



November 15, 1999

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RE: Mercury Information Collection Request
Presque Isle Power Plant
Unit 5 and 9 Mercury Speciation Test Report

Dear Mr. Grimley/Ms. Autry

Enclosed are (3) copies of the test report for the Information Collection Request (ICR) test program performed on Units 5 and 9 at the Presque Isle Power Plant. The test program was conducted by Roy F. Weston, Inc., the Energy & Environmental Research Center (EERC) and CONSOL, R&D during the period of July 12 through 20, 1999. The report was prepared by Roy F. Weston, Inc.

Please Contact me at (414) 221-2293 with any questions regarding this submittal.

Sincerely

A handwritten signature in cursive script that reads 'Terry Coughlin'.

Terry Coughlin
Air Quality Team Leader

**CONTRACT NO. DE-AC-22-93PC93255
WESTON PROJECT NO. 20009.001.006.0500
EMISSIONS TEST REPORT
WISCONSIN ELECTRIC POWER COMPANY
PRESQUE ISLE POWER PLANT
MARQUETTE, MICHIGAN**

NOVEMBER 1999

**DOE PROJECT:
INFORMATION COLLECTION REQUEST
ASSESSMENT OF SPECIATED MERCURY
EMISSIONS FROM TWO COAL-FIRED BOILERS**

Prepared for:

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EXECUTIVE SUMMARY

This test report presents the results of the speciated mercury test program performed on Units 5 and 9 at the Wisconsin Electric Power Company's (WEPCOs) Presque Isle Power Station.

The test program was sponsored by WEPCO and the U.S. Department of Energy (DOE). The work was completed by Roy F. Weston, Inc. (WESTON®), the Energy & Environmental Research Center (EERC), and CONSOL, R&D (CONSOL). The test program was performed during the period of 12 through 20 July 1999.

The test was performed to satisfy the U.S. Environmental Protection Agency (EPA), Office of Air Quality Planning Standards (OAQPS), Information Collection Request (ICR) requirements. Additional testing and analysis (not required as part of the ICR) was performed during this test program. This additional data was collected to further validate the ICR measurements, evaluate mercury continuous emission monitoring systems (CEMs) and obtain additional data to support the Lake Superior region mercury study.

During the test program mercury emissions testing using the Ontario Hydro method were performed on the inlet and outlet of the electrostatic precipitators (ESPs) serving Units 5 and 9. Representative samples of the coal and ESP ash streams were sampled in conjunction with the emissions testing. Mercury CEMs measured mercury concentrations at the ESP outlets (stacks) of both Units 5 and 9.

Table ES-1 presents a summary of the average speciated mercury concentrations and mass rate results for the Unit No. 5 and 9 ESP inlet and outlet test locations. In addition, the average percent of particulate bound, oxidized, and elemental mercury in comparison to the total mercury are provided. Also presented on Table ES-1 are the measured mercury removal efficiencies and calculated mercury material balance for the tests performed on Unit No. 5 and 9.

Detailed discussions and presentations of all test data and test results are provided in Section 3 of this report.

TABLE ES-1
SUMMARY OF MERCURY SPECIATION TEST RESULTS
UNIT NOS. 5 and 9

PARAMETERS	Unit 5 Inlet		Unit 5 Outlet		Unit 9 Inlet		Unit 9 Outlet	
	Average of Test Runs	Average % of Total	Average of Test Runs	Average % of Total	Average of Test Runs	Average % of Total	Average of Test Runs	Average % of Total
PROCESS DATA:								
Unit Load, MW	79.5	--	79.5	--	84.3	--	84.3	--
Coal feed rate, lb/hr.	72,933		72,933		101,200		101,200	
Coal Btu content, Btu/lb. (as received)	12,107		12,107		9,541		9,541	
Heat Input, 10 ⁶ Btu/hr (F-Factor)	869.6		870		996.7		996.7	
PARTICULATE BOUND MERCURY EMISSIONS:								
Conc., ug/m ³	3.88	84.8	0.009	0.6	0.04	0.2	0.0029	0.0
Conc., ug/Nm ³	4.17		0.010		0.05		0.003	
Emission rate, lbs/10 ¹² Btu.	3.17		0.008		0.03		0.002	
Emission rate, lbs/hr.	2.75E-03		6.81E-06		2.97E-05		2.13E-06	
OXIDIZED MERCURY EMISSIONS:								
Conc., ug/m ³	0.47	10.4	0.66	42.6	0.13	1.8	0.57	8.4
Conc., ug/Nm ³	0.50		0.70		0.14		0.61	
Emission rate, lbs/10 ¹² Btu.	0.38		0.54		0.09		0.42	
Emission rate, lbs/hr.	3.31E-04		4.67E-04		9.13E-05		4.14E-04	
ELEMENTAL MERCURY EMISSIONS:								
Conc., ug/m ³	0.31	4.8	0.87	56.7	7.03	97.9	6.19	91.6
Conc., ug/Nm ³	0.34		0.94		7.54		6.64	
Emission rate, lbs/10 ¹² Btu.	0.25		0.72		4.84		4.52	
Emission rate, lbs/hr.	2.21E-04		6.20E-04		4.83E-03		4.50E-03	
TOTAL MERCURY EMISSIONS:								
Conc., ug/m ³	4.56	--	1.54	--	7.17	--	6.76	--
Conc., ug/Nm ³	4.89		1.65		7.70		7.25	
Emission rate, lbs/10 ¹² Btu.	3.71		1.26		4.95		4.93	
Emission rate, lbs/hr.	3.23E-03		1.09E-03		4.93E-03		4.92E-03	
TOTAL MERCURY REMOVAL EFFICIENCY:								
	--	--	66.0%	--	--	--	1.47%	--
MERCURY MATERIAL BALANCE (1):								
	--	--	90.1%	--	--	--	87.3%	--

(1) Based on total mercury in coal, compared to mercury measured in ESP ash and at ESP outlet

1. INTRODUCTION

1.1 SUMMARY OF THE TEST PROGRAM

The U.S. Environmental Protection Agency (EPA), Office of Air Quality Planning and Standards (OAQPS) has undertaken a program to acquire information related to mercury emissions from electric utility steam generating units. As part of this Information Collection Request (ICR), EPA has selected certain utilities for emissions testing to characterize speciated mercury emissions and the effectiveness of available control measures on such emissions. In addition, the U.S. Department of Energy (DOE), EPA, and the Electric Power Research Institute (EPRI) have collectively undertaken a study to determine the fate of mercury in the Lake Superior region. This test program was designed to satisfy the requirements of the ICR and obtain additional data to support the Lake Superior region mercury study.

Wisconsin Electric Power Company's (WEPCOs) Presque Isle Power Station, located near Marquette, Michigan, was selected as one of the study sites. Mercury speciation sampling was performed at the Presque Isle facility using the Ontario Hydro method. The work was completed by Roy F. Weston, Inc., (WESTON), the Energy & Environmental Research Center (EERC), and CONSOL, R&D (CONSOL). The mercury speciation sampling activities were performed by WESTON. The EERC completed the mercury analysis and used mercury continuous emission monitoring systems (CEMS) to provide continuous mercury measurements. CONSOL was responsible for collection and analysis of the coal and ash samples.

The test program was performed during the period of July 12 through 20, 1999.

This test report presents the test data and test results of the mercury speciation sampling program performed at the Presque Isle Power Station. This report contains all test results and discussions for the speciated mercury testing performed on Units 5 and 9. Appendices of the detailed test data and test results, raw test data, process data, laboratory reports, equipment calibration records and sample calculations are also provided.

Per the requirements of the ICR and to satisfy the specific DOE contract requirements the report format followed EPA's Emissions Measurement Center (EMC) guideline document (GD-043) titled, Preparation and Review of Emission Test Reports.

1.2 TEST PROGRAM OBJECTIVES

During the test program mercury emissions testing using the Ontario Hydro method were performed on the inlet and outlet of the electrostatic precipitators (ESPs) serving Units No. 5 and 9. Mercury continuous emission monitoring systems (CEMS) measured mercury concentrations at the ESP outlets (stacks) of both Units 5 and 9. Representative samples of the coal and ash streams were sampled in conjunction with the emissions testing.

The specific objectives of this test program were as follows:

- Characterize the emissions of particulate-bound, elemental and oxidized mercury from the two coal fired boilers.
- Simultaneously measure concentrations and mass rates of speciated mercury at the inlet and outlet of the ESP on each of the two coal fired boilers.
- Obtain and analyze representative samples of the coal and ESP ash streams for the purpose of determining mercury levels and to establish a material balance for mercury.
- Obtain and analyze representative samples of the coal for the purpose of determining heating value, ash content, sulfur and chlorine levels.
- Determine the carbon content of the ESP ash streams.
- Perform mercury measurements using three mercury CEMS in conjunction with mercury testing at the ESP outlet (stack) on each of the two units.
- Document corresponding boiler and ESP operations along with facility CEMS data.

The sampling, analytical and Quality Assurance (QA) procedures used during this test program were documented in the Site-Specific Sampling/Testing, Analytical and QA/QC Plan and in the Quality Assurance Project Plan (QAPP) dated May 1999. Although not a specific objective of the original program the major ash elements for both the coal and ash ESP samples were determined to provide additional characterization of the coal and ESP ash samples.

1.3 SAMPLE LOCATIONS

Representative samples from the following two solid streams were collected and analyzed on each boiler:

- Clean Coal Feed.
- ESP Ash.

Flue gas stream emission samples were collected at the following two locations on each boiler.

- ESP Inlet.
- ESP Outlet (Stack)

1.4 POLLUTANTS MEASURED

Table 1-1 presents a summary of process solid and flue gas streams and the associated pollutants and parameters measured during the test program.

1.5 TEST PROGRAM KEY PERSONNEL

The key personnel who coordinated and performed the test program, their project responsibilities and their phone numbers are:

Contact Name	Project Responsibility	Telephone No.	Facsimile No.
WEPCO			
Mr. Dave Michaud	Corporate Environmental Contact	(414) 221-2187	(414) 221-2169
Mr. Todd Rapavi	Plant Environmental Contact	(906) 226-5738	(906) 226-5750
DOE			
Mr. Tom Brown	Project Manager	(412) 386-4691	(412) 386-5917
EPA			
Mr. William Grimley	ICR Program Manager	(919) 541-1065	(919) 541-1039
WESTON			
Mr. Jeff Burdette	Program Manager	(919) 462-6921	(919) 462-6901
Mr. Jeff O'Neill	Project Leader	(610) 701-7201	(610) 701-7401
EERC			
Mr. Dennis Laudal	Project Leader	(701) 777-5138	(701) 777-5181
CONSOL			
Mr. Matt Devito	Project Leader and Process Observer	(412) 854-6679	(412) 854-6613

Table 1-1
Presque Isle Power Station
Units 5 and 9
Process Solid and Flue Gas Streams with
Pollutants/Parameters

Location/Stream Type	Pollutants or Parameters	Frequency
Units 5 and 9 Clean Coal Feed	Heating value Moisture Ultimate and proximate analyses Mercury (Hg) content Chlorine (Cl) content Major ash elements	One composite sample per run in conjunction with flue gas sampling. Six (6) total (3 per unit).
Units 5 and 9 ESP Ash	Mercury content Carbon content Major ash elements	Grab samples obtained in conjunction with flue gas sampling. Six (6) total (3 per unit).
Units 5 and 9 ESP inlet and Outlet (Stack)	Particulate bound and vapor phase mercury (including oxidized and elemental mercury speciation of vapor phase). Particulate loading ¹	Inlet and outlet sampling by Ontario Hydro method. Mercury measurements by CEMS on the Unit 5 and 9 stacks (ESP outlet only).

(1) Based on pre and post test weights of Ontario Hydro method thimbles. This data was used to approximate the ash collected by each ESP and support material balance calculations.

2. PLANT AND SAMPLING LOCATION DESCRIPTIONS

2.1 PRESQUE ISLE POWER PLANT OVERVIEW

The Presque Isle Power Station has nine boilers. Flue gas from Units 1 through 4 are controlled by two newly installed baghouses. Baggouse A controls the emissions from Units 1 and 3 and baghouse B controls the emissions from Units 2 and 4. The flue gas from both baghouses are combined and exhausted through a single stack. Flue gas from Units 5 and 6 pass through conventional electrostatic precipitators (ESPs). Units 5 and 6 have dedicated ESPs. Units 5 and 6 are identical in design and configuration with the exception of an add-on control device to the Unit 6 ESP. The add-on control device, known as an agglomerator, is designed to increase the ESP particulate collection efficiency by clustering smaller particles into larger masses which should be easier for the ESP to capture and remove. The agglomerator has been in operation on Unit 6 since June of 1998.

Particulate matter from Units 7 through 9 are controlled by individual hot side ESPs.

Presently Units 1 through 6 burn an eastern (Sanborn Creek) coal mixed with ten percent petroleum coke. The coal fired in Units 7, 8 and 9 is a blend of western coal (~45% Spring Creek region and ~55% Antelope region).

2.2 PROCESS SOLID SAMPLING LOCATIONS AND SAMPLING PROCEDURES

2.2.1 Unit 5 and 9 Coal Sampling

Representative pulverized coal samples from Units 5 and 9 were obtained from burner feed pipes located immediately downstream of the coal mills. There are four mills, each with two feed pipes, supplying coal to the burners. Each pipe was equipped with a 1½ inch ball valve. Sampling was conducted through these valves as described in ASTM D197. In this technique a sampling pipe with a tapered nozzle was inserted through the ball valve. The sampling pipe is connected to a cyclone collector. A sample receiver bottle is attached to the collection cyclone. An air eductor is used to produce a sample flow rate. The sample is obtained by adjusting the air flow through the eductor and then by traversing the coal feed pipe with the sampling probe. The

entire pipe is sampled utilizing a uniform rate of movement for a period of 1 minute. This resulted in an average sample increment size of ~500 grams. Eight sample increments were obtained (one from each feed pipe) for each test period at ~20 minute intervals. The first sample increment was taken during the start of the flue gas sampling and the final sample increment was obtained near the completion of the flue gas sampling. The condition of the sampling equipment was checked and verified prior to each use as was the operating status of the boiler. All of the sample increments were stored in a double-lined plastic bag that was further sealed in an air-tight plastic bucket. The individual composite coal samples were shipped from the plant to the CONSOL laboratory located in Library, Pennsylvania by CONSOL personnel. The samples were then logged into the laboratory and analytical request sheets were completed. The samples were size-reduced and analyzed by standard ASTM methods.

2.2.2 Unit 5 ESP Ash Hopper Sampling

Unit 5 is equipped with a cold-side ESP. The ESP has two rows of hoppers. Each row has three collection hoppers. Each hopper is fitted with an access pipe located near the bottom of the hopper and angled toward the base of the hopper. Ash sampling was conducted through these access pipes. The hoppers were cleaned out by plant personnel just prior to the start of flue gas sampling. After clean-out, the hoppers were allowed to fill with ash. A fly ash sample was collected from each hopper utilizing a sample thief inserted into the base of the hopper through the access pipes. The three front row of samples were combined into one composite (front row sample). The three back row of samples were combined to make a separate composite (back row sample). Individual front and back row samples were obtained for each flue gas measurement period. The composite samples were size-reduced on-site, transferred to pre-labeled glass bottles, and transported to the CONSOL laboratory by CONSOL personnel. The samples were logged into the laboratory database. Analytical request sheets were completed and the samples were analyzed by standard ASTM procedures.

2.2.3 Unit 9 ESP Hopper Ash Sampling

Unit 9 is equipped with a hot side ESP. The ESP has three rows of hoppers. Each row has four collection hoppers. Due to safety concerns, ash samples were collected by WEPCO plant

personnel following a similar procedure to Unit 5. The hoppers were cleaned out just prior to the flue gas sampling period. Fly ash was allowed to accumulate throughout the flue gas sampling period. Near the completion of the flue gas measurements, ash samples were collected from the middle hoppers of the first row. This was accomplished by opening a clean-out window located near the discharge pipe of the hopper. One grab sample from the two inner first hoppers were obtained for each flue gas sampling period. These samples were size-reduced on-site, transferred to pre-labeled glass sample bottles, and transported to the CONSOL laboratory by CONSOL personnel. The samples were logged into the laboratory database. Analytical request sheets were completed and the samples were analyzed by standard ASTM procedures.

2.3 FLUE GAS SAMPLING LOCATIONS

2.3.1 Unit 5 ESP Inlet

The test site at the ESP inlet is vertically oriented and is a 7' deep by 21' 5" wide rectangular duct. The four (4) sample ports are positioned on the side of the duct immediately upstream of the ESP inlet. The ports are located <1.0 diameters upstream and downstream of the nearest gas flow disturbances. This test location does not satisfy the minimum requirements of EPA Method 1; however, it is the only test location at the inlet of the ESP suitable for sampling.

Of the four test ports present, only the two middle ports were accessible and used for sampling.

A total of six (6) traverse points per each of the two test ports (total of 12) were sampled. See Figure 2-1 for a schematic of the Unit 5 ESP inlet.

2.3.2 Unit 5 ESP Outlet (Stack)

A total of four (4) 6" test ports are in place on the 9' ID flue, which serves Unit 5. The test ports are located ~100' (11.1 diameters) from the nearest downstream disturbance and ~200' (22.2 diameters) from the nearest upstream disturbance. A total of 3 points per port (6 per axis, 12 total) were sampled. The Unit No. 5 stack is an ideal test location and satisfies all EPA Method 1 criteria for test port location. See Figure 2-2 for a schematic of the Unit No. 5 stack test location.

