RECOV. (%)	MATRIX SPIKE CONC. (pg/g dry)	MATRIX SPIKE 48190-15-11 S-R4-413B MS (pg/g dry)	RECOV. (%)	MS DUP 48190-15-12 S-R4-413B (pg/g dry)	RECOV. (%)	RECOV. RPD (%)	LAB BLANK 48190-15-15 LB-PCB (pg/g dry)	DL (pg/g dry)
PCB-77	40 - 160	84200	130300	101	115000	83	20	238	
PCB-123	40 - 160	84200	100090	119	99400	118	1	122	
PCB-118	40 - 160	84200	102570	107	88000	89	18	4690	
PCB-114	40 - 160	84200	135300	160	132800	157	2	253	
PCB-105	40 - 160	84200	116600	130	107700	119	8	2130	
PCB-126	40 - 160	8420	11040	114	10420	106	7	ND	176
PCB-167	40 - 160	84200	98450	116	99000	117	1	309	
PCB-156	40 - 160	84200	109500 #	128	106200 #	124	3	296 #	
PCB-157	40 - 160	84200	100000 #	118	102100 #	121	2	89.5 #	
PCB-169	40 - 160	16800	16750	96	17100	97	2	ND	67.9
PCB-180	40 - 160	84200	112500	126	107600	120	5	724	
PCB-170	40 - 160	16800	23600	124	21560	112	10	319	
PCB-189	40 - 160	16800	18800	110	19100	112	2	91.9	

INTERNAL STANDARDS	TARGET RECOV. (%)	MATRIX SPIKE CONC. (pg/sample)	MATRIX SPIKE 48190-15-11 S-R4-413B MS (pg/sample)	RECOV. (%)	MS DUP 48190-15-12 S-R4-413B MSD (pg/sample)	RECOV. (%)	LAB BLANK 48190-15-15 LB-PCB (pg/sample)	RECOV. (%)
13C-PCB-77	20 - 160	15000	7470	50	7830	52	2390	16 &
13C-PCB-118	20 - 160	15000	7870	52	7970	53	2040	14 &
13C-PCB-105	20 - 160	15000	7900	53	8240	55	2300	15 &
13C-PCB-126	20 - 160	15000	6150	41	6640	44	3290	22
13C-PCB-167	20 - 160	15000	7770	52	7490	50	3160	21
13C-PCB-156	20 - 160	15000	9630 #	64	9730 #	65	4350 #	29
13C-PCB-157	20 - 160	15000	9600 #	64	9380 #	63	4030 #	27
13C-PCB-169	20 - 160	15000	5550	37	5640	38	4380	29
13C-PCB-180	20 - 160	15000	7960	53	8150	54	4160	28
13C-PCB-189	20 - 160	15000	7540	50	7570	50	4900	33
13C-PCB-209	20 - 160	30000	14500	48	14900	50	10560	35
CLEANUP STANDARDS								
13C-PCB-81	20 - 160	6000	2500	42	2400	40	872	15 &
13C-PCB-111	40 - 140	30000	15000	50	15800	53	3980	13 &

DL = Detection limit.
Matrix Spike % Recovery is calculated as ((concentration found - Run 4, 48190-1570, S-R4-4138 conc.)/spike conc.) x 100.
& = QC value outside the accuracy of precision goal for Method (40-160% spike recovery of lab spike samples.
# = Value from second column confirmation.

confidence in the accuracy of the concentrations reported for PCB-77, PCB-118, and PCB-105 for the sewage sludge lab blank.

The target detection limit for sewage sludge samples was 55 pg/g dry weight. Actual detection limits achieved ranged from 45-209 pg/g dry weight. Since actual levels of PCBs in the three sewage sludge feed samples are at least two times the detection limits, and since the detection limits achieved were all lower than the lower calibration range for the PCBs, these higher than expected detection limits are not expected to affect the quality of the PCB sludge concentrations reported.

The target turnaround time for the PCB analysis of sewage sludge samples was to extract within 30 days of collection and analyze within 45 days of extraction. These turnaround times were met with extractions occurring within 18 days of collection and analysis occurring within 18 days of extraction.

D/F Analysis. The initial calibration met the requirement for response factors having less than 20% relative standard deviation (RSD) for native analytes and less than 35% RSD for labeled analytes (actual range = <10% for native analytes and < 8% for labeled analytes). All analytes in all continuing calibrations met the requirement for response factors being within 20% of the initial calibration response factors for native analytes and being within 30% of the initial calibration response factors for labeled analytes.