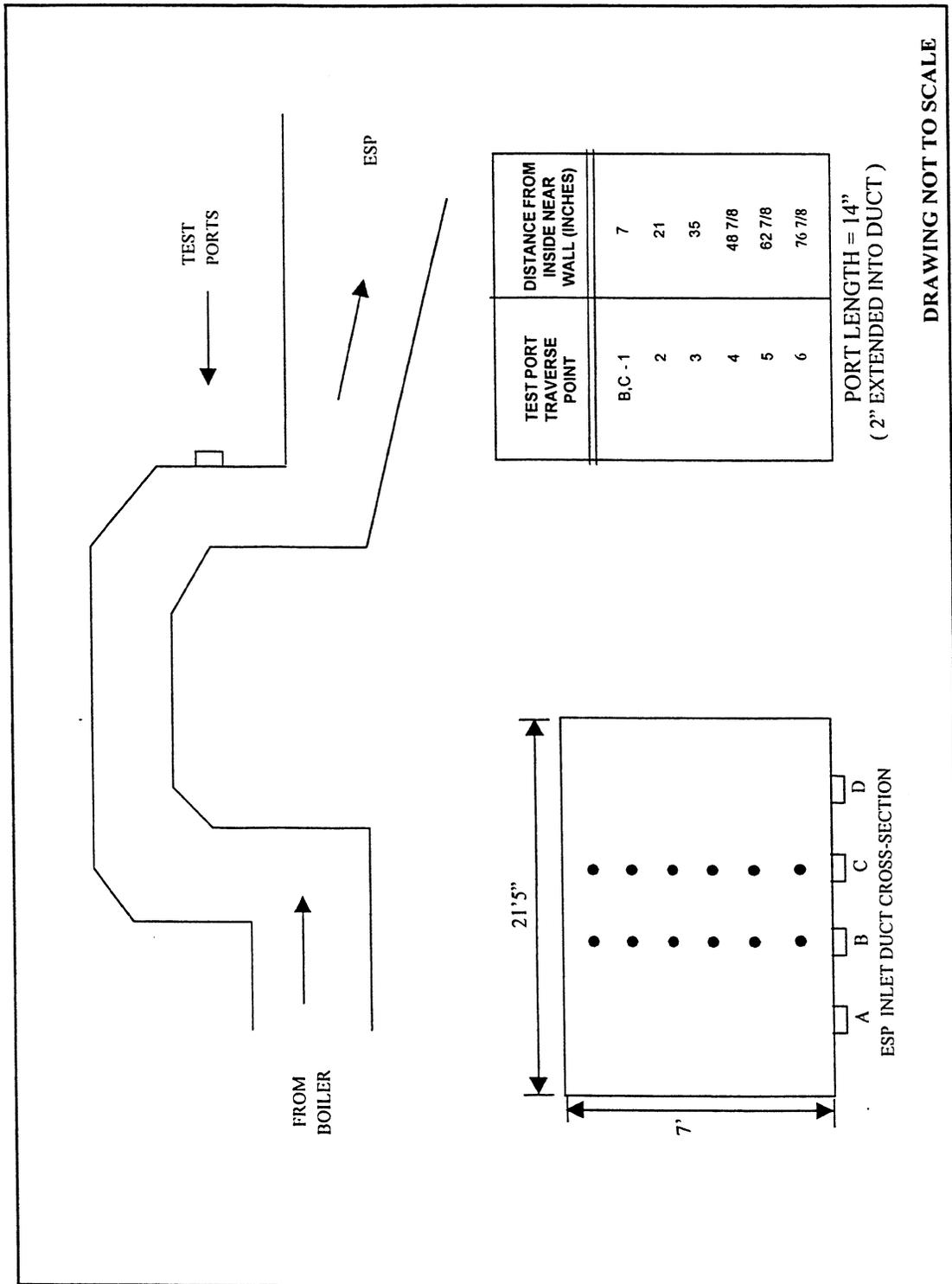


FIGURE 2-1
UNIT 5 ESP INLET DUCT TEST SITE
PORT AND TRAVERSE POINT LOCATIONS

2.3.3 Unit 9 ESP Inlet

A total of fourteen (14) test ports are located on the 7'x 26'5" rectangular duct. The test ports are located on the exit of the boiler upstream of the hot side ESP inlet. The ports are located <1.0 diameters upstream and downstream of the nearest gas flow disturbances. This test location does not satisfy the minimum requirements of EPA Method 1; however, it is the only test location at the inlet of the ESP suitable for sampling. During each test run a total of five traverse points were sampled in every other port (seven ports for a total of 35 points).

See Figure 2-3 for a schematic of the Unit 9 ESP inlet.

2.3.4 Unit 9 ESP Outlet (Stack)

A total of four (4) 6" ID test ports are in place on the 9'6" ID flue which serves Unit 9. The test ports are located ~100' (10.5 diameters) from the nearest downstream disturbance and ~200' (21.2 diameters) from the nearest upstream disturbance.

Two of the four test ports were used for sampling. A total of six points per port (12 points total) were sampled. The Unit No. 9 stack is an ideal test location and satisfies all EPA Method 1 criteria for test port location. See Figure 2-4 for a schematic of the Unit No. 9 stack test location.

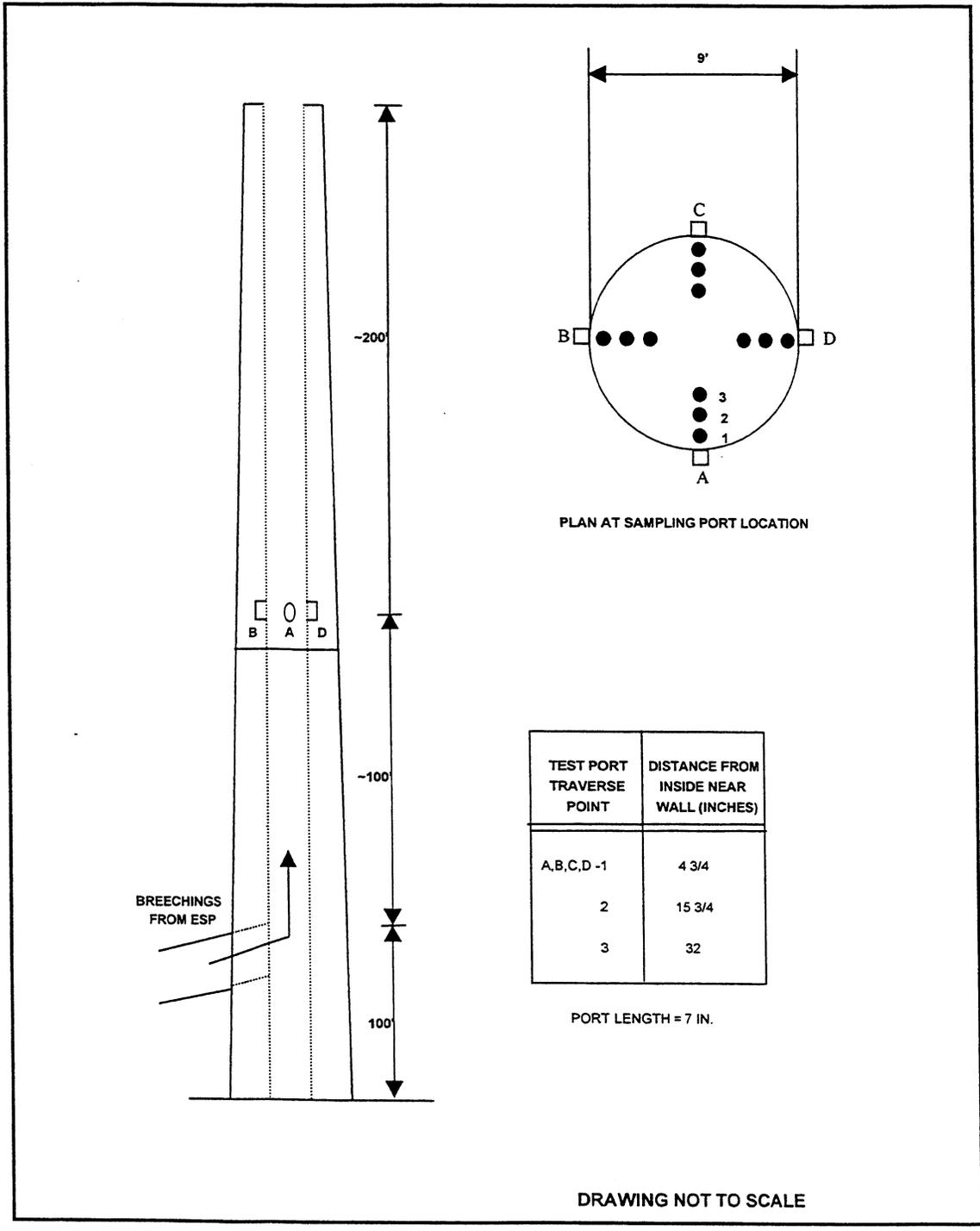


FIGURE 2-2
UNIT 5 STACK TEST SITE
PORT AND TRAVERSE POINT LOCATIONS

DOE1_03-D20

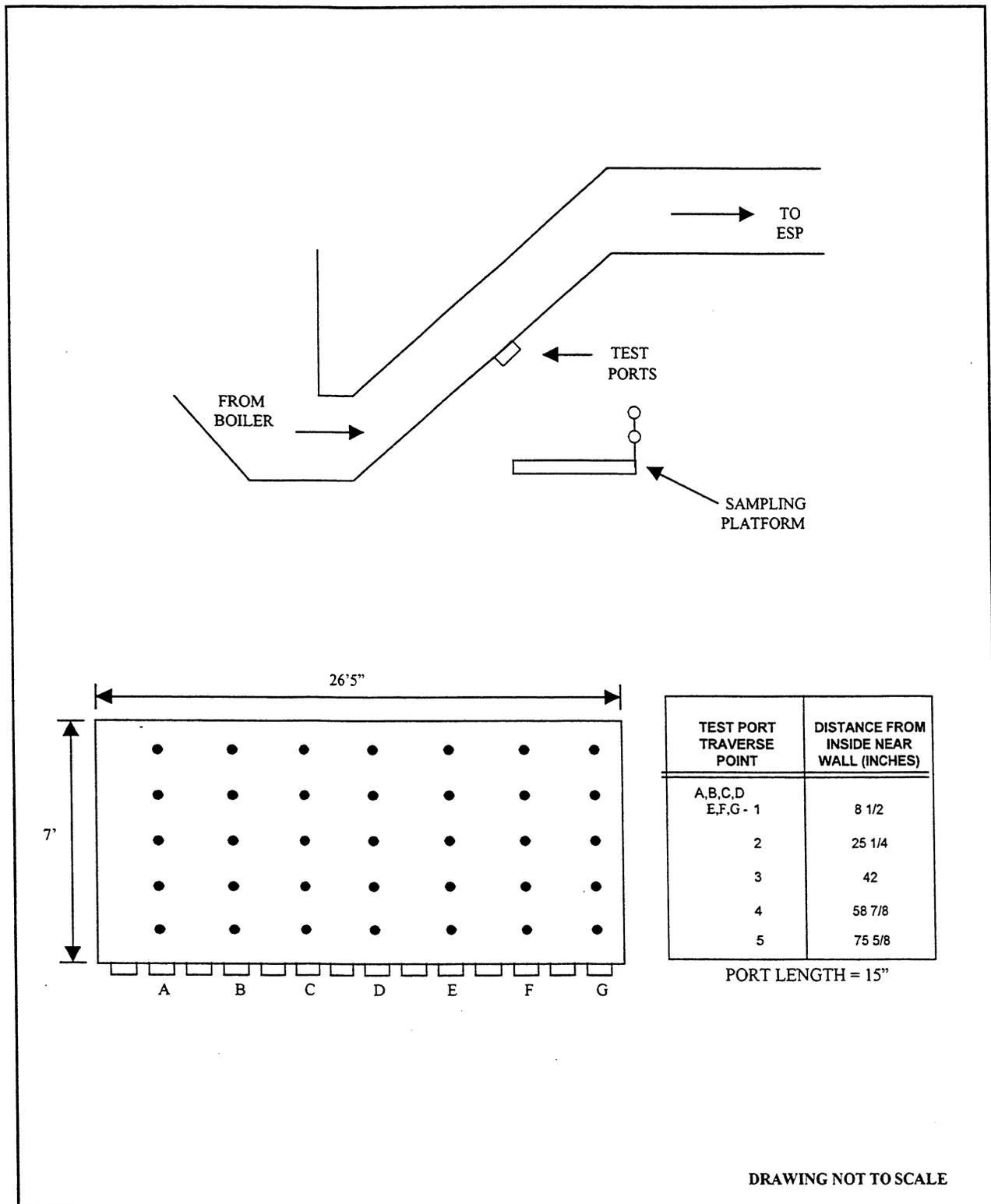


FIGURE 2-3
UNIT 9 ESP INLET DUCT TEST SITE
PORT AND TRAVERSE POINT LOCATIONS

DOE1_04-D20

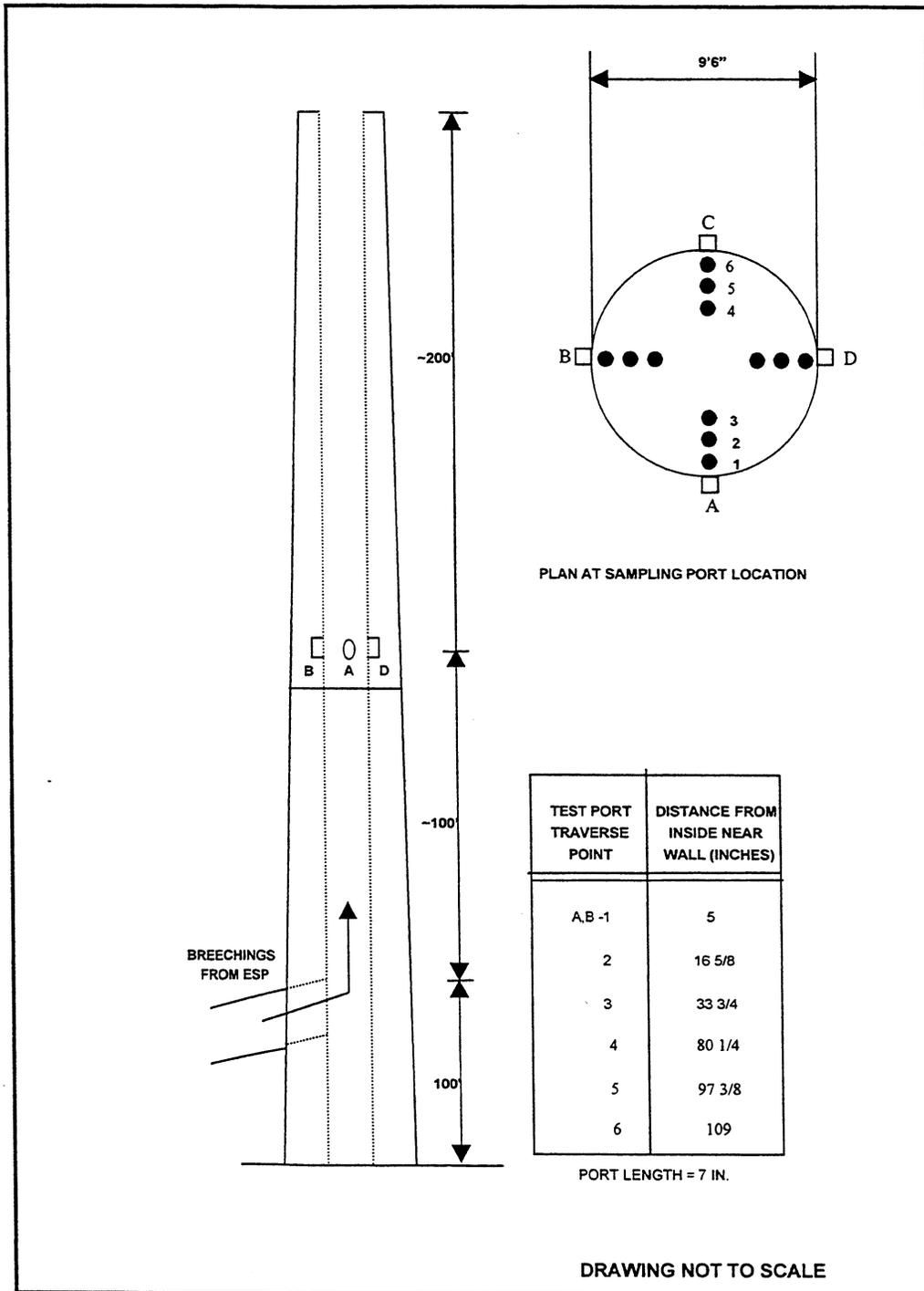


FIGURE 2-4
UNIT 9 STACK TEST SITE
PORT AND TRAVERSE POINT LOCATIONS

DOE1_03-D20

3. SUMMARY AND DISCUSSION OF TEST RESULTS

3.1 TEST PROGRAM OBJECTIVES

The specific objectives of this test program are restated in this section and are as follows:

- Characterize the emissions of particulate-bound, elemental and oxidized mercury from the two coal fired boilers.
- Simultaneously measure concentrations and mass rates of speciated mercury at the inlet and outlet of the ESP on each of the two coal fired boilers.
- Obtain and analyze representative samples of the coal and ESP ash streams for the purpose of determining mercury levels and to establish a material balance for mercury.
- Obtain and analyze representative samples of the coal for the purpose of determining heating value, ash content, sulfur and chlorine levels.
- Determine the carbon content of the ESP ash streams.
- Perform mercury measurements using three mercury CEMS in conjunction with mercury testing at the ESP outlet (stack) on each of the two units.
- Document corresponding boiler, ESP operations and facility CEMS data.

3.2 SAMPLING/TESTING, ANALYTICAL AND QC MATRICES

The detailed sampling/testing, analytical and QC matrices for this survey are presented on Tables 3-1, 3-2 and 3-3 for the coal, ESP ash, and flue gas sampling locations, respectively. Each table specifies the following components:

- Sampling point identification and description.
- Test objective, number and length of test runs performed, and samples/data collected.
- Parameters measured.
- Sampling or monitoring methods employed, including sample preservation technique. Maximum sample holding time.
- Sample preparation/extraction and analysis methods applied.

Table 3-1
Sampling/Testing, Analytical, and QC Plan
Units 5 and 9 – Clean Coal Feed

No. of Test Runs: 3 per unit
 Test Objective: Perform a material characterization/balance for mercury
 Sampling Objective: Collect a representative sample.