Field Samples. D/F results and standard recoveries for the sewage sludge samples are shown in Table 6-34. All internal standard and cleanup standard recoveries were within the target range of 40-135% (actual range = 60-87%).

Lab Control and Blank Samples. Table 6-35 presents D/F results and standard recoveries for the matrix spike, matrix spike duplicate (MS/MSD), and lab blank sewage sludge samples. Recovery of all native D/F spiked into the MS/MSD were within the 40-135% target range (actual range = 49-65%). The relative percent difference (RPD) between analytes in the duplicates were all under the target <20% and ranged from 0-8%. Internal standard and cleanup standard recoveries for the MS/MSD were all within the 40-135% target and ranged from 60-87%. Only a trace of OCDD was detected in the lab blank. Low levels of OCDD in blanks are not uncommon due to the prevalence of this analyte in the environment. Internal and cleanup standard recoveries in the lab blank ranged from 47-74% and were all within the target range of 40-135%.

Table 6-34. Summary of Dioxin/Furan Results and Standard Recoveries for Sewage Sludge Samples

ANALYTES	RUN 2 48190-15-02 S-R2-213A (pg/g dry)	DL (pg/g dry)	RUN 3 48189-15-03 S-R3-313A (pg/g dry)	DL (pg/g dry)	RUN 4 48190-15-04 S-R4-413A (pg/g dry)	DL (pg/g/dry)
2378-TCDD	(2.75) #,J	14.6	(2.87) #,J	15.6	(5.14) #,J	14.5
12378-PeCDD	ND	15.0	ND	17.0	ND	17.4
123478-HxCDD	ND	4.71	ND	5.40	8.24	
123678-HxCDD	15.0		17.9		30.7	
123789-HxCDD	27.3		{24.3}		39.9	
1234678-HpCDD	229		281		384	
OCDD	2510		2690		3690	
2378-TCDF	20.6 #		24.1 #		35.1 #	
12378-PeCDF	ND	7.75	ND	8.48	12.7	
23478-PeCDF	8.09		9.21		13.4	
123478-HxCDF	{13.7}		19.3		29.6	
123678-HxCDF	ND	5.04	7.51		{10.0}	
123789-HxCDF	ND	5.88	ND	6.64	ND	6.99
234678-HxCDF	6.21		{8.43}		13.3	
1234678-HpCDF	134		159		222	
1234789-HpCDF	ND	11.7	ND	12.6	ND	13.0
OCDF	313		340		441	
Total Tetra-Furans	{76.5}		{95.9}		{120}	
Total Tetra-Dioxins	67.7		82.6		95	
Total Penta-Furan	90.2		95.2		163	
Total Penta-Dioxins	30.3		23.3		29.6	
Total Hexa-Furans	97.7		117		171	
Total Hexa-Dioxins	128		135		{204}	
Total Hepta-Furans	239		284		377	
Total Hepta-Dioxins	431		520		701	
Total TEQ	19.5		22.8		34.6	

Table 6-34. (Continued)

INTERNAL STANDARDS	TARGET RECOV. (%)	SPIKE CONC. (pg/sample)	RUN 2 48190-15-02 S-R2-213A (pg/sample)	RECOV. (%)	RUN 3 48190-15-03 S-R3-313A (pg/sample)	RECOV. (%)	RUN 4 48190-15-04 S-R4-413A (pg/sample)	RECOV. (%)
13-2378-TCDD	40 - 135	2000	1470 #	73	1590 #	79	1600 #	80
13C-12378-PeCDD	40 - 135	2000	1600	80	1740	87	1700	85
13C-123478-HxCDD	40 - 135	2000	1470	73	1590	79	1570	79
13C-123678-HxCDD	40 - 135	2000	1550	77	1580	79	1650	82
13C-1234678-HpCDD	40 - 135	2000	1370	69	1430	72	1470	74
13C-OCDD	40 - 135	4000	2470	62	2720	68	2900	72
13C-2378-TCDF	40 - 135	2000	1390 #	70	1540 #	77	1410 #	71
13C-12378-PeCDF	40 - 135	2000	1520	76	1670	84	1670	83
13C-23478-PeCDF	40 - 135	2000	1440	72	1630	82	1530	77
13C-123478-HxCDF	40 - 135	2000	1410	71	1510	76	1560	78
13C-123678-HxCDF	40 - 135	2000	1460	73	1540	77	1590	79
13C-123789-HxCDF	40 - 135	2000	1360	68	1440	72	1450	72
13C-234678-HpCDF	40 - 135	2000	1490	74	1530	77	1610	80
13C-1234678-HpCDF	40 - 135	2000	1330	67	1410	71	1440	72
13C-1234789-HpCDF	40 - 135	2000	1200	60	1260	63	1290	64
CLEANUP STANDARDS								
37Cl-2378-TCDD	40 - 135	200	121	61	131	65	134	67

DL = Detection Limits; {EMPC}; (Below Detection Limit).
# = Value from second column confirmation.
ND = Not detected.
J = Estimated value, below detection limit.

Table 6-35. Summary of Dioxin/Furan Results and Standard Recoveries for Matrix Spike, Spike Duplicate, and Background Sewage Sludge Samples

ANALYTES	TARGET RECOVERY (%)	MATRIX SPIKE CONC. (pg/g dry)	MATRIX SPIKE 48190-15-05 S-R4-413A MS (pg/g dry)	RECOV. (%)	MS DUP 48190-15-06 S-R4-413A MSD (pg/g dry)	RECOV. (%)	RECOV. RPD (%)	BACKGROUND 48190-15-04 S-R4-413A (pg/g dry)	DL (pg/g dry
2378-TCDD		1400	898	64	922	65	3	(5.14) #,J	14.5
12378-PeCDD		7020	4220	60	4270	61	1	ND	17.4
123478-HxCDD		7020	3470	49	3760	53	8	8.24	
123678-HxCDD		7020	4220	60	4050	57	4	30.7	
123789-HxCDD		7020	4220	60	4000	57	5	39.9	
1234678-HpCDD		7020	4460	58	4450	58	0	384	
OCDD		14000	10660	50	10800	51	2	3690	
2378-TCDF		1400	828	56	862	59	4	35.1 #	
12378-PeCDF		7020	4320	61	4320	61	0	12.7	
23478-PeCDF		7020	4100	58	4140	59	1	13.4	
123478-HxCDF		7020	4210	60	4260	60	1	29.6	
123678-HxCDF		7020	4160	59	4210	60	1	{10.0}	
123789-HxCDF		7020	4020	57	4070	58	1	ND	6.99
234678-HxCDF		7020	4420	63	4410	63	0	13.3	
1234678-HpCDF		7020	4430	60	4440	60	0	222	
1234789-HpCDF		7020	4230	60	4310	61	2	ND	13.0
OCDF		14000	7540	51	7570	51	1	441	
Total Tetra-Furans			865		884			{120}	
Total Tetra-Dioxins			967		985			95	
Total Penta-Furan			8430		8460			163	
Total Penta-Dioxins			4220		4270			29.6	
Total Hexa-Furans			16820		16950			171	
Total Hexa-Dioxins			11910		11800			{204}	
Total Hepta-Furans			8760		8850			377	
Total Hepta-Dioxins			4670		4660			701	
Total TEQ			8380		8450			34.6	

Table 6-35. (Continued)

INTERNAL STANDARDS	TARGET RECOVERY (%)	MATRIX SPIKE CONC. (pg/sample)	MATRIX SPIKE 48190-15-05 S-R4-413B MS (pg/sample)	RECOVERY (%)	MS DUP 48190-15-06 S-R4-413B MSD (pg/sample)	RECOVERY (%)	BACKGROUND 48190-15-04 S-R4-413A (pg/sample)	RECOVERY (%)
13C-2378-TCDD	40 -135	2000	1570	79	1610	80	1600 #	80
13C-12378-PeCDD	40 - 135	2000	1680	84	1740	87	1700	85
13C-123478-HxCDD	40 - 135	2000	1490	75	1530	76	1570	79
13C-123678-HxCDD	40 - 135	2000	1460	73	1620	81	1650	82
13C-1234678-HpCDD	40 - 135	2000	1420	71	1450	72	1470	74
13C-OCDD	40 - 135	4000	2760	69	2850	71	2900	72
13C-2378-TCDF	40 - 135	2000	1390	70	1500	75	1410 #	71
13C-12378-PeCDF	40 - 135	2000	1590	80	1630	82	1670	83
13C-23478-PeCDF	40 - 135	2000	1540	77	1630	81	1530	77
13C-123478-HxCDF	40 - 135	2000	1390	70	1440	72	1560	78
13C-123678-HxCDF	40 - 135	2000	1430	71	1460	73	1590	79
13C-123789-HxCDF	40 - 135	2000	1370	68	1360	68	1450	72
13C-234678-HpCDF	40 - 135	2000	1410	71	1510	76	1610	80
13C-1234678-HpCDF	40 - 135	2000	1310	65	1370	68	1440	72
13C-1234789-HpCDF	40 - 135	2000	1210	60	1230	62	1290	64
CLEANUP STANDARDS								
37Cl-2378-TCDD	40 - 135	200	132	66	141	70	134	67

DL = Detection Limits; {EMPC}; (Below Detection Limit).
Matrix Spike % Recovery is calculated as ((concentration found - (Run 4, 48190-15-04, S-R4-413A conc.)/spike conc.) x 100.
J = Estimated value, below detection limit.
# = Value from second column confirmation.