Parameters Determined	Mercury	Chlorine	Heating Value	Ultimate/Proximate Analysis	Sulfur	Major Ash Elements	Mass flow Rate
Sampling or Monitoring Method:	Representative sample increments were obtained from the individual boiler coal feed tubes every 20 minutes (alternating tubes) using the ASTM D-197 pulverized coal sampling procedure. Samples stored in air-tight, plastic-lined bucket						
Sample Preparation/Extraction and Analysis Method(s):	Proposed ASTM Method	ASTM E776 and EPA Method 300	ASTM D 1989-91 (calorimeter)	ASTM D-5373 C/H/N; ASTM D 5142-90	ASTM D 4239-85	ASTM D3682-78 ICP-AES	Gravimetric feeder readings recorded in control room
Maximum Holding Time (days):	28	28	28	28	28	28	NA
Sampling or Monitoring Design:							
Total No. of Samples	6	6	6	6	6	6	NA
Site Blanks	0	0	0	0	0	0	NA
Trip Blanks	0	0	0	0	0	0	NA
Lab Blanks	0	0	0	0	0	0	NA
Blank Spikes ²	0	0	0	0	0	0	NA
Replicates ³	All samples	All samples	All samples	All samples	All samples	All samples	NA
QC Spikes ⁴	1/batch ¹	1/batch	1/batch	1/batch	1/batch	1/batch	NA
Approximate No. of Samples Analyzed	13	13	13	13	13	13	NA
Analytical Laboratory:	CONSOL	CONSOL	CONSOL	CONSOL	CONSOL	CONSOL	NA

Notes: ¹A batch consists of a maximum of 20 samples.

²A blank spike (or method spike) is a sample of reagent-grade water spiked with the analyte(s) of interest that is prepared and analyzed with the associated sample batch.

³This indicates that a duplicate analysis is made on one or more samples as a QC mechanism to measure analytical precision.

⁴A sample of similar matrix is spiked with a known amount of the analyte(s) of interest to determine percent recovery.

Table 3-2
Sampling/Testing, Analytical, and QC Plan
Units 5 and 9 — ESP Ash

No. of Test Runs: 3 per unit
 Test Objective: Perform a material characterization/balance for mercury.
 Sampling Objective: Collect a representative sample.

Parameters Determined	Mercury	Carbon	Major Ash Elements	Mass Flow Rates
Sampling or Monitoring Method	Sampling Method S005 – Thief sampling procedure. Collect one sample from representative hoppers following test completion. Store in borosilicate sample bottles with Teflon lids.			Ash rate calculated based on coal feed rate, ash content of coal and gravimetric analysis of thimble samples.
Sample Preparation/Extraction and Analysis Method(s):	Proposed ASTM Method	ASTM 5373	ICP-AES	NA
Maximum Holding Time (days):	28	28	28	NA
Sampling or Monitoring Design:				
Total No. of Samples	6 for Unit 5; 3 for Unit 9	6 for Unit 5; 3 for Unit 9	6 for Unit 5; 3 for Unit 9	NA
Site Blanks	0	0	0	NA
Trip Blanks	0	0	0	NA
Lab Blanks	0	0	0	NA
Blank Spikes ²	0	0	0	NA
Replicates ³	All samples	All samples	All Samples	NA
QC Spikes ⁴	1/batch	1/batch	1/batch	NA
Approximate No. of Samples Analyzed	13 for Unit 5; 7 for Unit 9	13 for Unit 5; 7 for Unit 9	13 for Unit 5; 7 for Unit 9	NA
Analytical Laboratory:	CONSOL	CONSOL	CONSOL	NA

Notes: ¹A batch consists of a maximum of 20 samples.

²A blank spike (or method spike) is a sample of reagent-grade water spiked with the analyte(s) of interest that is prepared and analyzed with the associated sample batch.

³This indicates that a duplicate analysis is made on one or more samples as a QC mechanism to measure analytical precision.

⁴A sample of similar matrix is spiked with a known amount of the analyte(s) of interest to determine percent recovery.

Table 3-3

Sampling/Testing, Analytical, and QC Plan
Units 5 and 9 – ESP Inlet and Outlet

No. of Test Runs: 3 per unit
Test Objective: Perform a material characterization/balance and mercury speciation.
Sampling Objective: Collect a representative sample.

Parameters Determined:	Speciated Mercury	Total Mercury
Sampling or Monitoring and Preservation Method(s)	Ontario Hydro	CEM Analyzers ⁽¹⁾
Sample Preparation/Extraction and Analysis Method(s):	Ontario Hydro	NA
Maximum Holding Time (days):	28	NA
Sampling or Monitoring Design:		
Length of Test:	≥ 120 min	≥ 120 min
Sample Size	≥ 2.0 m ³ (2)	NA
Total No. of Samples	3 at inlet and outlet	3 at outlet of each ESP
Site/Reagent Blanks	Minimum of 1 per sample type	NA
Train Blanks	1 per test location (total of 4)	NA
Lab Blanks	1 per batch ³	NA
Blank Spike Audit ⁴	1 per day	NA
Blank Spikes ⁵	1 per batch	NA
Replicates ⁶	1 per batch	NA
QC spikes ⁷	1 per batch	NA
Approximate No. of Samples Analyzed ⁸	~100	6
Analytical Laboratory:	EERC	NA

- 1 Mercury CEMS at ESP Outlet (stack only)
- 2 Sample volume at ESP inlet was lower due to high particulate loading resulting in smaller nozzle sizes and lower samples rates.
- 3 A batch consists of a maximum of 10 samples.
- 4 A blank spike audit sample is independently prepared and unknown to the analyst.
- 5 A blank spike (or method spike) is a sample of reagent-grade water spiked with the analyte(s) of interest that is prepared and analyzed with the associated
- 6 This indicates that a duplicate or triplicate analysis is made on one or more samples as a QC mechanism to measure analytical precision.
- 7 A sample of similar matrix is spiked with a known amount of the analyte(s) of interest to determine percent recovery.
- 8 Approximate number of total samples and individual fractions, duplicates and other QC samples.

Note: The facility CEMS measured sulfur dioxide (SO₂), oxides of nitrogen (NO_x), carbon monoxide (CO), carbon dioxide (CO₂) and opacity on the ESP outlet (stacks) of Units 5 and 9.

- Sampling and analytical program design (i.e., number of samples collected/analyzed by type and method). This includes the number, or frequency and type, of QC samples analyzed for each parameter.
- Laboratory that analyzed each type of sample.

3.3 PRESENTATION OF RESULTS

3.3.1 Mercury Speciation Test Results

A summary of the Ontario Hydro method mercury speciation test results are presented on Tables 3-4, 3-5, and 3-6 for Units 5 and 9, respectively.

Table 3-4 presents the measured mercury concentrations in micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) for each test run and provides the percent of particulate, oxidized and elemental mercury in comparison to the total mercury.

Tables 3-5 and 3-6 presents the mercury concentrations and mass rate values for particulate, oxidized, elemental and total mercury for each individual test run along with the measured volumetric flow rates. Average values with the standard deviation (SDEV) and percent relative standard deviation (% RSD) have been calculated and are presented.

3.3.1.1 Unit 5

For Unit 5 ESP Inlet an average of 85% of the total mercury measured is particulate bound mercury. On average the oxidized mercury was 10 percent of the total and the elemental mercury was approximately 5 percent of the total mercury collected. At the Unit 5 ESP outlet, elemental mercury comprised the highest of the total at 57 percent. The oxidized mercury was 43 percent of the total and the particulate bound mercury was less than one percent.

Based on the total mercury measurements the average removal efficiency for the ESP was 66 percent with an average mass emission rate of 0.0011 pound per hour.

The elevated mercury levels found in the particulate sample fractions and the associated mercury removal efficiency is believed to be attributed to the high carbon content of the ash (average of 27.6% in the front row of hoppers sample and 46.0% in the back row sample).

TABLE 3-4
COMPARISON OF MERCURY SPECIATION TO TOTAL MERCURY RESULTS
UNITS NO. 5 AND 9

Mercury Species	Unit 5 Inlet						
	Run 1		Run 2		Run 3		Average
	(ug/m3)	% of Total	(ug/m3)	% of Total	(ug/m3)	% of Total	% of Total
Particulate Bound Mercury Emissions	3.93	88.01	3.20	74.54	4.53	91.87	84.80
Oxidized Mercury Emissions	0.41	9.29	0.59	13.67	0.40	8.13	10.36
Elemental Mercury Emissions	0.12	2.70	0.51	11.79	< 0.22	0.00	7.25
Total Mercury Emissions	4.46	100.00	4.29	100.00	4.93	100.00	

Mercury Species	Unit 5 Outlet						
	Run 1		Run 2		Run 3		Average
	(ug/m3)	% of Total	(ug/m3)	% of Total	(ug/m3)	% of Total	% of Total
Particulate Bound Mercury Emissions	0.01	0.63	0.004	0.26	0.01	1.02	0.63
Oxidized Mercury Emissions	0.62	39.91	0.73	44.63	0.62	43.32	42.62
Elemental Mercury Emissions	0.93	59.47	0.90	55.11	0.80	55.66	56.75
Total Mercury Emissions	1.56	100.00	1.63	100.00	1.43	100.00	

Mercury Species	Unit 9 Inlet						
	Run 1		Run 2		Run 3		Average
	(ug/m3)	% of Total	(ug/m3)	% of Total	(ug/m3)	% of Total	% of Total
Particulate Bound Mercury Emissions	0.04	0.61	< 0.02	0.00	< 0.03	0.00	0.61
Oxidized Mercury Emissions	0.15	2.04	0.15	1.97	0.11	1.53	1.85
Elemental Mercury Emissions	7.04	97.35	7.28	98.03	6.76	98.47	97.95
Total Mercury Emissions	7.23	100.00	7.43	100.00	6.86	100.00	

Mercury Species	Unit 9 Outlet						
	Run 1		Run 2		Run 3		Average
	(ug/m3)	% of Total	(ug/m3)	% of Total	(ug/m3)	% of Total	% of Total
Particulate Bound Mercury Emissions	0.003	0.05	< 0.005	0.00	< 0.004	0.00	0.05
Oxidized Mercury Emissions	0.54	8.34	0.65	9.11	0.52	7.76	8.41
Elemental Mercury Emissions	5.96	91.61	6.44	90.89	6.17	92.24	91.58
Total Mercury Emissions	6.51	100.00	7.09	100.00	6.69	100.00	

Note: Non-detect values are not included in the Total Mercury Emissions values. Therefore, the "% of Total" values for non-detects are presented as zeros.

TABLE 3-5
SUMMARY OF MERCURY SPECIATION TEST RESULTS
UNIT NO. 5

TEST DATA:	1			2			3			AVERAGE			STDEV			%RSD			
	Test run number	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	7/13/99	
Location	0905-1135	1342-1606	0815-1040	0905-1144	1335-1622	0815-1039	0905-1144	1335-1622	0815-1039	0905-1144	1335-1622	0815-1039	0905-1144	1335-1622	0815-1039	0905-1144	1335-1622	0815-1039	
Test date	7/13/99	7/13/99	7/14/99	7/13/99	7/13/99	7/14/99	7/13/99	7/13/99	7/14/99	7/13/99	7/13/99	7/14/99	7/13/99	7/13/99	7/14/99	7/13/99	7/13/99	7/14/99	
Test time period	0905-1135	1342-1606	0815-1040	0905-1144	1335-1622	0815-1039	0905-1144	1335-1622	0815-1039	0905-1144	1335-1622	0815-1039	0905-1144	1335-1622	0815-1039	0905-1144	1335-1622	0815-1039	
PROCESS DATA:																			
Unit Load, MW	79.0	78.6	80.9	79.5	79.5	80.9	79.0	78.6	80.9	79.0	78.6	80.9	79.0	78.6	80.9	79.5	79.5	80.9	79.5
Coal feed rate, lb/hr.	72,700	71,700	74,400	72,933	72,933	74,400	72,700	71,700	74,400	72,700	71,700	74,400	72,700	71,700	74,400	72,933	72,933	74,400	72,933
Coal Btu content, Btu/lb. (as received)	12,096	12,149	12,076	12,107	12,107	12,076	12,096	12,149	12,076	12,096	12,149	12,076	12,096	12,149	12,076	12,107	12,107	12,076	12,107
Heat input, 10 ⁶ Btu/hr (F-Factor)	811.0	886.0	911.8	869.6	869.6	911.8	811	886	912	811	886	912	811	886	912	870	870	912	870
GAS STREAM VELOCITY AND VOLUMETRIC FLOW DATA:																			
Avg. temperature, deg. F	338	342	346	342	342	346	340	342	346	340	342	341	340	342	346	341	341	341	341
Avg. gas stream velocity, ft./sec.	46.1	47.9	47.7	47.2	47.2	47.7	46.1	47.2	47.7	46.1	47.2	46.1	46.1	47.2	47.7	46.4	46.4	47.7	46.4
Avg. gas stream volumetric flow, wscf/min (as measured)	413,800	429,300	427,900	423,700	423,700	427,900	413,800	423,700	427,900	413,800	423,700	427,900	413,800	423,700	427,900	423,700	423,700	427,900	423,700
Avg. gas stream volumetric flow, dscf/min. (as measured) ⁽¹⁾	239,800	247,800	245,200	244,300	244,300	245,200	239,800	244,300	245,200	239,800	244,300	245,200	239,800	244,300	245,200	244,300	244,300	245,200	244,300
Avg. gas stream outlet volumetric flow, dscf/min. (adjusted) ^{(1),(2)}	182,700	189,300	194,900	189,000	189,000	194,900	182,700	189,000	194,900	182,700	189,000	194,900	182,700	189,000	194,900	189,000	189,000	194,900	189,000
PARTICULATE BOUND MERCURY EMISSIONS:																			
Conc., ug/m ³	3.93	3.20	4.53	3.88	3.88	4.53	3.93	3.20	4.53	3.93	3.20	4.53	3.93	3.20	4.53	3.88	3.88	4.53	3.88
Conc., ug/Nm ³ ⁽²⁾	4.21	3.43	4.86	4.17	4.17	4.86	4.21	3.43	4.86	4.21	3.43	4.86	4.21	3.43	4.86	4.17	4.17	4.86	4.17
Emission rate, lbs/10 ¹² Btu. ⁽³⁾	3.31	2.56	3.63	3.17	3.17	3.63	3.31	2.56	3.63	3.31	2.56	3.63	3.31	2.56	3.63	3.17	3.17	3.63	3.17
Emission rate, lbs/hr. ⁽³⁾	2.69E-03	2.27E-03	3.31E-03	2.75E-03	2.75E-03	3.31E-03	2.69E-03	2.27E-03	3.31E-03	2.69E-03	2.27E-03	3.31E-03	2.69E-03	2.27E-03	3.31E-03	2.75E-03	2.75E-03	3.31E-03	2.75E-03
OXIDIZED MERCURY EMISSIONS:																			
Conc., ug/m ³	0.41	0.59	0.40	0.47	0.47	0.40	0.41	0.59	0.40	0.41	0.59	0.40	0.41	0.59	0.40	0.47	0.47	0.40	0.47
Conc., ug/Nm ³ ⁽²⁾	0.44	0.63	0.43	0.50	0.50	0.43	0.44	0.63	0.43	0.44	0.63	0.43	0.44	0.63	0.43	0.50	0.50	0.43	0.50
Emission rate, lbs/10 ¹² Btu. ⁽³⁾	0.35	0.47	0.32	0.38	0.38	0.32	0.35	0.47	0.32	0.35	0.47	0.32	0.35	0.47	0.32	0.38	0.38	0.32	0.38
Emission rate, lbs/hr. ⁽³⁾	2.84E-04	4.16E-04	2.93E-04	3.31E-04	3.31E-04	2.93E-04	2.84E-04	4.16E-04	2.93E-04	2.84E-04	4.16E-04	2.93E-04	2.84E-04	4.16E-04	2.93E-04	3.31E-04	3.31E-04	2.93E-04	3.31E-04
ELEMENTAL MERCURY EMISSIONS:																			
Conc., ug/m ³	0.12	0.51	<	0.31	0.31	<	0.12	0.51	<	0.12	0.51	<	0.12	0.51	<	0.31	0.31	<	0.31
Conc., ug/Nm ³ ⁽²⁾	0.13	0.54	<	0.34	0.34	<	0.13	0.54	<	0.13	0.54	<	0.13	0.54	<	0.34	0.34	<	0.34
Emission rate, lbs/10 ¹² Btu. ⁽³⁾	0.10	0.40	<	0.25	0.25	<	0.10	0.40	<	0.10	0.40	<	0.10	0.40	<	0.25	0.25	<	0.25
Emission rate, lbs/hr. ⁽³⁾	8.25E-05	3.59E-04	<	2.21E-04	2.21E-04	<	8.25E-05	3.59E-04	<	8.25E-05	3.59E-04	<	8.25E-05	3.59E-04	<	2.21E-04	2.21E-04	<	2.21E-04
TOTAL MERCURY EMISSIONS:																			
Conc., ug/m ³	4.46	4.29	4.93	4.56	4.56	4.93	4.46	4.29	4.93	4.46	4.29	4.93	4.46	4.29	4.93	4.56	4.56	4.93	4.56
Conc., ug/Nm ³ ⁽²⁾	4.79	4.60	5.29	4.89	4.89	5.29	4.79	4.60	5.29	4.79	4.60	5.29	4.79	4.60	5.29	4.89	4.89	5.29	4.89
Emission rate, lbs/10 ¹² Btu. ⁽³⁾	3.76	3.43	3.95	3.71	3.71	3.95	3.76	3.43	3.95	3.76	3.43	3.95	3.76	3.43	3.95	3.71	3.71	3.95	3.71
Emission rate, lbs/hr. ⁽³⁾	3.05E-03	3.04E-03	3.60E-03	3.23E-03	3.23E-03	3.60E-03	3.05E-03	3.04E-03	3.60E-03	3.05E-03	3.04E-03	3.60E-03	3.05E-03	3.04E-03	3.60E-03	3.23E-03	3.23E-03	3.60E-03	3.23E-03
TOTAL MERCURY REMOVAL EFFICIENCY:																			
				65.08%	65.08%		65.08%			65.08%			65.08%			65.08%	65.08%		65.08%
			61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%	61.87%
			70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%	70.97%

(1) Standard conditions = 68 deg. F. (20 deg. C.) and 29.92 inches Hg (760mm Hg).
 (2) Nm3 = Normal cubic meter (32 deg. F. (0 deg. C.) and 29.92 inches Hg (760mm Hg)).
 (3) Measured volumetric flow from the corresponding test run on the Unit 5 outlet corrected for O₂ measured at the inlet location.
 (4) Emission rates at the ESP inlet are based on the adjusted volumetric flow.