The target detection limit for these samples, based on the lowest calibration level using the Method 1613 calibration curve was 44 pg/g dry weight for tetra compounds, 217 pg/g dry weight for penta-hepta compounds and 435 pg/g dry weight for octa compounds. Actual detection limits achieved were significantly lower than this.

The target turnaround time for the D/F analysis of sewage sludge samples was to extract within 30 days of collection and analyze within 45 days of extraction. These turnaround times were met with extractions occurring within 18 days of collection and analysis occurring within 10 days of extraction.

6.2 QA PERFORMANCE AUDITS

6.2.1 Field Sampling Audits

6.2.1.1 Dry Gas Meter

The dry gas meter used for MM5 sampling was fully calibrated every six months against a NIST-traceable wet test meter. This calibration audit is documented in Appendix C-2. The full calibration factor Y was used to correct actual metered sample volume to true sample volume. This full calibration was verified by performing a post-test dry gas meter calibration. This post-test calibration is presented in Appendix C-2. The full and post-test calibration coefficients were within 5 percent which met internal QA/QC criteria. The results of the full calibration and post-test calibration are summarized in Table 6-36.

6.2.1.2 Pitot Tube

The Type S pitot tube utilized in the MM-5 sampling train was inspected to ensure that its orientation met the requirements of EPA Method 2. The pitot tube calibration record is documented in Appendix C-2. The pitot tube orientation during all sampling runs met the established criteria for alignment in Figures 2-2 through 2-4, 2-7 and 2-8 of EPA Method 2 in 40 CFR 60, Appendix A.

Table 6-36. QA Results for Dry Gas Meter

Meter Box ID	ETS-12		
Meter Box Gamma	0.9968		
Meter Box dH @	1.6953 in. w.c.		
Full Calibration Date	**7/14/1999**		
Test Meter No.	9548		
Barometric Pressure	29.02 in. Hg		
Previous Gamma	0.9792		
Post-Test Calibration Date	**7/22/1999**		
Audit - Run 2 Audit - Run 3 Audit - Run 4	1.047 1.0718 0.9985	Ave. Cal. Check Error	QA Criteria
Ave. Cal. Check (Yga)	1.039	4.23%	<5%

Notes: Complete calibration data sheets are presented in Appendix C, Sections C-2-1 and C-2-2 for Full Calibration and Post-Test Calibration Audit.

6.2.1.3 Thermocouples

The thermocouples utilized in the MM5 sampling train were checked against an ASTM-traceable thermometer. Calibration log sheets documenting the results of these checks appear in Appendix C-2.

6.2.1.4 Analytical Balance

The gravimetric balance used in the moisture determination (EPA Method 4) was checked against ASTM-traceable weights. Documentation of this performance check is provided in Appendix C-2.

6.2.1.5 Total Hydrocarbon Analyzer

The calibration of the total hydrocarbon analyzer operated by the MSD at the site was verified via a calibration gas audit (CGA), consistent with the procedure of 40 CFR 60, Appendix F, Section 5.1.2.

The total hydrocarbon analyzer was challenged with two audit gases of known propane concentrations based on a span value of 300 ppm. The audit gases were introduced into the sampling system at the sampling probe. A high calibration gas of 124.6 ppm_v and a low calibration gas of 86.6 ppm_v propane were used. Each gas was introduced into the sampling system three times, and the average value was used to calculate analyzer accuracy. Audit results are provided in Table 6-37.