The average total mercury emission rates for Unit 5 are 1.5 ug/m³, 1.3 lbs/10¹² Btu and 0.0011 lb/hr.

Detailed test data and test results for the Unit 5 ESP inlet and outlet are provided on Tables A-1 and A-2 in Appendix A.

3.3.1.2 Unit 9

The Unit 9 mercury test results are in sharp contrast to the Unit No. 5 results. For both the Unit No. 9 inlet and outlet most all mercury present is in the form of elemental mercury. On average the level of elemental mercury at the inlet location was 98 percent with 92 percent of the total mercury being in the elemental form at the ESP outlet. The balance of the mercury at the inlet and outlet is oxidized with less than one-half of a percent as particulate bound mercury.

Since very little mercury was particulate bound, the removal efficiency across the ESP was very low (<2.0 percent on average). Considering the elevated temperature of the hot-side ESP and the low ash content of the coal it is not surprising that most of the mercury is in the elemental form and very little is removed by the ESP.

The average total mercury emissions for Unit No. 9 are 6.8 ug/m³, 4.9 lbs/10¹² Btu and 0.0049 lb/hr.

Detailed test data and test results for the Unit 9 ESP inlet and outlet are provided on Tables A-3 and A-4 in Appendix A.

3.3.2 Mercury CEMS Comparison Results

A comparison of the gas phase mercury concentrations measured in ug/m³ by the mercury CEMS and gas phase mercury concentrations determined using the Ontario Hydro method sampling data are provided in Figures 3-1 and 3-2 for Units 5 and 9. The instrument data are compared to the three Ontario Hydro mercury speciation samples.

It became clear that these instruments operate better when the ambient temperature is relatively constant. For the Presque Isle tests, the instruments were located in the stacks. Often the

temperature was over 100°F. When the operator opened an outside access door, the temperature quickly decreased which often resulted in the instrument shutting down. To prevent this in the future, only the conversion pretreatment system should be located in the stack. The instruments would be located inside a temperature-controlled instrument trailer with the mercury transferred to the CEMS via Teflon heat-traced line. Because the flue gas pretreatment system converts any oxidized (Hg^{2+}) to elemental (Hg^0), there should be no problem getting the mercury to the instruments.

Although there were times when the mercury CEMS were not operating (either intentionally or because of operating problems), the CEMS compared quite favorably with the Ontario Hydro results.

At the Units 5 stack the results showed that the two CEMs gave excellent results for both total mercury and Hg^0 . The PS Analytical CEM gave more consistent results than the Semtech. The results were not quite as good at the Unit 9 stack. At this location the total mercury results for the Semtech were significantly lower than the Ontario Hydro method, and the PS Analytical CEM was somewhat higher. Although the PS Analytical CEM total mercury results were higher than those provided by the Ontario Hydro method, the Hg^0 results were within statistical variation for the two methods. The Semtech results were very consistent, and it is possible that there was a problem with the calibration. The statistical results for the CEMS compared to the Ontario Hydro method for all tests are shown in Table 3-7.

3.3.3 Mercury Material Balance

Mercury material balance closures were calculated for each of the individual measurements (three for each unit). A description of the calculation technique and assumptions and a discussion of the results is contained in this section.

3.3.3.1 Material Balance Procedure

Gas phase mercury measurements are difficult to obtain because typical flue gas mercury concentrations are in the 1 to 10 $\mu\text{g}/\text{m}^3$ range. Measurements made with the Ontario Hydro

Figure 3-1
 Comparison of Mercury CEMS
 to Ontario Hydro Results - Unit 5 Stack

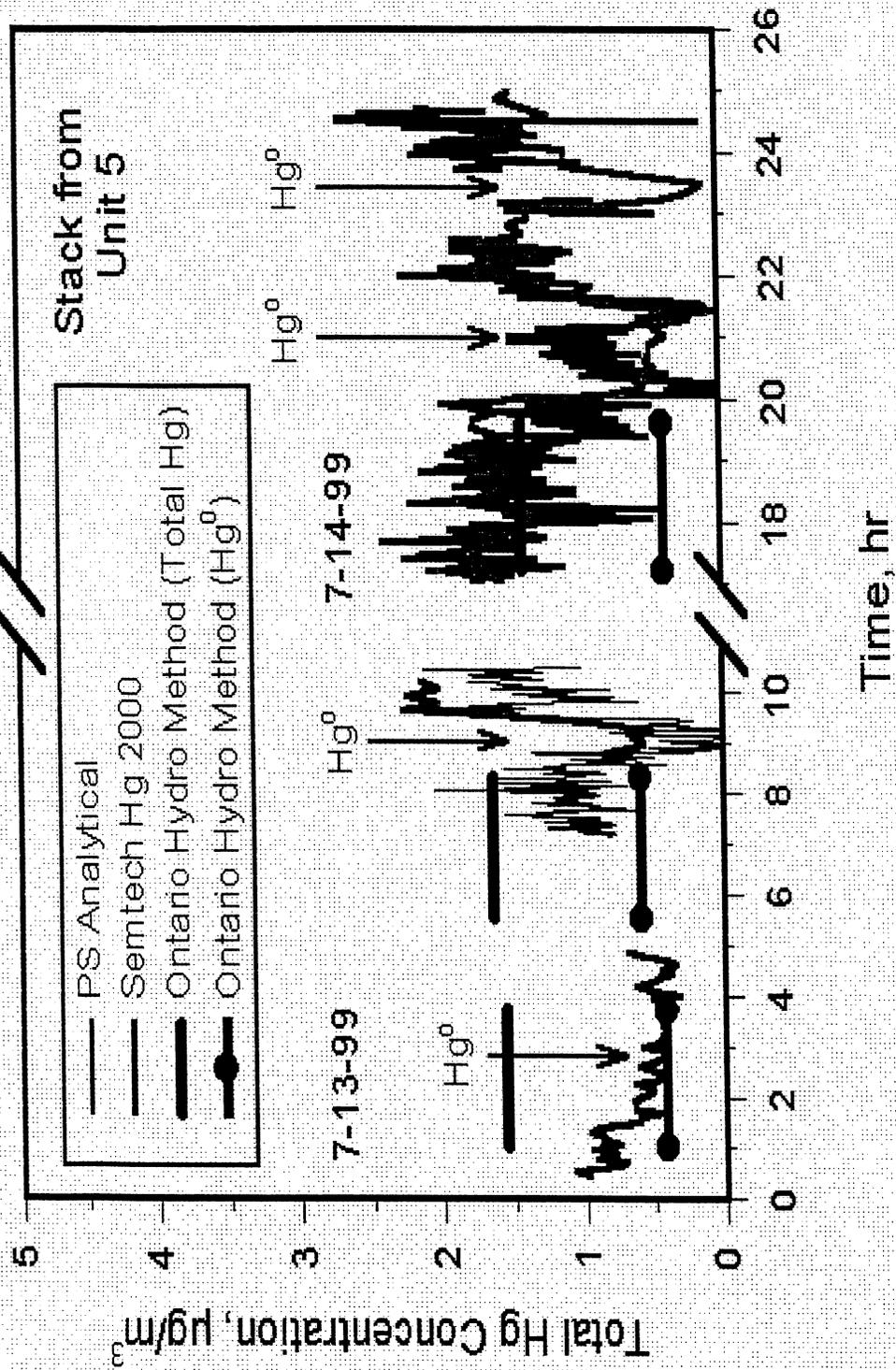


Figure 3-2
 Comparison of Mercury CEMS
 to Ontario Hydro Results - Unit 9 Stack

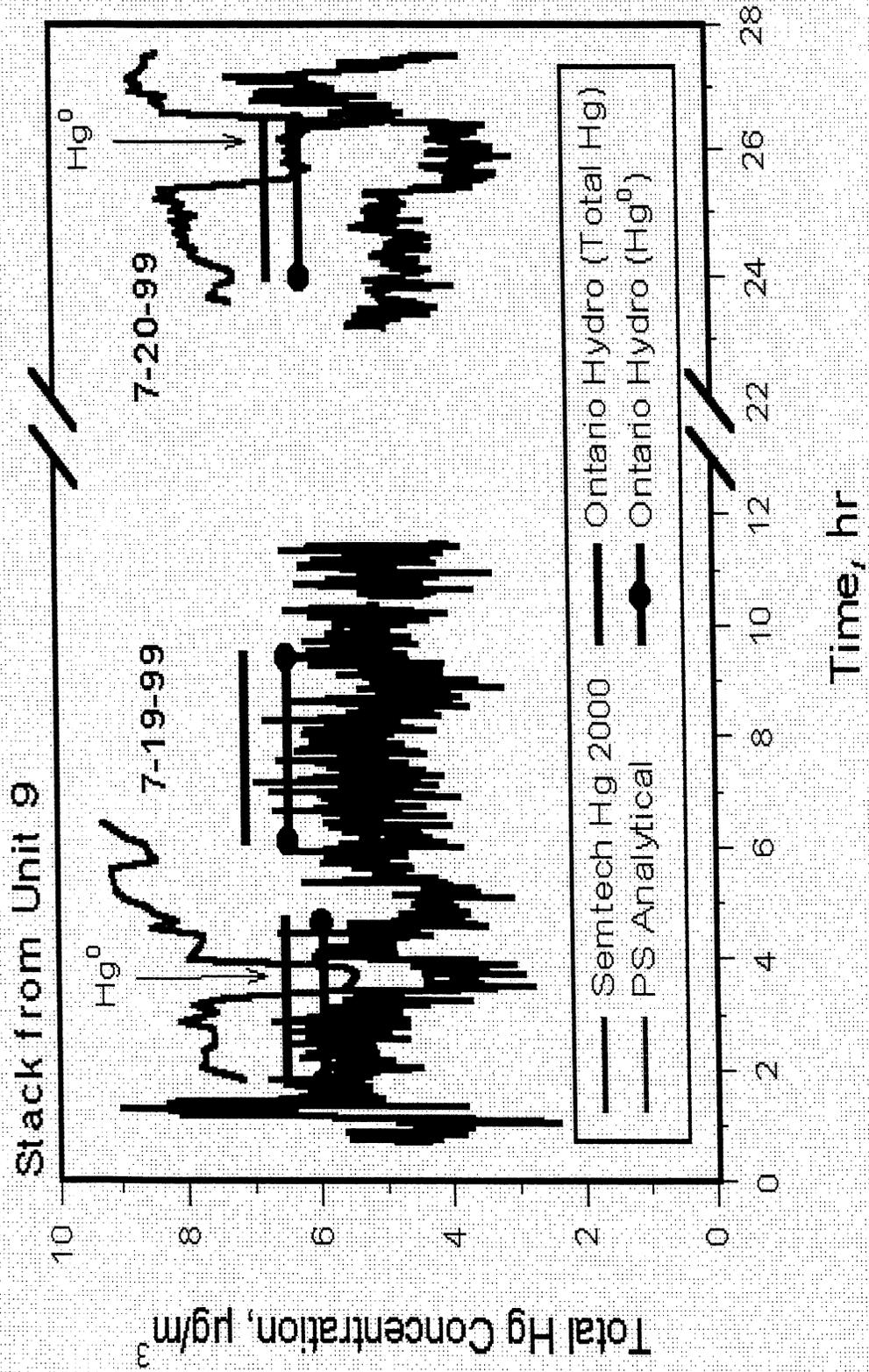


TABLE 3-7

SUMMARY OF MERCURY CEM RESULTS FOR UNITS 5 AND 9

Test Location	Average Measured Values			
Unit 5 Stack	Hg ⁰ μg/m ³	SDEV μg/m ³	Total Hg μg/m ³	SDEV μg/m ³
PS Analytical	0.60	0.19	1.64	0.31
Semtech Hg 2000	0.58	0.43	1.40	0.41
Ontario Hydro Method	0.87	0.07	1.54	0.11
Unit 9 Stack				
PS Analytical	6.02	0.46	8.10	0.57
Semtech Hg 2000	3.52	0.46	4.73	1.14
Ontario Hydro Method	6.19	0.25	6.76	0.31

speciation train typically result in Hg-in-solution concentrations of 1 to 10 µg/L. Because of the difficulty of these low level measurements, material balance calculations provide a useful tool for assessing the accuracy of the flue gas measurements. Mercury balance closures were calculated for each of the individual flue gas measurements for each of the two units sampled. Because flue gas mercury measurements were completed at both the ESP inlet and stack locations, material balance closures were calculated at both locations.

The material balance closure is defined as the mass of mercury measured in the output streams divided by the mass of mercury in the input streams. The only mercury input stream is the coal. The mercury outlet streams are the flue gas exiting the stack and the fly ash.

The mercury input is calculated from the mercury concentration of the coal based on a three-test average and the coal firing rate determined from the coal's F-factor. The average mercury concentration is used to reduce the uncertainty associated with the variability of the mercury analysis of coal. The mercury analysis technique used by the CONSOL lab has a variability of ~10-15%. Because of this, it is believed that the use of the average mercury concentration, in place of the individual coal mercury measurements, provides a more useful and accurate value for material balance calculations. The F-factor calculation of the coal firing rate uses the measured volumetric flow rate, the measured gas composition, and the measured coal quality data. A fossil fuel's F-factor is the amount of flue gas that is produced (DSCF) from the combustion of 1 MM Btu of fuel. This is referred to as the fuel's F-factor and is calculated from the elemental composition. By knowing the F-factor and the volumetric flue gas flow rate corrected to a 0% oxygen basis, the coal firing rate can be accurately determined. This value was further re-fined by accounting for the un-burned carbon in the fly ash. In most cases, the coal firing rate determined from this procedure is more accurate than the firing rate determined from the plant coal feeders.

The material balance closure for the ESP inlet location is calculated from the mass rate of mercury obtained from the Ontario Hydro measurement at this location compared to the mercury input from the coal. Since bottom ash and economizer ash samples were not collected it is assumed for this program that no mercury was present in either of those ash streams. The Ontario Hydro method yields a flue gas mercury concentration value. This concentration value

is converted into a mass flow value using the volumetric gas flow rate at this location. The gas flow rate is obtained from the measured gas flow at the stack, and correcting for oxygen difference between the stack and ESP inlet sampling locations. The outlet stack gas flow measurement was used rather than the inlet flow rate because it is more accurate. Accurate ESP inlet flow measurements were difficult to obtain due to poor duct configuration and close proximity to both upstream and downstream flow disturbances. The material balance closure for the ESP outlet location is calculated from the mass rate of mercury obtained from the Ontario Hydro measurement at this location combined with the mass flow rate of mercury in the ESP ash compared to the mercury input value. The ESP ash flow rate was determined from the particulate measurements at the ESP inlet obtained as part of the Ontario Hydro sampling method. Representative ESP ash samples were taken from the hoppers located below the ESP. Unit 5 has a two-field, cold-side precipitator. Ash was obtained from both fields and individually analyzed for mercury. The composite mercury concentration of the ESP ash was mathematically calculated by assuming a 70:30 ratio between the two ESP fields. Unit 9 has a three-field, hot-side precipitator. It was theorized that no mercury was absorbed on the ESP ash at the flue gas temperature. Because of this, a single grab sample was obtained from the first field hoppers only.

3.3.3.2 Discussion of Mercury Material Balance Results

Unit 5 - The average coal firing rate for Unit 5 using the F-factor calculation was 73,209 lb/hr. This compares well with the coal feeder value of 72,933 lb/hr. The average mercury concentration of the coal was 0.043 $\mu\text{g/g}$ (ppm). This results in an average mercury mass flow rate of 23.79 milligram per minute (mg/min) into the boiler. The average mercury mass flow rate at the ESP inlet as determined from the Ontario Hydro measurements was 24.42 mg/min. The average material balance closure at the ESP inlet for Unit 5 was 102.6% with the individual closures being 103.9%, 94.9%, and 108.9%. The mass flow rate of mercury in the flue gas at the ESP outlet was 8.26 mg/min. The mercury mass flow rate in the ESP ash was 13.05 mg/min. The average material balance closure at the ESP outlet for Unit 5 was 90.1% with the individual closures being 104.1%, 85.5%, and 80.6%. These closures are excellent when considering the low value of mercury in the ESP outlet gas ($\sim 1.5 \mu\text{g/m}^3$) and the uncertainty in the calculation of

the ESP mass flow rate. The ESP ash hopper samples show that 55% of the mercury released during combustion is captured with the ash.

Unit 9 - The average coal firing rate for Unit 9 using the F-factor calculation was 82,775 lb/hr. This compares well with the coal feeder value of 80,116 lb/hr. The average mercury concentration of the coal was 0.068 µg/g (ppm). This results in an average mercury mass flow rate of 42.54 mg/min into the boiler. The average mercury mass flow rate at the ESP inlet as determined from the Ontario Hydro measurements was 35.77 mg/min. The average material balance closure at the ESP inlet for Unit 9 was 84.1% with the individual closures being 85.2%, 86.3%, and 80.8%. The mass flow rate of mercury in the flue gas at the ESP outlet was 37.16 mg/min. No detectable mercury was measured in the ESP ash. The average material balance closure at the ESP outlet for Unit 9 was 87.3% with the individual closures being 85.3%, 91.0%, and 85.6%. These closures are excellent when considering the low levels of mercury encountered.

3.3.3.3 Overall Summary of Material Balance Data

The material balance closures calculated are reasonable for the emission levels measured. These balances are within the reproducibility of the measurement methods and help validate the gas phase measurements. The material balance closures at the ESP inlet and outlet also show excellent agreement, again validating the reported flue gas measurements at both locations.

Detailed summaries of the mercury material balance data are provided on Tables A-5 and A-6 in Appendix A.

3.3.4 Process Solid Sample Stream Results

Tables 3-8 through 3-12 provide a summary of the analytical results obtained on the coal feed and ESP ash samples collected on Units No. 5 and 9.

For each parameter measured on the coal and ash streams, the concentration or percent value is presented for each individual test run along with the average standard deviation and percent relative standard deviation.

A review of the process solid sample stream results indicate a consistent coal quality was fired to both Units 5 and 9 during all test periods.

The results obtained on the Units 5 and 9 ESP ash samples are comparable from run to run and indicate consistent boiler firing and ESP operations.

Detailed analytical summaries are provided in Appendix D of this report.

3.3.5 Unit Operation and Key Operational Parameters

This section describes the Units 5 and 9 operations during the test program and provides the key operating parameters that were monitored and documented during testing.

3.3.5.1 Unit Operation During Testing

Operation of Units 5 and 9 during testing was representative of normal daily operation at or near full load. Steady-state testing conditions were maintained during all test periods. The normal sootblowing activities were maintained on each boiler during testing.

3.3.5.2 Process Control Data

All key power generation process operating parameters and control data were manually recorded by the operators during each test at a frequency of once per hour during each test period. ESP operational indicators data were recorded by a data acquisition system at 6-minute intervals. The facilities CEMS data acquisition system provided concentration values at 1-minute intervals.

A summary of the key operating data is provided in Tables 3-13 and 3-14 for Units 5 and 9. All additional process, ESP operations data and CEM data are provided in Appendix B.

Table 3-8

Summary of Process Solid Sample Stream Results
Unit No. 5 Coal Feed Samples

As Determined Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
Date	7/13/99	7/13/99	7/14/99			
Moisture	5.29	5.07	5.41	5.26	0.17	3.3
Volatile Matter	36.37	36.26	33.13	35.25	1.83	5.2
Ash	9.28	9.44	9.82	9.51	0.28	2.9
Carbon	70.05	69.15	69.09	69.43	0.54	0.8
Hydrogen	5.10	5.04	5.04	5.06	0.03	0.7
Nitrogen	1.53	1.49	1.46	1.49	0.03	2.3
Sulfur	0.96	0.97	0.98	0.97	0.01	1.0
Oxygen	7.79	8.84	8.20	8.28	0.53	6.4
Chlorine, ppm	171	209	161	180	25	14.1
Mercury, ppb	43	38	41	41	3	6.2
Btu/lb	12096	12150	12075	12107	39	0.3
Total Moisture	5.29	5.07	5.41	5.26	0.17	3.3

Dry Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
Volatile Matter	38.40	38.20	35.02	37.21	1.89	5.1
Ash	9.80	9.94	10.38	10.04	0.30	3.0
Carbon	73.96	72.84	73.04	73.28	0.60	0.8
Hydrogen	4.76	4.71	4.69	4.72	0.04	0.8
Nitrogen	1.62	1.57	1.54	1.58	0.04	2.3
Sulfur	1.01	1.02	1.04	1.02	0.01	1.1
Oxygen	8.83	9.94	9.29	9.35	0.56	5.9
Chlorine, ppm	180	220	170	190	26	13.9
Mercury, ppb	45	40	43	43	3	6.3
Btu/lb	12772	12799	12766	12779	18	0.1
F Factor, O ₂	9962	9753	9820	9845	106	1.1
F Factor, CO ₂	1859	1827	1837	1841	16	0.9

Major Ash Elements: (% of Coal Ash)

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
SiO ₂	57.31	56.49	56.09	56.63	0.62	1.1
Al ₂ O ₃	25.07	25.47	24.99	25.18	0.26	1.0
TiO ₂	0.84	0.83	0.83	0.83	0.01	0.7
Fe ₂ O ₃	4.94	4.81	4.89	4.88	0.07	1.3
CaO	3.51	3.48	3.24	3.41	0.15	4.3
MgO	1.78	1.77	1.76	1.77	0.01	0.6
Na ₂ O	2.23	2.22	2.19	2.21	0.02	0.9
K ₂ O	1.03	1.02	1.07	1.04	0.03	2.5
P ₂ O ₅	0.47	0.48	0.47	0.47	0.01	1.2
SO ₃	3.39	3.52	3.18	3.36	0.17	5.1
Undetermined	-0.57	-0.09	1.29	0.21	0.96	—

Table 3-9

Summary of Process Solid Sample Stream Results
Unit No. 5 ESP Ash Samples (Front Row of Hoppers)

As Determined Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Date	7/13/99	7/13/99	7/14/99			
Moisture	0.32	0.51	0.35	0.39	0.10	25.9%
Ash	72.15	68.40	69.71	70.09	1.90	2.7%
Carbon	25.66	29.31	27.89	27.62	1.83	6.6%
Sulfur	0.83	0.90	0.84	0.86	0.04	4.4%
Mercury, ppb	159	114	93	122	34	27.5%

Dry Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Ash	72.38	68.75	69.95	70.36	1.84	2.6%
Carbon	25.74	29.46	27.99	27.73	1.87	6.7%
Sulfur	0.83	0.90	0.84	0.86	0.04	4.5%
Mercury, ppb	160	115	93	122	34	27.5%

Major Ash Elements:

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
SiO ₂	43.51	41.24	42.08	42.28	1.14	2.7%
Al ₂ O ₃	17.25	17.01	17.25	17.17	0.14	0.8%
TiO ₂	0.55	0.51	0.56	0.54	0.03	4.9%
Fe ₂ O ₃	4.30	3.79	3.97	4.02	0.26	6.4%
CaO	2.63	2.43	2.53	2.53	0.10	3.9%
MgO	1.34	1.22	1.25	1.27	0.06	4.9%
Na ₂ O	1.58	1.44	1.53	1.52	0.07	4.7%
K ₂ O	0.74	0.64	0.71	0.70	0.05	7.3%
P ₂ O ₅	0.32	0.28	0.33	0.31	0.03	8.5%
SO ₃	0.51	0.43	0.43	0.46	0.05	10.1%
Undetermined	27.27	31.01	29.36	29.21	1.87	6.4%

Table 3-10

Summary of Process Solid Sample Stream Results
Unit No. 5 ESP Ash Samples (Back Row of Hoppers)

As Determined Basis

Test ID	T-1	T-2	T-3			
Date	7/13/99	7/13/99	7/14/99	AVG	SDEV	PRSD
Moisture	0.56	0.49	0.56	0.54	0.04	7.5%
Ash	48.86	51.55	51.57	50.66	1.55	3.1%
Carbon	47.82	45.84	44.40	46.02	1.71	3.7%
Sulfur	1.51	1.38	1.21	1.37	0.15	11.0%
Mercury, ppb	234	214	228	225	10	4.5%

Dry Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Ash	49.14	51.80	51.86	50.93	1.55	3.0%
Carbon	48.09	46.07	44.65	46.27	1.72	3.7%
Sulfur	1.52	1.39	1.22	1.37	0.15	11.0%
Mercury, ppb	235	215	229	227	10	4.6%

Note: Major ash analysis on back hopper rows was not performed.

Table 3-11

**Summary of Process Solid Sample Stream Results
Unit No. 9 Coal Feed Samples**

As Determined Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
Date	7/19/99	7/20/99	7/20/99			
Moisture	21.39	20.55	20.51	20.82	0.49	2.4
Volatile Matter	34.56	35.22	34.62	34.80	0.36	1.0
Ash	5.52	5.55	5.63	5.57	0.06	1.0
Carbon	54.68	55.00	55.33	55.00	0.32	0.6
Hydrogen	6.02	5.90	5.93	5.95	0.06	1.0
Nitrogen	0.76	0.76	0.77	0.76	0.01	1.0
Sulfur	0.32	0.33	0.33	0.33	0.00	1.5
Oxygen	11.31	11.93	11.50	11.58	0.31	2.7
Chlorine, ppm	165	199	167	177.00	19.00	10.7
Mercury, ppb	54	52	56	54	2	3.7
Btu/lb	9454	9587	9580	9540	75	0.8
Total Moisture	21.39	20.55	20.51	20.82	0.49	2.4

Dry Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
Volatile Matter	43.96	44.33	43.55	43.95	0.39	0.9
Ash	7.02	6.99	7.08	7.03	0.05	0.7
Carbon	69.56	69.22	69.61	69.46	0.21	0.3
Hydrogen	4.61	4.53	4.57	4.57	0.04	0.9
Nitrogen	0.97	0.95	0.97	0.96	0.01	1.0
Sulfur	0.41	0.41	0.42	0.41	0.00	1.0
Oxygen	17.41	17.88	17.33	17.54	0.30	1.7
Chlorine, ppm	210	250	210	223	23	10.4
Mercury, ppb	69	65	70	68	3	3.7
Btu/lb	12026	12067	12052	12048	20	0.2
F Factor, O ₂	9610	9492	9587	9563	62	0.6
F Factor, CO ₂	1857	1841	1854	1851	8	0.4

Major Ash Elements: (% of Coal Ash)

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
SiO ₂	35.87	35.91	36.87	36.22	0.56	1.6
Al ₂ O ₃	17.32	17.05	17.05	17.14	0.16	0.9
TiO ₂	1.14	1.13	1.11	1.13	0.02	1.4
Fe ₂ O ₃	4.97	4.83	4.97	4.92	0.08	1.6
CaO	16.11	16.13	15.93	16.06	0.11	0.7
MgO	4.18	4.13	4.07	4.13	0.05	1.3
Na ₂ O	3.63	3.70	3.49	3.61	0.11	3.0
K ₂ O	0.78	0.78	0.81	0.79	0.02	2.2
P ₂ O ₅	1.13	1.13	1.07	1.11	0.03	3.1
SO ₃	13.92	13.11	13.60	13.54	0.41	3.0
Undetermined	0.95	2.10	1.03	1.36	0.64	47.0

Table 3-12

Summary of Process Solid Sample Stream Results
Unit No. 9 ESP Ash Samples

As Determined Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
Date	7/19/99	7/19/99	7/20/99			
Moisture	0.08	0.01	0.02	0.04	0.04	102.9
Ash	98.85	98.80	98.58	98.74	0.14	0.1
Carbon	0.73	0.93	1.21	0.96	0.24	25.1
Sulfur	0.00	0.00	0.00	0.00	0.00	0.0
Mercury, ppb	8.4	5.9	7.9	7	1	17.8

Dry Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
Ash	98.93	98.81	98.60	98.78	0.17	0.2
Carbon	0.73	0.93	1.21	0.96	0.24	25.1
Sulfur	0.00	0.00	0.00	0.00	0.00	0.0
Mercury, ppb	8	6	8	7	1	17.8

Major Ash Elements:

Test ID	T-1	T-2	T-3	AVG	SDEV	%RSD
SiO ₂	39.05	40.13	38.69	39.29	0.75	1.9
Al ₂ O ₃	19.05	18.87	19.11	19.01	0.12	0.7
TiO ₂	1.35	1.34	1.30	1.33	0.03	2.0
Fe ₂ O ₃	5.50	5.47	5.37	5.45	0.07	1.2
CaO	19.63	19.49	19.54	19.55	0.07	0.4
MgO	4.78	4.69	4.75	4.74	0.05	1.0
Na ₂ O	4.50	4.33	4.49	4.44	0.10	2.1
K ₂ O	0.85	0.84	0.89	0.86	0.03	3.1
P ₂ O ₅	1.31	1.22	1.30	1.28	0.05	3.8
SO ₃	1.89	1.54	1.88	1.77	0.20	11.2
Undetermined	2.09	2.08	2.68	2.28	0.34	15.0

Table 3-13
Summary of Key Process Control Data
Unit No. 5

Parameter	Units	Run No.		
		1	2	3
Gross Generation	MWH	79.0	78.6	80.9
Station Service	MWH	4.3	4.0	4.3
Net Generation	MWH	74.9	74.6	76.3
Coal Total	lbs/hr	72,700	71,700	74,400
Lbs. Coal/Net KWH	Lbs	0.98	0.96	0.98
Lbs. Steam/Net KWH	Lbs	7.0	7.2	7.1
Control Valve Position	%	92.0	92.0	93.3
Main Steam Flow	lbs/hr	523,250	518,000	539,000
Feed Water Flow	lbs/hr	509,250	500,500	525,000
Feed Water Pressure	Psig	1606	1578	1590
First Stage Pressure	Psig	1032	1021	1057
Cold Reheat Pressure	Psig	384	384	395
Hot Reheat Pressure	Psig	360	362	372
Feed Water	Loading %/Temp °F	48% / 414	48% / 414	49% / 416
Main Steam Temp.	°F	1000	1000	1000
Cold Reheat Temp.	°F	665	668	670
Hot Reheat Temp.	°F	1000	1000	1000
Superheat Spray Flow	lbs/hr	4420	4533	4357
Reheat Spray Flow	lbs/hr	13,060	15,343	16,983
Air Flow	Acfm	610,000	622,500	633,300
Excess Oxygen	%	3.2	3.4	3.2
Inlet Air Temp.	°F	154	155	150
Gas Outlet Temp.	°F	305	314	306
Stack Opacity	%	7	8	10
Stack CEMs (SO ₂)	Ppm/v	570	553	563
Stack CEMs (NO _x)	Ppm/v	489	494	509
Stack CEM (CO)	Ppm/v	23	27	19
Stack CEM (CO ₂)	%	12.6	12.2	12.6
Induced Draft Fan	(Loading %/Amps)	90% / 162	92% / 167	98% / 168
Forced Draft Fan	(Loading %/rpm/Amps)	78% / 92	80% / 95	80% / 95

Table 3-14
Summary of Key Process Control Data
Unit No. 9

Parameter	Units	Run No.		
		1	2	3
Gross Generation	MWH	84.0	84.3	84.7
Station Service	MWH	6.8	6.8	7.3
Net Generation	MWH	77.5	77.5	77.3
Coal Total	Lbs	101,300	101,000	101,300
Lbs. Coal/Net KWH	Lbs	1.3	1.3	1.3
Lbs. Steam/Net KWH	Lbs	7.1	7.1	7.7
Control Valve Position	%	94.8	94.3	95
Main Steam Flow	lbs/hr	548,000	549,000	590,300
Feed Water Flow	lbs/hr	528,000	527,500	542,300
Feed Water Pressure	Psig	1580	1570	1590
First Stage Pressure	Psig	1171	1159	1177
Cold Reheat Pressure	Psig	404	405	403
Hot Reheat Pressure	Psig	400	400	393
Feed Water	Loading %/Temp °F	70% / 450	69% / 450	70% / 450
Main Steam Temp.	°F	1003	1003	1003
Cold Reheat Temp.	°F	685	685	689
Hot Reheat Temp.	°F	1001	1001	1004
Superheat Spray Flow	Lbs/hr	16,100	17050	10,500
Reheat Spray Flow	Lbs/hr	12,700	16380	8200
Air Flow	Acfm	560,000	557,500	555,000
Excess Oxygen	%	1.8	1.6	1.5
Inlet Air Temp.	°F	107	109	112
Gas Outlet Temp.	°F	350	350	350
Stack Opacity	%	7	7	7
Stack CEMs (SO ₂)	ppm/v	272	275	279
Stack CEMs (NO _x)	ppm/v	287	283	292
Stack CEM (CO)	ppm/v	19	32	20
Stack CEM (CO ₂)	%	13.6	13.7	13.7
Induced Draft Fan	(Loading %/Amps)	89% / 276	87% / 271	90% / 274
Forced Draft Fan	(Loading %/rpm/Amps)	72% / 165	72% / 163	75% / 165

4. SAMPLING AND ANALYTICAL PROCEDURES

4.1 DESCRIPTION OF SAMPLING EQUIPMENT

4.1.1 Ontario Hydro Mercury Speciation Method

The Ontario Hydro sampling train contained the following components:

- A calibrated borosilicate nozzle attached to a borosilicate thimble holder containing a high capacity in-stack quartz fiber thimble.
- The thimble holder was attached to a heated borosilicate probe equipped with a calibrated thermocouple to measure flue gas temperature and a calibrated S-type pitot tube to measure flue gas velocity pressure.
- A rigid borosilicate connector to join the outlet of the probe to the inlet of the impinger train was used at the Unit No. 5 ESP inlet and both outlet test locations. A flexible heated Teflon line was used at the Unit No. 9 ESP inlet test location to transport the sample gas from the probe exit to the first impinger inlet.
- An impinger train consisting of eight impingers. The first, second, and third impingers each contained 100 ml of 1 Normal (N) potassium chloride (KCl). The fourth impinger contained 100 ml of 5% nitric acid (HNO₃) and 10% hydrogen peroxide (H₂O₂). The fifth, sixth and seventh impingers each contained 100 ml of 4% potassium permanganate (KMnO₄) and 10% sulfuric acid (H₂SO₄). The eighth impinger contained 300 grams of dry preweighed silica gel. The third and seventh impingers were a Greenburg-Smith type; all other impingers were of a modified design. All impingers were maintained in a crushed ice bath.
- A vacuum line (umbilical cord) with adapter to connect the outlet of the impinger train to a control module.
- A control module containing a 3-cfm carbon vane vacuum pump (sample gas mover), a calibrated dry gas meter (sample gas volume measurement device), a calibrated orifice (sample gas flow rate monitor) and inclined manometers (orifice and gas stream pressure indicators).
- A switchable calibrated digital pyrometer to monitor flue and sample gas temperatures.

See Figure 4-1 for a schematic of the Ontario Hydro test train.