Table 6-37. Total Hydrocarbon Analyzer Audit Results

CEMS Information **Analyzer Type** **Manufacturer** **Model Number** **Serial Number** **Span Value/Range**	**PRE-TEST AUDIT** **Total Hydrocarbon** **Horiba** **MPA-510** **57034007** **300 ppm_{wv}**				**POST-TEST AUDIT** **Total Hydrocarbon** **Horiba** **MPA-510** **57034007** **300 ppm_{dv}**			
Cylinder Gas Information Certified Audit Value (C_2) Cylinder ID Number Type of Certification Certification Date	***Audit Point 1*** *86.6 ppm* *CC90980* *Protocol 1* *04/22/98*		***Audit Point 2*** *124.6 ppm* *CC94773* *Protocol 1* *10/05/98*		***Audit Point 1*** *86.6 ppm* *CC90980* *Protocol 1* *04/22/98*		***Audit Point 2*** *124.6 ppm* *CC94773* *Protocol 1* *10/05/98*	
Audit Results	July 19, 1999		July 19, 1999		July 22, 1999		July 22, 1999	
	(ppm)	Time	(ppm)	Time	(ppm)	Time	(ppm)	Time
Test 1	85.9	11:22	124.1	11:17	82.8	14:47	120.3	14:43
Test 2	85.9	11:28	124.1	11:24	81.0	14:57	119.1	14:57
Test 3	85.9	11:37	124.1	11:33	78.1	15:11	114.6	15:03
Average Response (C_m) **Accuracy (A)**	85.9 ppm		124.1 ppm		80.6 ppm		118.0 ppm	

Accuracy was calculated as a percentage of the audit gas value as stated in Equation 1.1 of 40 CFR 60, Appendix F. During the post-test calibration on July 22, 1999, moisture condensation was observed in the flow panel of the analyzer. The post-test CGA revealed a slower response time and degraded accuracy. However, the accuracy (5.3%) was still within the allowed 15 percent of the audit gas value. The data were thus deemed valid since the accuracy error of the hydrocarbon analyzer did not exceed the QA criteria of ±15 percent.

6.2.1.6 CEM Systems Audit

A performance audit of the CO, CO_2 and O_2 CEMs was conducted on July 22, 1999. This was accomplished by challenging each of the measurement system with an independent reference standard in accordance with procedures described in 40 CFR 60, Appendix F. Results of this audit are presented in Table 6-38.

Results of this audit found that all three analyzers were operating well within the allowable ±10% limit.

Table 6-38. Results of the CEM Audit

	CO_2	O_2	CO
Reference Standard	6.13%	14.2%	467 ppm
Uncorrected Analyzer Reading	5.93% 5.94% 5.90%	14.10% 13.94% 14.25%	443.8 ppm 444.4 ppm 444.3 ppm
Average Concentration	5.92%	14.10%	444.2 ppm
Percent Difference	-3.4	-0.7	-4.9

6.2.2 Laboratory Analysis Audit

EPA provided a blind evaluation sample to Battelle for D/F analysis as a laboratory performance evaluation audit for the test program. This sample was prepared and analyzed in

exactly the same manner as the emission samples, including sample splitting. Results of the analysis of this audit sample are provided in Table 6-39 and Appendix F-3-2.

EPA's evaluation of these audit results concluded that for the reported data, 9 of 11 dibenzodioxins were within the 90% confidence limit including the 2,3,7,8-TCDD isomer. For the dibenzofurans, 11 of 14 dibenzofurans were within the 90% confidence limit. According to EPA the reported data for this audit sample met the acceptable performance limits for the target analytes.

Table 6-39. Results of the Lab Dioxin/Furan Audit

Compound	Auditee Result (ng/sample)	Compound	Auditee Result (ng/sample)
2,3,7,8-TCDD	0.504	2,3,7,8-TCDF	0.662
Other TCDD	7.09	**Other TCDF**	1.015
1,2,3,7,8-PeCDD	0.481	1,2,3,7,8-PCDF	0.586
Other PeCDD	1.291	2,3,4,7,8-PCDF	0.568
1,2,3,4,7,8-HxCDD	0.466	**Other PCDF**	1.061
1,2,3,6,7,8-HxCDD	0.537	1,2,3,4,7,8-HxCDF	0.589
1,2,3,7,8,9-HxCDD	0.564	1,2,3,6,7,8-HxCDF	0.625
Other HxCDD	1.067	1,2,3,7,8,9-HxCDF	0.529
1,2,3,4,6,7,8-HpCDD	1.133	2,3,4,6,7,8-HxCDF	0.604
Other HpCDD	0.622	**Other HxCDF**	1.274
OCDD	1.301	1,2,3,4,6,7,8-HpCDF	1.192
		1,2,3,4,7,8,9-HpCDF	0.953
		Other HpCDF	0
		OCDF	0.615

6.3 QA/QC PERFORMANCE REVIEW

6.3.1 Program Performance Targets and Results

Program performance targets for precision, accuracy, and completeness were specified in the QAPP. These program performance targets and the achieved results are provided in Table 6-40. The detailed discussion of these objectives and the achieved results are found in Section 6.1.3.1.