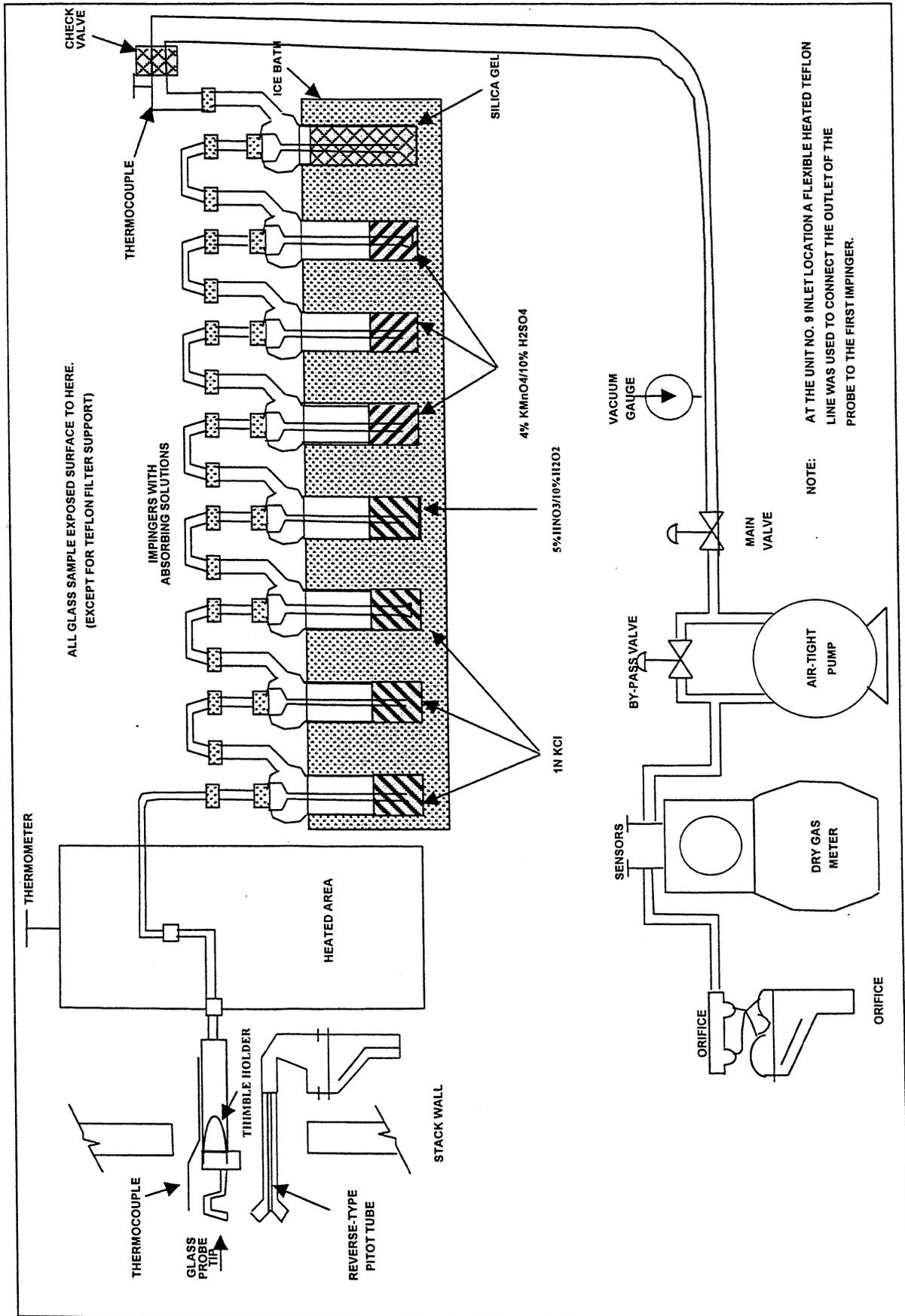


FIGURE 4 - 1
ONTARIO HYDRO SAMPLING TRAIN

4.2 CO₂ AND O₂ SAMPLING EQUIPMENT

The fixed gases sampling train (Figure 4-2) used at the ESP inlet and outlet test sites was assembled in accordance with EPA Method 3 and consisted of the following components:

- A stainless steel or Teflon probe (fastened to the Ontario Hydro sampling probe) with a plug of glass wool to remove particulate.
- An ice-cooled condenser to remove moisture from the sampled gases.
- A diaphragm pump to draw a sample of the gases.
- A valve and rate meter to control and monitor gas stream sampling rates, respectively.
- A Tedlar® bag to contain the sample of flue gases.

The CO₂ and O₂ concentrations of each bag were analyzed using a Servomex 1440B CEM. The analyzers were calibrated before and after each set of analysis using EPA Protocol CO₂ and O₂ gas standards with nitrogen used as the zero gas.

4.3 MERCURY CEMS EQUIPMENT

Automated on-line mercury analyzers are being developed based on well-established techniques, including cold-vapor atomic absorption spectroscopy (CVAAS), cold-vapor atomic fluorescence spectroscopy (CVAFS), and atomic emission spectroscopy (AES), as well as on the emerging technology of chemical microsensors. The analyzers can be used to directly measure Hg⁰ in fossil fuel combustion flue gas on a continuous or semi-continuous basis and can be equipped with converters for reducing Hg²⁺ forms to Hg⁰ to determine total mercury; the Hg²⁺ concentration can be determined by difference. Although costly to purchase, install, and maintain, on-line continuous emission analyzers offer several advantages:

- An analyzer can be used for feedback on process control of mercury control systems, thus maximizing removal efficiency.
- A properly designed analyzer requires minimal operator input.
- An analyzer can provide information on the temporal variations of mercury emissions for a process that may be variable in its emission characteristics.

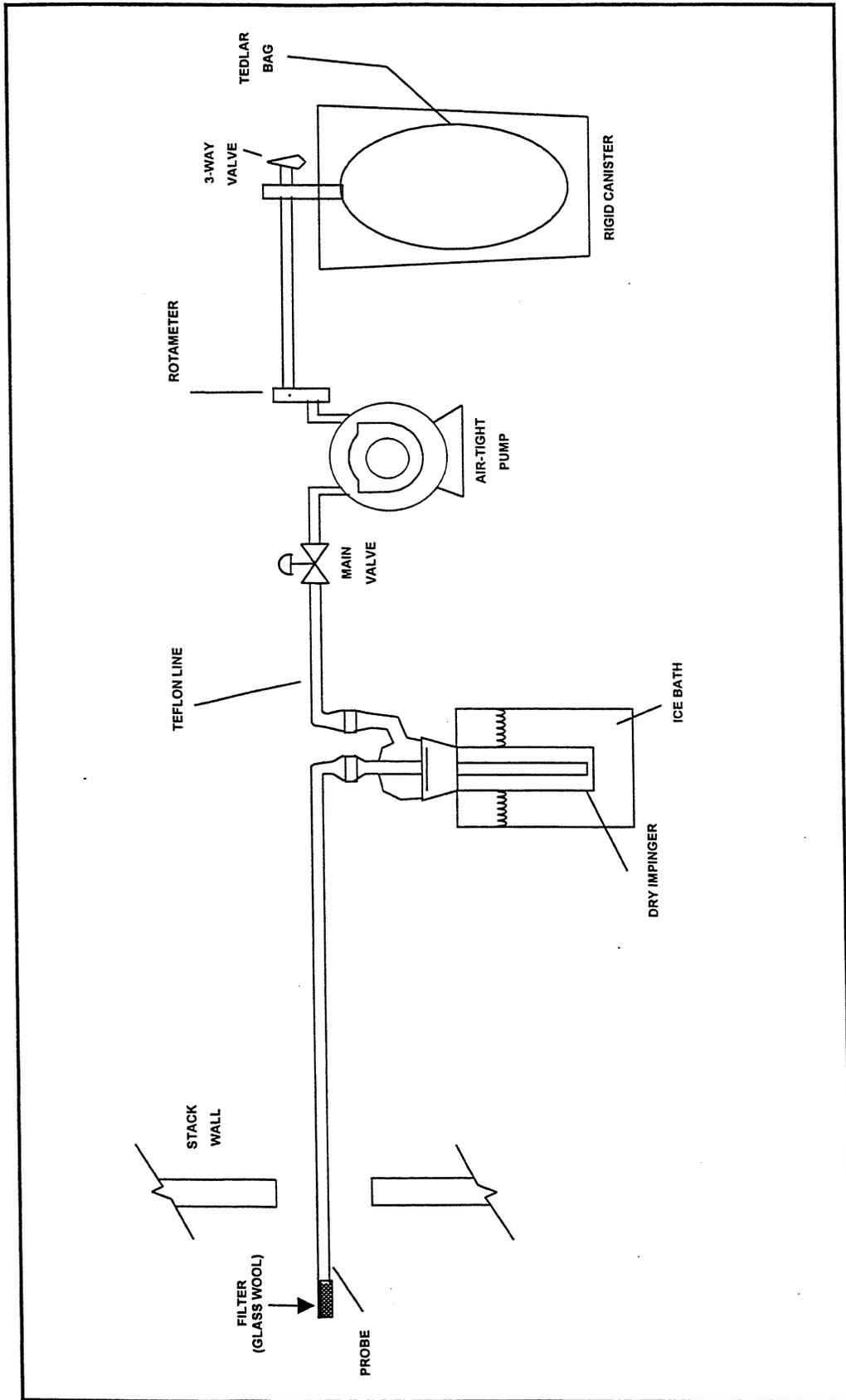


FIGURE 4-2
EPA METHOD 3 - DRY GAS STREAM COMPOSITION SAMPLING TRAIN

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On-line mercury emission analyzers can be categorized as either extractive or in situ. Extractive analyzers are usually located remote to the sample extraction point; therefore, a flue gas sample is removed, transported, and conditioned before it actually enters the mercury analyzer. In situ mercury emission analyzers are mounted on the stack or duct and do not require sample transport or gas conditioning. All on-line analyzers use elaborate calibration systems.

Several on-line mercury analyzers have recently been developed primarily for measuring total mercury emissions from waste incinerators. Application of these analyzers to coal combustion flue gas is difficult because mercury emission levels from coal combustion are much lower than those from waste incinerators, and the presence of acid gases and other flue gas components causes interference with the mercury measurement techniques. For the tests at Presque Isle, two different mercury analyzers were used: the PS Analytical Sir Galahad and the Semtech Hg 2000. These two instruments are described below.

4.3.1 Description of Mercury CEMS

Semtech Hg 2000 The commercial Semtech Hg 2000 mercury analyzer (Semtech Metallurgy AB, Lund, Sweden) is essentially a portable Zeeman-modulated CVAAS that can monitor Hg^0 continuously. By using an on-line reduction unit, total mercury can be monitored continuously. In the reduction unit, a reducing solution, such as stannous chloride (SnCl_2) or sodium borohydride (NaBH_4) is pumped to the sampling probe. The extracted gas sample and reducing solution are transported continuously through a mixing spiral to maximize the gas solution residence time and ensure complete conversion of Hg^{2+} to Hg^0 . The presence of SO_2 (>200 ppm) interferes with the chemical conversion process in the reduction unit and, therefore, must be removed prior to the measurement of total mercury. After conversion to Hg^0 , the sample gas is transferred to a Peltier cooled gas/liquid separator. The conditioned dry gas is then analyzed using the Semtech Hg 2000 analyzer. The analyzer uses Zeeman effect background correction by applying a modulated magnetic field to a mercury lamp to minimize interferences from the presence of SO_2 , hydrocarbons, and fine particulate in the flue gas sample. The Semtech has recently been upgraded to a Hg 2010. This upgrade was designed to reduce noise in the mercury signal. The operating range of the analyzer is $0.3 \mu\text{g}/\text{Nm}^3$ to $20 \text{mg}/\text{Nm}^3 \text{Hg}^0$, as specified by Semtech Metallurgy AB. The Semtech Hg 2000 has also been certified by TUEV Rheinland for

determining compliance with the German legal limit of $50 \mu\text{g}/\text{Nm}^3$ for total mercury from waste incinerators.

The Semtech Hg 2000 was designed to measure only Hg^0 . However, by passing the gas through a solution such as SnCl_2 which reduces all the mercury present in gas to Hg^0 , total mercury can be measured. However, it has been shown that the presence of SO_2 (>200 ppm) interferes with the chemical conversion process in the reduction unit and, therefore, must be removed prior to the measurement of total mercury. Therefore, a new conversion system was designed at the EERC to reduce the Hg^{2+} to Hg^0 . The primary feature of this system is an acid gas trap that removes the SO_2 without removing the mercury. The system can then be operated such that it can measure either total mercury or Hg^0 .

PS Analytical Sir Galahad The Sir Galahad analyzer was initially used to monitor total mercury continuously in the urban environment and in natural gas, but it can also be used in a variety of gaseous media including combustion flue gas. The analyzer is based on the principle of atomic fluorescence which provides an inherently more sensitive signal than atomic absorption. The system uses a gold-impregnated silica support for preconcentrating the mercury and separating it from potential interferences that degrade sensitivity.

The Sir Galahad was initially used in the manual mode which requires a four-step process to obtain a flue gas mercury measurement. In the first step, 2 L of flue gas is pumped through a gold trap which is maintained at a constant temperature. The gold trap is then removed from the flue gas stream and placed into the analyzer. Before the mercury is desorbed from the gold trap, a flushing step is initiated to remove any flue gas that may be present because it has a damping effect on the mercury fluorescence. When this is completed, the analysis step begins. The heating coil is activated, and the gold trap is heated to approximately 500°C . This desorbs the mercury from the trap, and the mercury is carried into the fluorescence detector. The gold trap is rapidly cooled by pumping argon over it, in preparation for the next sample. The total time for the entire process is about 5 minutes. The analyzer has since been automated.

The system is calibrated using Hg^0 as the primary standard. The Hg^0 is contained in a closed vial which is held in a thermostatic bath. The temperature of the mercury is monitored, and the

amount of mercury is calculated using vapor pressure calculations. Typically, the calibration of the unit has proven stable over a 24-hr period.

Although no mercury conversion is necessary for this instrument, a pretreatment system is also necessary for the PS Analytical CEM. It has been found that hydrochloric acid (HCl) in the presence of NO_x in the gas stream results in a low bias. Therefore, the same pretreatment system used for the Semtech Hg 2000 CEM is also used with the PS Analytical CEM. In this case, the acid trap removes the HCl in addition to the SO₂.

4.4 SAMPLING PROCEDURES

The following paragraphs and flow charts summarize the procedures used to sample the flue gases, recovery of the resultant samples and analyze the samples.

4.4.1 Preliminary Tests

Following equipment setup, preliminary test data was compiled at each of the emission test sites to verify pretest data/assumptions, determine nozzle sizes, and compute isokinetic sampling rates.

Test site geometric measurements were measured and sampling point distances were recalculated. A pitot traverse was performed to determine velocity profiles and to check for the presence/absence of cyclonic flow at each site. The cyclonic flow checks proved negative at all test locations. As appropriate, flue gas temperatures, dry gas composition, and moisture content were also determined by EPA Reference Methods 2, 3, and 4, respectively.

The preparation, sampling, and recovery procedures used to sample the emission points for speciated mercury conformed to those specified in the draft Ontario Hydro method and as described in the Site-Specific Sampling/Testing, Analytical and QA/QC plan. With the exception of the Unit No. 9 inlet test location, all tests were 120 minutes in duration. Each of the twelve traverse points at the Unit No. 5 inlet and outlet and Unit No. 9 outlet were sampled for 5 minutes each resulting in a 120 minute test period. At the Unit No. 9 inlet, each of the 35 traverse points were sampled for 4 minutes each. This resulted in a total test time of 140 minutes at the Unit No. 9 inlet test location. Readings were recorded at each traverse point at all test locations. Leak checks were performed at the beginning and end of each test run and before and after test port changes. Figure 4-3 depicts the

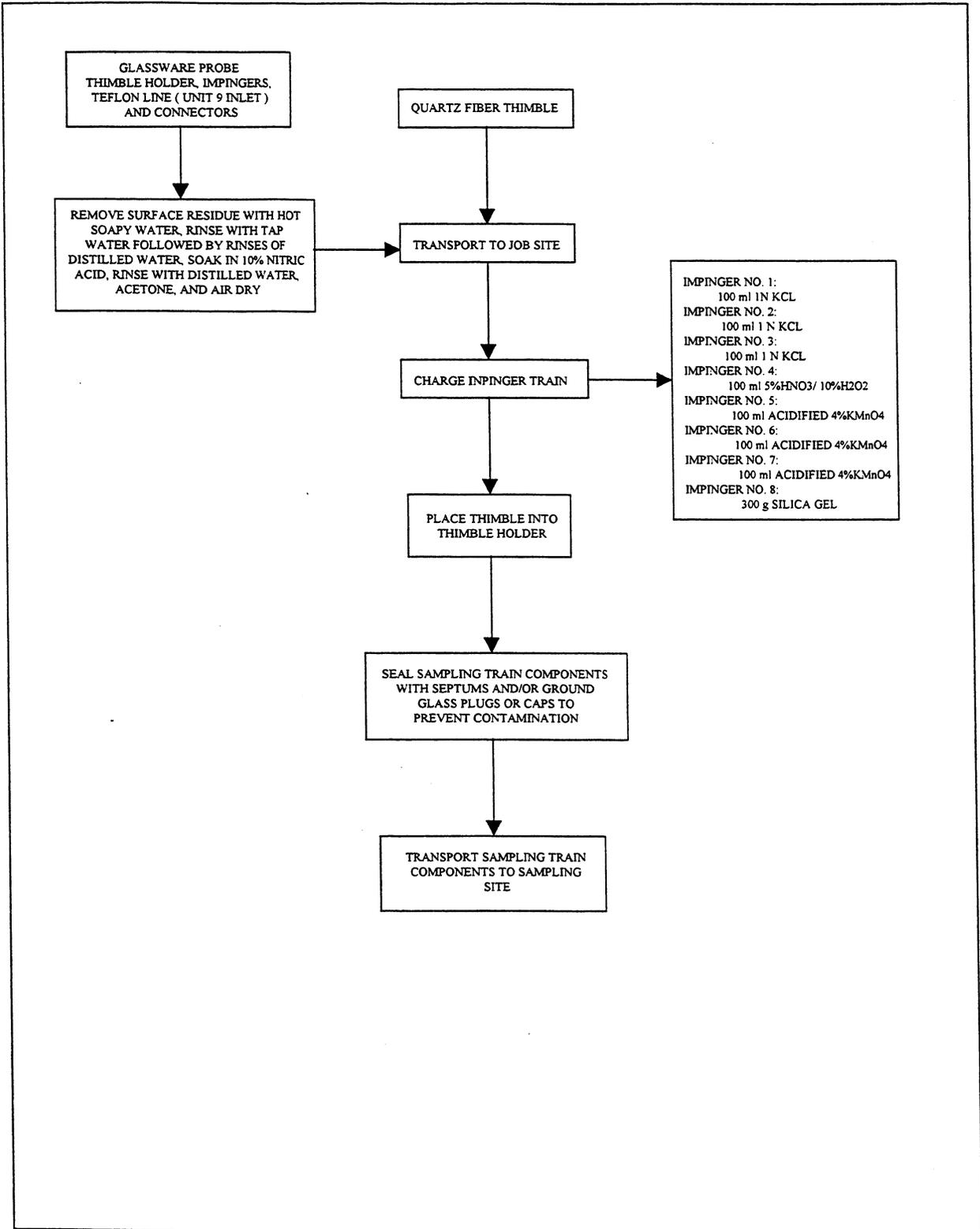


FIGURE 4-3
PREPARATION PROCEDURES FOR ONTARIO HYDRO SAMPLING TRAIN

train preparation. Figure 4-4 illustrates the sampling procedures. Figure 4-5 illustrates the sample recovery procedures.