Table 6-40. Overall Program QA/QC Results

QC Type/ Parameter	Analyte	Program Targets[a]	Achieved Result[a]
Precision	D/Fs	< 50% RSD	<50% for all 2,3,7,8 isomers but 2,3,7,8-TCDD and 1,2,3,7,8-PCDD
	PAHs	< 50% RSD	Not met on any analyte, range 62.30-144.86% RSD. Corrected recoveries for Run 4 would put all <50% except perylene and benzo(a)pyrene
	Toxic PCBs	< 50% RSD	<50% for all but PCB-169, PCB-126, and PCB-77.
Accuracy[b]	D/Fs	40% - 135%	Met (79-134%) except for 3 analytes[c]
	PAHs	50% - 150%	All within range except as specified on Quanterra's report and surrogate spike in field blanks.
	Toxic PCBs	70% - 130%	84-128% except for PCB-114 (FH-146%, LCS DUP BH-167%) LCS DUP
Completeness	D/Fs	100%	100%
	PAHs	100%	100%
	Toxic PCBs	100%	100%

[a] For emission testing only.
[b] Based on recovery of laboratory spikes for emission samples.
[c] 1,2,3,7,8,9-HxCDF, FH-LCS DUP (141%), BH-LCS DUP (147%); 2,3,4,6,7,8-HxCDF, FH-LCS DUP (138%), FH-LCS (138%); OCDF, FH-LCS DUP (136%), BH-LCS DUP (143%).

Table 6-41 indicates the data quality objectives for CEM and moisture parameters for this test program. The objectives for the THC measurement were not specifically addressed in the QAPP since the equipment was operated by the MSD facility. However, the accuracy of the CGA performed is reported in Table 6-41.

Table 6-41. Data Quality Objectives for Precision, Accuracy, and Completeness for CEM and Moisture Field Measurements

Measurement	Precision (RSD)		Accuracy		Completeness	
	Target	Achieved	Target	Achieved	Target	Achieved
Carbon Dioxide (EPA Method 3A)	5%	1.0%	10%	-3.4%	90%	100%
Oxygen (EPA Method 3A)	5%	1.2%	10%	-0.7%	90%	100%
Moisture (EPA Method 4)	Not determinable	--	Not determinable	--	90%	100%
CO (EPA Method 10)	5%	0.8%	10%	-4.9%	90%	100%
THC	5%	Not evaluated	Not defined	-5,3%	Not defined	100%

6.3.2 Method Specific Performance Targets and Results

6.3.2.1 Air Emissions

Detailed discussion of achieved results are contained in Section 6.1.3.1.

PCB Analysis. Performance criteria for the draft PCB emission analytical method and achieved results are listed in Table 6-42.

D/F Analysis. Performance criteria for D/F analysis of emission samples and achieved results are listed in Table 6-43.

PAH Analysis. Target performance criteria for PAH analysis of emission samples and achieved results are listed in Table 6-44.

Table 6-42. Draft PCB Emission Method Performance Target Criteria and Results

Performance Parameter	Target Criteria	Achieved Result
Initial Calibration	<35% RSD	Met
Continuing Calibration	RFs within 35% of initial calibration results for 70% of analytes	Met
Accuracy	70-130% spike recovery for laboratory spike samples	84-128% except for PCB-114 (147-167%)
Precision	≤30% RPD for duplicate laboratory spike samples	Met
Laboratory Method Blank	<20% of sample concentration	<20% for all BH samples; >20% for 10 of 13 PCBs in FH samples[b]
Estimated Detection Limit	100 pg/sample[a]	Met
Sample Turnaround	Extraction within 30 days of collection; analyze within 45 days of extraction	Met

[a] Will vary for each PCB isomer; assumes 50% of MM5 train is used for PCB analysis.
[b] PCB-123 (~1500%), PCB-118 (159%), PCB-114 (1000%), PCB-105 (240%), PCB-126 (114%), PCB-167 (~800%), PCB-156 (520%), PCB-157 (1100%), PCB-169 (93%), PCB-70 (98%), PCB-189 (350%).

Table 6-43. D/F Emission Analysis Performance Target Criteria and Results

Performance Parameter	Target Criteria	Achieved Result
Initial Calibration	5-point; %RSD<20 percent for native standards; <35% for labeled standards	Met
Continuing Calibration	Beginning of each 12-hour shift; RF ±20% of average value from initial calibration	OCDF RF >20% on two of three continuing calibrations
Accuracy	40 - 135% recovery of labeled internal standards in all samples	Met (40-106%)
	40 - 135% recovery of native compounds in laboratory spike sample	Met (79-134%) except for 234678-HxCDF (138%), 123789-HxCDF (141-147%), and OCDF (136-143%)
	40 - 135% recovery of native compounds in emission audit sample	9 of 11 CDD native compounds were within the 90% confidence interval, 11 of 14 CDF native compounds were within the 90% confidence interval.
Precision	<20% RPD for laboratory duplicates	<18% except 23478-PeCDF in one FH sample (40%)
Laboratory Method Blank	<3 x target detection limit	Met
Sample Turnaround	Extracted within 30 days; analyzed within 45 days of extraction; or to meet test report delivery date	Met

Table 6-44. PAH Emission Performance Target Criteria and Results

Performance Parameter	Target Criteria	Achieved Result
Initial Calibration	5-point; %RSD ≤30 percent for unlabeled and labeled standards	Met; <20% in all cases
Continuing Calibration	Beginning of each 12-hour shift; RF ± 30% of average value from initial calibration	Met except on 8-30-99, RF of 77.3% for fluorene
Accuracy	50-150% recovery of labeled internal standards and surrogate standards	Generally within range except for three internal standards; and surrogate level in field blanks >250%
Precision	RPD<50% for ongoing analysis of test 2 duplicate samples	Maximum RPD 22% for anthracene in BH controls
Laboratory Method Blank	Target analytes ≤PQL or 5 percent of analyte concentration in emission samples	Limit of detection reported as maximum of five times blank level or 10 ng/split. Reporting limit of 20 ng in most cases except as noted in appendix for naphthalene, phenanthrene which varied with samples. All analytes in blanks below reporting levels
Sample Turnaround	Extract within 30 days and analyze within 45 days of extraction; or to meet test report delivery date	Met

6.3.2.2 Scrubber Water Samples

PCB Analysis. Target performance criteria for the draft PCB scrubber water method and achieved results are listed in Table 6-45.

Table 6-45. Draft PCB Scrubber Water Method Performance Criteria

Performance Parameter	Target Criteria	Achieved Result
Initial Calibration	<35% RSD	Met
Continuing Calibration	RFs within 35% of initial calibration results for 70% of analytes	Met
Accuracy	40-160% spike recovery for laboratory spike samples	54-141% except PCB77 in LCS (166%)
Precision	≤50% RPD for duplicate laboratory spike samples	Met (≤48%)
Laboratory Method Blank	<20% of sample concentration	>20% for 10 of 13 PCBs (outlet) and all 13 PCBS (inlet)[b]
Detection Limit	25 pg/L[a]	1.35-13 pg/L except LCS
Sample Turnaround	Extraction within 30 days of collection; analysis within 45 days of extraction	Met

[a] Will vary for each PCB isomer; assumes 50% of scrubber water extract is used.
[B] PCB-77 (25%), PCB-123 (124%), PCB-118 (89%), PCB-114 (132%), PCB-105 (85%), PCB-126 (70%), PCB-167 (105%), PCB-156 (91%), PCB-157 (99%), PCB-169 (78%), PCB-180 (74%), PCB-170 (69%), PCB-189 (95%).

D/F Analysis. Target performance criteria for D/F analysis of scrubber water samples and achieved results are listed in Table 6-46.

Table 6-46. D/F Scrubber Water Analysis Performance Target Criteria and Results

Performance	Target Criteria	Achieved Result
Initial Calibration	5-point; %RSD<20 percent for native standards; <35% for labeled standards	Met
Continuing Calibration	Beginning of each 12-hour shift; RF ±20% of average value from initial calibration	Met
Accuracy	40-135% recovery of labeled internal standards in all samples	31-74% excluding sample WO-R3-312A
	40-135% recovery of native compounds in laboratory spike sample	Met (54-111%)
Precision	<20% RPD for laboratory duplicates	≤8% except OCDF (57% RPD)
Laboratory Method Blank	<3 x target detection limit	Met
Sample Turnaround	Extracted within 30 days; analyzed within 45 days of extraction; or to meet test report delivery date	Met

6.3.2.3 Sewage Sludge Feed Samples

PCB Analysis. Target performance criteria for the draft PCB sewage sludge method and achieved results are listed in Table 6-47.