4.5 ANALYTICAL PROCEDURES

The analytical procedures utilized during this program are described in the following subsections.

4.5.1 Process Stream Samples – CONSOL R&D

This subsection contains a list of the methods used by CONSOL for analysis of non-air process stream samples for mercury and other parameters. The analytical methods employed were those published by the American Society for Testing and Materials (ASTM), EPA, the American Public Health Association, EPRI or self validating methods used by CONSOL. Analytical methods applied are listed by analyte and matrix in Table 4-1. Method descriptions are provided below for the coal and ash samples.

4.5.2 Analytical Procedures for Coal Analysis

Proximate analysis – moisture, volatile matter, fixed carbon, ash

ASTM D 5142 Proximate Analysis of Coal and Coke by Instrumental Procedures

Moisture, volatile matter, fixed carbon, and ash were determined by establishing the loss in mass of a test specimen under rigidly controlled conditions of temperature, time, atmosphere, and specimen mass.

All samples were analyzed in duplicate. Duplicate results must meet ASTM criteria for repeatability. Since this is an empirical test, no certified standards are available. An in-house quality control sample, whose limits have been established by its utilization in analyzing round robin samples, was analyzed along with each batch of test specimens. Results for the control sample must be within established limits for the parameters being measured or the results for the entire set of test specimens are rejected and the test procedure is repeated.

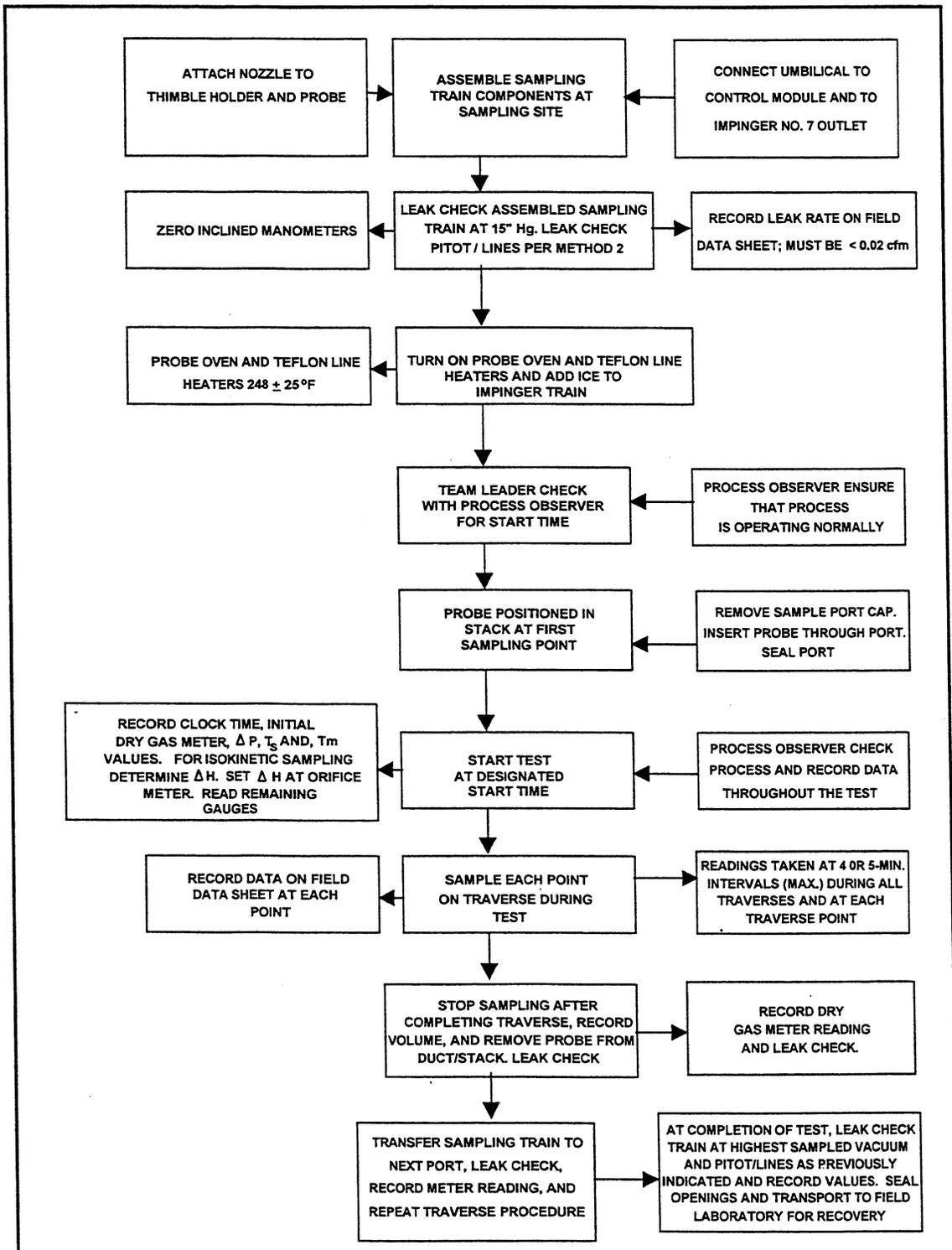


FIGURE 4 - 4
SAMPLING PROCEDURES FOR ONTARIO HYDRO TRAIN

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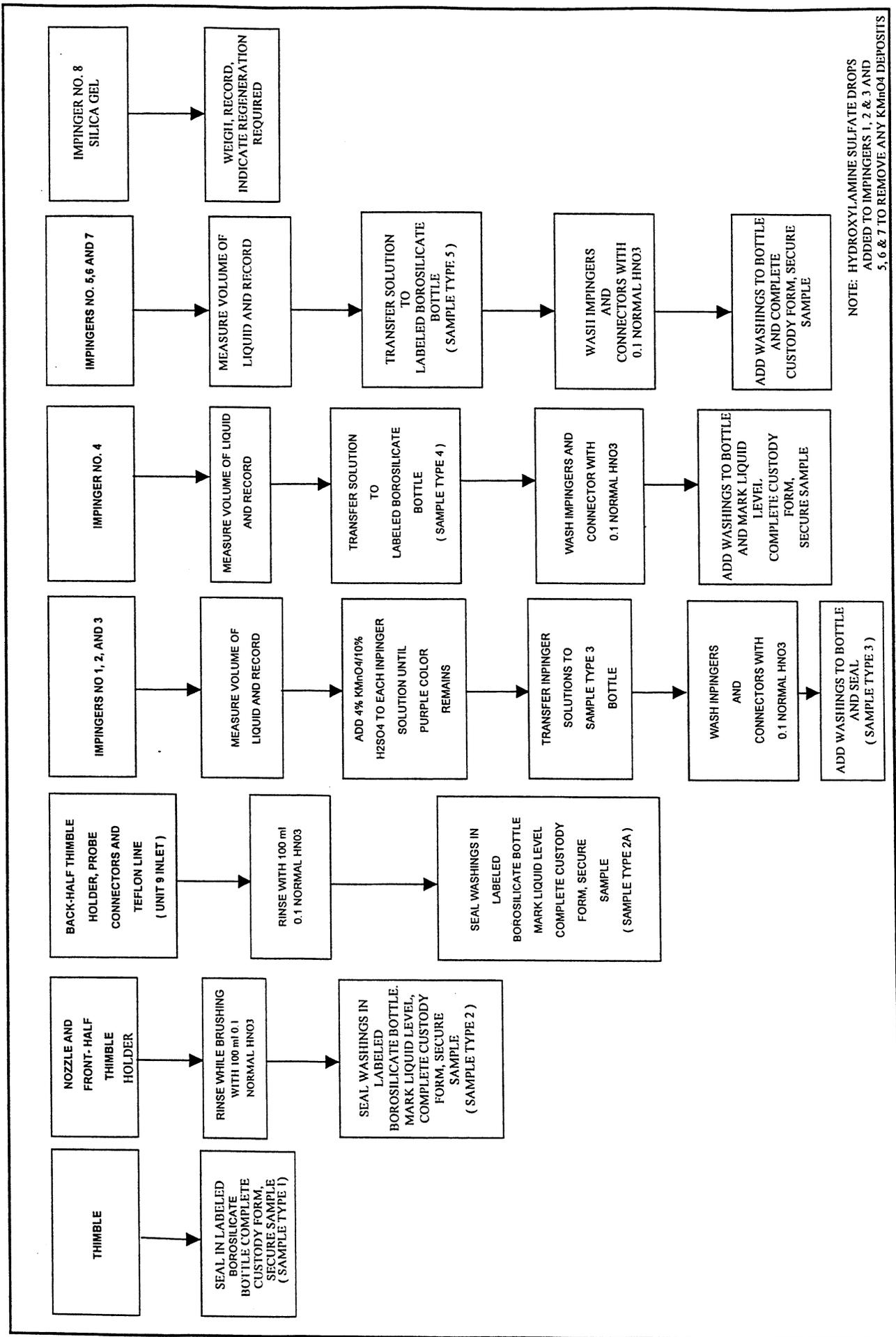


FIGURE 4 - 5
SAMPLE RECOVERY PROCEDURES FOR ONTARIO HYDRO METHOD

Table 4-1

Methods Used For the Analysis of
 Presque Isle Units 5 and 9
 Process Solids Streams Samples

Analyte	Coal	ESP Ash
Moisture, Ash, Volatile Matter, Fixed Carbon	ASTM D 5142-90	NA
Carbon, Hydrogen, Nitrogen	ASTM D 5373	ASTM D 5373
Sulfur	ASTM D 4239-85	NA
Oxygen	By Difference	NA
Higher Heating Value	ASTM D 1989-91	NA
Mercury	Proposed ASTM Method	Proposed ASTM Method
Chlorine	ASTM E 776 and EPA Method 300	NA
Major Ash Elements	ASTM D3682-78 ICP-AES	ICP-AES

4.5.2.1 Ultimate Analysis for Carbon, Hydrogen, and Nitrogen

ASTM D-5373 Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Coal Samples

Carbon, hydrogen, and nitrogen were determined concurrently in a single instrumental procedure. The procedure provides for the combustion and conversion of the subject elements in an oxygen stream in their entirety to carbon dioxide, water vapor, and nitrogen oxides. Carbon dioxide and water vapor were determined by infrared detection, nitrogen oxides are reduced to nitrogen and determined by thermal conductivity.

The instrument is calibrated daily by analyzing, as samples, National Institute for Standards and Technology (NIST) Standard Reference Material (SRM) Coal 1632b. The calibration is verified by analyzing an in-house quality control standard whose limits have been established by utilizing it in the analysis of round robin samples. The quality control sample is analyzed at least once for every ten samples analyzed. The results for the control sample must be within established limits for the parameters being measured or the test results obtained up to the last acceptable analyses of the control sample are rejected. All samples were analyzed in duplicate. Duplicate results must meet ASTM criteria for repeatability or the samples are reanalyzed.

4.5.2.2 Sulfur

ASTM D 4239-85 Method C Sulfur in the Analysis Sample of Coal Using High Temperature Tube Furnace Combustion with Infrared Absorption Detection.

A known mass of the test specimen was burned at high temperature in a stream of oxygen. Sulfur in the test specimen is quantitatively converted to sulfur dioxide that is measured by an infrared absorption detector.

The equipment is calibrated or has proper calibration verified daily by analyzing as samples, NIST Coal SRMs 1632b, 2682, 2683a, 2684a, 2685, or 2692. The calibration standard used is dependent on the expected concentration of sulfur in the samples. The calibration is validated and linearity is established by analyzing two other coal standards whose sulfur concentrations, when possible, brackets the calibration standard sulfur concentration.

All test specimens were analyzed in duplicate. Duplicate results must meet ASTM criteria for repeatability. An in-house quality control sample whose limits have been determined by its use in round robin programs, was analyzed at least once during each hour the equipment was in operation. The result for the control sample must be within established limits or the results for the test specimens analyzed up to the last acceptable analysis of the control sample are rejected.

4.5.2.3 Oxygen

The percentage of oxygen in a dried sample is calculated as follows:

$$\% \text{ Oxygen} = 100 - (\% \text{ash} + \% \text{carbon} + \% \text{hydrogen} + \% \text{nitrogen} + \% \text{sulfur})$$

When the chlorine concentration in the sample is determined, the following calculation applies:

$$\% \text{ Oxygen} = 100 - (\% \text{ash} + \% \text{carbon} + \% \text{hydrogen} + \% \text{nitrogen} + \% \text{sulfur} + \% \text{chlorine})$$

The accuracy and precision are a function of all the analytical results used in the calculation.

4.5.2.4 Higher Heating Value

ASTM D 1989-91 Gross Calorific Value of Coal by Microprocessor Controlled Isoperibol Calorimeters

The heating value of the test specimen was determined by burning a known mass under controlled conditions, in an atmosphere of oxygen, using a microprocessor-controlled isoperibol calorimeter. The system is calibrated by burning certified benzoic acid. Results are expressed in British thermal units per pound (Btu/lb).

Verification of proper calibration is established daily by analyzing benzoic acid. All samples were analyzed in duplicate and results must agree within ASTM limits.

Thermochemical corrections for fuse wire, nitric acid and sulfuric acid are made as per ISO and British Standard Methods.

4.5.2.5 Mercury

Proposed ASTM Method

A one gram, 60 mesh coal sample was digested with a solution of HCl and HNO₃ in a polycarbonate bottle at 80°C for 1 hour. The solid residue was filtered and the remaining solution was analyzed for Hg using cold vapor atomic adsorption. This technique is currently being reviewed by ASTM for Hg analysis of coal samples.

A known concentration of vapor-phase mercury is used to calibrate the instrument. The calibration is validated by analyzing NIST 1632b and/or NIST 1635.

4.5.2.6 Chlorine

Preparation of the coal samples will follow ASTM Method D-2013. Following air drying and riffing the coal samples were pulverized until 100% of the sample passed the 60-mesh screen.

The prepared coal sample was weighed. The weighed sample was oxidized by combustion in a bomb with a bicarbonate/carbonate solution and the amount of chlorine present was determined by ion-chromatography (IC) using EPA Method 300 procedures.

4.5.2.7 Major Ash Elements

Major Ash Elements analysis included Na₂O, K₂O, MgO, CaO, Fe₂O₃, TiO₂, P₂O₅, SiO₂, Al₂O₃, and SO₃.

A sample of 60 mesh coal was ashed according to the method outlined in ASTM D3682-78. The resulting ash is pressure-digested using hydrochloric acid, hydrofluoric acid and nitric acid.

The concentrations of the ten major ash elements were determined by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). All samples are digested and analyzed in duplicate. Duplicate analyses must meet the repeatability limits listed in ASTM D3682-78. A mass balance of 97.5-101.5 weight percent must be obtained for the ten elemental oxides. Samples not meeting this requirement are redigested and reanalyzed.

NIST fly ash 1633a is used to calibrate the ICP-AES. The calibration is checked with a secondary coal ash standard. The calibration is reassessed every eight samples by analyzing a quality control standard. The instrument is recalibrated as required.

4.5.3 Analytical Procedures for ESP Ash Samples

4.5.3.1 Carbon

ASTM D 5373

Carbon was determined using the same procedure outlined for coal analysis, Section 7.1.1.1. The instrument was calibrated daily by analyzing as samples NIST SRM 2704. All samples were analyzed in duplicate. Duplicate results must agree within 5% of the of the average of the two results for samples with carbon contents >1% or within 15% for samples <1% or the analysis is repeated. A quality control sample was analyzed once for every ten samples analyzed. The result for the control sample must be within ASTM repeatability or the results for the test specimens analyzed up to the last acceptable analysis of the control sample are rejected. Proper calibration must be re-established before resuming analysis of test specimens.

4.5.3.2 Mercury

Proposed ASTM Method

Analysis and quality control follow the description outlined under coal analysis, Section 4.5.2.5. The calibration was validated by analyzing NIST fly ash 1633a.

4.5.3.3 Major Ash Elements

Analysis and quality control follow the description outlined under coal analysis Section 4.5.2.7 except that the ash samples were not prepared (ashed) per ASTM D3682-78.