Table 6-47. Draft PCB Sewage Sludge Method Performance Criteria

Performance Parameter	Target Criteria	Achieved Result
Initial Calibration	<35% RSD	Met
Continuing Calibration	RFs within 35% of initial calibration results for 70% of analytes	Met
Accuracy	40-160% spike recovery for laboratory spike samples	Met (96-157%)
Precision	≤50% RPD for duplicate laboratory spike samples	Met (≤20% RPD)
Laboratory Method Blank	<20% of sample concentration	Met for 5 of 11 PCBs (>20% for six PCBs)[b]
Detection Limit	55 pg/g (dry weight)[a]	45-169 pg/g
Sample Turnaround	Extraction within 30 days of collection; analysis within 45 days of extraction	Met

[a] Will vary for each PCB isomer; assumes 30% of sewage sludge extract is used for analysis.
[b] PCB-123 (50%), PCB-118 (36%), PCB-114 (36%), PCB-105 (29%), PCB-167 (33%), PCB-189 (46%).

D/F Analysis. Target performance criteria for D/F analysis of sewage sludge feed samples are listed in Table 6-48.

Table 6-48. D/F Sewage Sludge Feed Analysis Performance Target Criteria and Results

Performance Parameter	Target Criteria	Achieved Result
Initial Calibration	5-point; %RSD < 20 percent for native standards; < 35% for labeled standards	Met
Continuing Calibration	Beginning of each 12-hour shift; RF ± 20% of average value from initial calibration	Met
Accuracy	40-135% recovery of labeled internal standards in all samples	Met (54-87%)
	40-135% recovery of native compounds in laboratory spike sample	Met (51-65%)
Precision	< 20% RPD for laboratory duplicates	Met (≤8%)
Laboratory Method Blank	< 3 x target detection limit	Met
Sample Turnaround	Extracted within 30 days; analyzed within 45 days of extraction; or to meet test report delivery date	Met

TECHNICAL REPORT DATA

(Please read Instructions on reverse before completing)

1. REPORT NO. EPA-454/R-00-038b	2.	3. RECIPIENT'S ACCESSION NO.
4 TITLE AND SUBTITLE Source Characterization For Sewage Sludge Incinerators Final Emissions Report, Volume I of III Metropolitan Sewer District (MSD) Mill Creek Wasterwater Treatment Plant Cincinnati, Ohio		5. REPORT DATE September 2000 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Clyde E. Riley, USEPA Anthony S. Wisbith, Battelle Jeffery A. Ferg, Battelle Dennis A. Falgout, PES		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Battelle 505 King Avenue Columbus, Ohio 43201-2693		10. PROGRAM ELEMENT NO. 11. CONTRACT/GRANT NO. 68-D-99-009
12. SPONSORING AGENCY NAME AND ADDRESS Emissions, Monitoring and Analysis Division Office of Air Quality Planning and Standards U.S. Environmental Protection Agency Research Triangle Park, North Carolina 27711		13 TYPE OF REPORT AND PERIOD COVERED Final; January 99 to September 2000 14 SPONSORING AGENCY CODE EPA/200/04

15 SUPPLEMENTARY NOTES

16 ABSTRACT

The Clean Air Act Amendments of 1990 require the U.S. Environmental Protection Agency's (EPA) Office of Air Quality Planning and Standards (OAQPS) to establish standards of performance for sewage sludge incineration. These standards are necessary to protect public health and the environment from any adverse effects of pollutant emissions from sewage sludge incineration. The regulations will contain general regulatory requirements, pollutant characterization, and emission limits. To assess control technologies as well as associated strategies for cost-effective standards, EPA requires data on PCB, D/F, and PAH emissions from sewage sludge incinerators . While some emission data exist for sewage sludge incinerators, data on coplanar polychlorinated biphenyls (PCBs) from sewage sludge incinerators are very limited.

The test report summarizes testing of a multiple hearth incinerator at the Metropolitan Sewer District (MSD) Mill Creek Wastewater Treatment Plant in Cincinnati, Ohio in July, 1999. The emission data collected in this test program will be used by EPA/OAQPS and EPA's Office of Water (OW) to support a decision about further data gathering efforts in support of MACT standards for sewage sludge incinerators. During the testing, a second EPA contractor monitored and recorded the process and emission control system operating parameters, and prepared Section 4.0, Process Description And Operation of the report. The report consist of five documents: Executive Summary Report; Volume I-Main Report; Volume II-Appendices A-J; Volume III-Appendices K-P; and a Data Quality Assessment Report.

17 KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
PCBs PAHs Dioxins/furans	Air Pollution control	
18. DISTRIBUTION STATEMENT Release Unlimited	19. SECURITY CLASS *(Report)* Unclassified	21. NO OF PAGES
	20 SECURITY CLASS *(Page)* Unclassified	22. PRICE

EPA Form 2220-1 (Rev. 4-77)