4.6 AIR SAMPLE ANALYSIS PROCEDURES

Speciated Mercury Analysis Procedures

Samples collected for speciated mercury analysis were contained in six different media:

Sample Fraction	Container No.	Mercury Form
▪ Thimble	Container No. 1	Particulate Hg
▪ Nozzle and front-half thimble holder nitric acid	Container No. 2	Particulate Hg
▪ Back-half thimble holder, probe, and connector rinses	Container No. 2A	Oxidized (Hg ²⁺)
▪ KCl impingers 1, 2, and 3 and rinses	Container No. 3	Oxidized (Hg ²⁺)
▪ HNO ₃ /H ₂ O ₂ impingers and rinses	Container No. 4	Elemental (Hg ⁰)
▪ KMnO ₄ /H ₂ SO ₄ impingers 5, 6, and 7 and rinses	Container No. 5	Elemental (Hg ⁰)

Analytical procedures and calculations for the mercury determination were performed as specified in the Ontario Hydro Method. The analytical procedures are outlined below.

All Ontario Hydro impinger sample fractions were analyzed onsite by EERC following sample collection and recovery. Following field sample preparation, samples 2A, 3, 4 and 5 were analyzed onsite by EERC using cold vapor atomic absorption (CVAA). The particulate sample fractions (samples 1 and 2) digestion and analysis were not performed in the field.

A Leeman Labs P-5200 CVAA was used in the field for mercury determination. Each day, a four-point calibration curve was completed using matrix-matched standards. A QC standard of a known analyte concentration was analyzed immediately after the instrument was standardized in order to verify the calibration. The values obtained must read within 5% of the true value. After the initial QC standardization was completed, standards were run every five samples to verify the slope of the calibration curve. The check standards must read within 5% of the expected value. All samples were then run in duplicate, and one in every ten samples were spiked to verify analyte recovery.

The particulate samples (1 and 2) were combined, digested and analyzed using CVAA at the EERC laboratory in Grand Forks, North Dakota.

The mercury results for each sample fraction were reported as total micrograms (ug) per sample.

In addition, each quartz thimble used for sampling at the ESP inlets and outlets were tared and the total particulate collected in each thimble was measured to determine the particulate collected by the ESP. This data was used to support the material balance calculations.

A schematic of the Ontario Hydro method analytical procedures is provided in Figure 4-6.

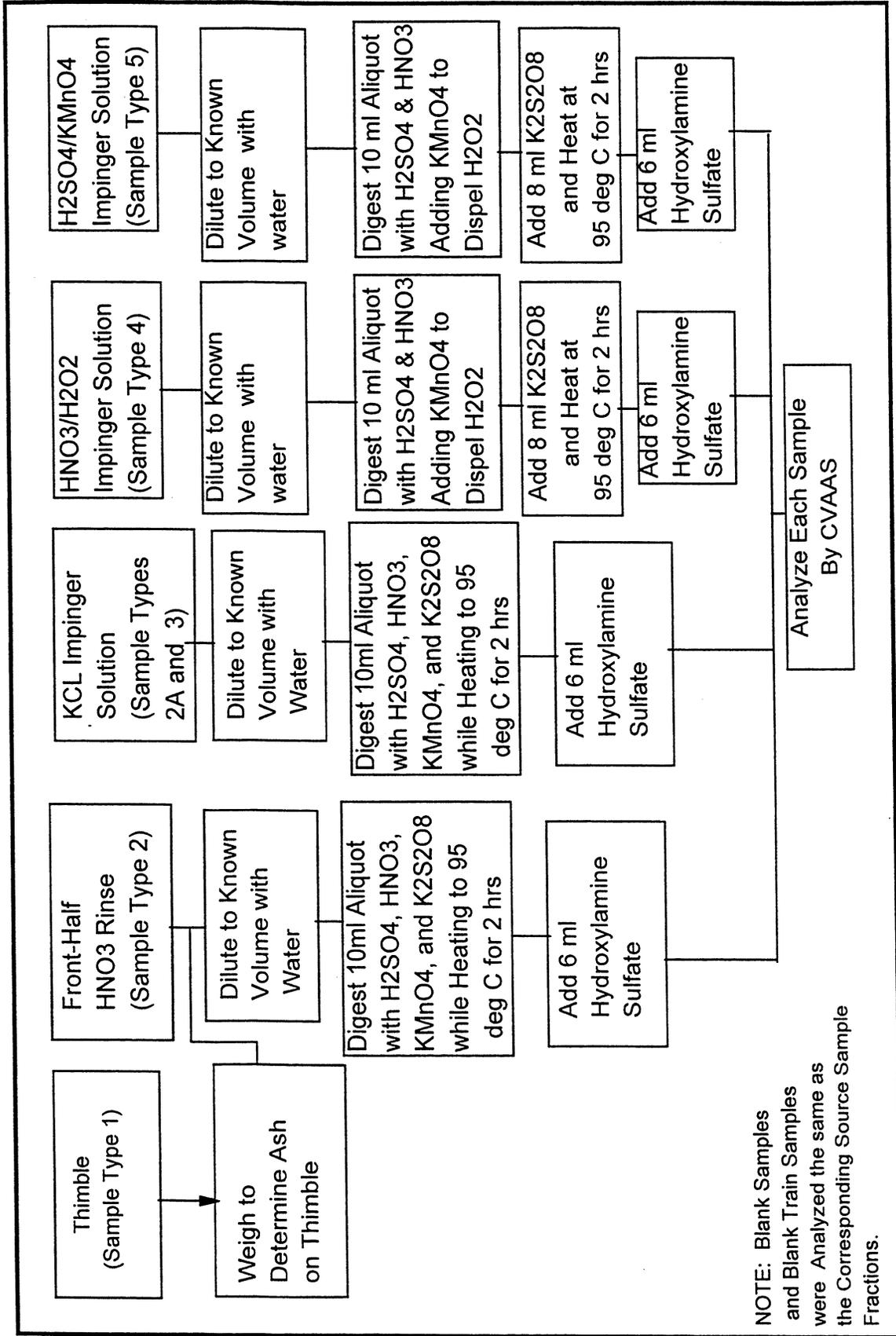


FIGURE 4 - 6

ANALYTICAL PROCEDURE FOR ONTARIO HYDRO SAMPLING TRAIN

5. QUALITY ASSURANCE SUMMARY

This section discusses results for QC samples collected during the test program. Discussions are provided for stack gas samples (Subsection 5.1) and coal and ash samples (Subsection 5.2).

5.1 STACK SAMPLE QA/QC RESULTS

This section provides detailed information regarding the QA/QC activities associated with stack sample collection, analysis, and reporting.

This summary pertains to all test data collected from sampling activities performed on Units 5 and 9 during the period of 13 through 20 July 1999. Analyses were performed on these samples for speciated mercury.

Project data quality objectives, as measured by precision, accuracy and completeness, were evaluated. Additionally, holding times, spike recoveries, laboratory blanks, and calibrations were evaluated to determine overall data quality based on criteria specified in the Site-Specific Sampling/Testing, Analytical and QA/QC Plan and the Quality Assurance Project Plan.

5.1.1 Stack Sample Collection and Calculations

Field QA/QC activities associated with the collection of stack Ontario Hydro method emission samples included pre- and post-test calibrations of sampling equipment, adherence to the proper sampling method procedures, documentation of field data, recovery of samples without contamination, and collection of appropriate field train and site blank samples.

Copies of the field data sheets are contained in Appendix C. Chain of custody forms are included in each laboratory report and provide a list of all samples collected and submitted for analysis during the test program. The laboratory reports are provided in Appendix D.

Proper field sampling procedures include sampling at 100% isokinetic $\pm 10\%$ and maintaining sample train leakage rates at ≤ 0.02 CFM. Table 5-1 contains a summary of all isokinetic sampling rates for all tests, initial and final leak check rates, and pre- and post-test dry gas meter calibration results. This table indicates that all test runs were within the acceptable ranges for all

Table 5-1

Stack Emission Sampling Field QA/QC Results

Test Location	Test Run	Isokinetic Sampling Rate ¹	Initial Leak Check Rate ²	Final Leak Check Rate ²	Gas Meter Calibration Values ³	
					Pre	Post
Unit No. 5 ESP Inlet	1	98.7	0.012	0.012	1.009	0.9772
	2	97.6	0.009	0.007	1.009	0.9772
	3	100.7	0.008	0.004	1.009	0.9772
Unit No. 5 ESP Outlet	1	98.1	0.010	0.006	1.0098	1.0322
	2	98.9	0.008	0.004	1.0098	1.0322
	3	98.8	0.005	0.004	1.0098	1.0322
Unit No. 9 ESP Inlet	1	102.8	0.006	0.006	1.009	0.9772
	2	102.5	0.009	0.008	1.009	0.9772
	3	102.1	0.005	0.008	1.009	0.9772
Unit No. 9 ESP Outlet	1	100.7	0.005	0.006	1.0098	1.0322
	2	100.4	0.006	0.006	1.0098	1.0322
	3	100.3	0.010	0.004	1.0098	1.0322

- 1 Isokinetic rate must be $100 \pm 10\%$. All sampling rates met isokinetic criteria.
- 2 Initial and final leak check value must be ≤ 0.02 CFM. All leak checks were acceptable.
- 3 Post-test calibration must be ± 0.05 of pre-test value. All calibration values were acceptable.

Note:

Silica gel impinger exit temperature maintained $\leq 68^\circ\text{F}$ during all test periods.

field measurements. Appendix F contains the stack test equipment calibration data.

5.1.2 Sample Chain of Custody

Sample custody procedures were followed per Section B-2 of the QAPP. Following collection and recovery, all samples were transferred to representatives of EERC. As described in Section 4 of this report, sample types 2A, 3, 4, and 5 were analyzed onsite. Sample types 1 and 2 (which contained the particulate bound mercury) were analyzed off site at the EERC laboratory. The sample storage area was locked and secured during off-hours when test representatives were not on-site.

All samples arrived in good condition to the EERC laboratory.

5.1.3 Stack Emission Blank Sample Results

Blank samples were submitted with the stack emissions samples as designated in the test method and QAPP. During each set of the three test runs, a blank sample train was setup, leak checked and recovered at each of the test locations on Units 5 and 9. Site blanks of the thimbles impinger train solutions and recovery solutions were retained and analyzed. No mercury above the analytical detection limit was present in any of the site blanks or blank train samples collected for both Units 5 and 9.

5.1.4 Ontario Hydro Analysis Holding Times

Holding time is the period from sample collection to sample analysis. All holding times for all Ontario Hydro sample parameters were within the maximum time period of 28 days per the Site-Specific Sampling/Testing Analytical and QA/QC Plan. As previously mentioned the bulk of the sample analysis were performed onsite within 1 to 2 days following sample collection.

5.1.5 External Performance Evaluation Audits

No performance evaluation audits were provided to WESTON by the regulatory agencies during the test program.

5.1.6 Ontario Hydro Analysis QA/QC Results and Conclusions

As previously stated the Site-Specific Sampling/Testing, Analytical and QA/QC Plan and QAPP developed for the Presque Isle sampling program identified a number of QA/QC procedures to ensure the quality of the data generated from the testing. For the Ontario Hydro sample analysis these included the following.

- Blank Reagent Sample Analyses.
- Field Blank Train Samples.
- All samples analyzed in duplicate using CVAA.
- Every 10th sample analyzed in triplicate using CVAA.
- The instrument calibrated and properly set up prior to use each day.
- The use of check standards every 5 samples to verify the calibration.

For the tests at Presque Isle, there was a deviation from the test plan. Each individual sample was not analyzed in duplicate. Triplicate analysis were performed on one in every ten samples. This deviation does not impact the quality of data.

Based on previous experience it is clear that once a mercury sample has been prepared, the actual precision of CVAA (based on a single analysis) is very high. The EERC has maintained extensive logs documenting this precision and these logs are available upon request. As required by the EERC Standard Operating Procedures (SOPs), the State of North Dakota and EPA certifications, new or different type analyses requires that all samples be done in duplicate until the precision of the analyses is demonstrated. For mercury analyses using CVAA this demonstration has been done for approximately 2 years after EERC began routinely analyzing these type samples. Once this has been demonstrated, the samples are no longer done in duplicate. The 1 in 10 triplicate analysis requirement is always done.

It is clear from the data obtained during the Presque Isle test and all the previous data the EERC has obtained that the duplicate requirement is not needed once the precision of the instrument has clearly been established. This is shown by the excellent precision obtained by the triplicate samples that were analyzed for this program. As shown in Table 5-2, in all cases the standard deviation is well below 10% which is the criteria for duplicate analysis per the Ontario Hydro method.

If a problem occurs such that the instrument is giving incorrect readings the most likely cause is that the instrument is going out of calibration. For this reason it is important that calibration check samples be analyzed on a regular basis. The test plan required a calibration check every 5 samples as was done at Presque Isle. As can be seen from Table 5-3 the calibration check samples were always within 10% of the expected value during each day.

If an error exist in the Ontario Hydro Method it is not the analysis of the sample using CVAA but in the preparation steps that allow the analyses to be completed. These steps require experienced people with a solid chemistry background to perform the preparation properly. The individuals who performed sample preparation have over 3 years experience in doing these types analyses in the laboratory and in the field. As a quality control check, the EERC has initiated a policy of doing field and laboratory spiked samples as well as field blanks. A field spike is a sample train that is completely assembled and a known amount of mercury (unknown to the chemist) is added to each of the impingers. The train is then brought to the sampling site with the other trains and remains at the site until sampling is completed for the day. Once the sampling is completed, the field spike train is then disassembled and the samples recovered, prepped, and analyzed. Although this is not a quality control measure that is required by the method, it was performed at Presque Isle and one field spike was completed by the EERC for each day of the analysis. The results of field spikes and laboratory spikes are shown in Table 5-4. As can be seen, the recovery of the field and laboratory spikes were excellent. All were easily within the 80 to 120% criteria.

All the data (including the mass balance calculations) shows that the results obtained for the Presque Isle sampling activities were of high quality. The greatest source of error in the method is the sample preparation steps. The excellent recovery of the field spikes show that the EERC preformed these procedures well. The low standard deviation obtained for the triplicate analyses demonstrates that the instrument was precise and the calibration check standards show that the instrument was calibrated properly and was not out of calibration.

All mercury speciation stack emissions data and results are believed to be representative of the emissions encountered during the test periods and appear to be acceptable following QA/QC review.

Table 5-2
 Triplicate Analyses Results of Mercury Samples Data Using CVAA

SAMPLE ID	KCl Solution						H ₂ O ₂ Solution						KMnO ₄ Solution					
	Sample No. 1			Sample No. 2			Sample No. 2			Sample No. 3			Sample No. 2			Sample No. 3		
	1	2	3	S.D.	1	2	3	S.D.	1	2	3	S.D.	1	2	3	S.D.		
BO5-IN-1	1.5	1.41	1.46	0.0451	----	----	----	----	0.37	0.42	0.43	0.0321	0.37	0.42	0.43	0.0321		
BO5-IN-3	1.58	1.57	1.57	0.0058	----	----	----	----	0.83	0.83	0.83	0.0000	0.83	0.83	0.83	0.0000		
BO9-IN-1	0.51	0.48	0.49	0.0153	----	----	----	----	28.92	28.84	28	0.5096	28.92	28.84	28	0.5096		
BO9-OUT-1	2.09	2.09	2.09	0.0000	----	----	----	----	----	----	----	----	----	----	----	----		
BO9-IN-3	0.36	0.43	0.39	0.0351	0.09	0.29	0.24	0.1041	26.72	27.48	29.2	1.2706	26.72	27.48	29.2	1.2706		
BO9-OUT-3	1.93	1.92	1.9	0.0153	----	----	----	----	----	----	----	----	----	----	----	----		

Table 5-3

Percent Recovery of Calibration Check Standards

Analysis Day No.				
DAY 1, %	DAY 2, %	DAY 3, %	DAY 4, %	DAY 5, %
101	102	103	101	105
104	101	101	105	102
103	104	101	102	103
102	101	99.9	104	103
	101	98.5	101	
	103	97.5	101	
			100	
			98.8	
			99.6	

Table 5-4
Results of Field and Laboratory Spiked Samples.

Spike Added To Solution, ppb	Spike Location	Measured Hg in Original Solution, µg/L	Measured Hg in solution After Spike, µg/L	Measured Spike, ppb	Spike Recovery, %
5	Lab	1.46	7.06	5.6	112.0
10	Lab	1.46	12.08	10.62	106.2
2	Field	0	2.06	2.06	103.0
5	Lab	2.06	7.67	5.61	112.2
5	Lab	0	4.59	4.59	91.8
10	Lab	0	10.53	10.53	105.3
2	Field	0	1.95	1.95	97.5
10	Lab	0	10.62	10.62	106.2
5	Lab	0.41	5.56	5.15	103.0
2	Field	0	1.77	1.77	88.5

5.2 PROCESS SOLID SAMPLE QA/QC RESULTS

The Site-Specific Sampling/Analytical and QA/QC Plan and the QAPP for this program identified the analytical QC objectives for the process solid sample analysis.

A detailed QA/QC discussion with results of all QA/QC analysis results is provided in Appendix D of this report. A brief summary of the results follows.

Analytical Precision

All process stream coal and ash samples were analyzed in duplicate. Analytical precision was determined by the percent relative difference (RPD) obtained by the duplicate sample analyses. The RPD objective for the mercury in coal and ash was $\leq 20\%$. The RPD for moisture ultimate/proximate, carbon and major ash elements is $\leq 10\%$. The RPD objectives for duplicate analyses were met in all cases for all analytes.

Analytical Accuracy

The objectives for accuracy for spike samples and laboratory control samples were 70 to 130% for the mercury in coal and ash, 90 to 110% for ultimate/proximate and carbon and 80-120% for chlorine and the major ash elements. The objectives for accuracy were satisfied in all cases. Most values ranged between 94 and 104% recovery.

5.2.1 Holding Times

All process samples were analyzed within the required holding times as specified in the Site-Specific Sampling/Testing, Analytical and QA/QC Plan.

5.2.2 Process Sample QA/QC Conclusions

All solid sample process data and results appear to be acceptable following QA/QC review.

5.3 COMPLETENESS

Laboratory completeness is a measure of the amount of valid measurements obtained from all the laboratory measurements associated with this test program. The number of valid measurements satisfied the laboratory completeness goal identified in the Site-Specific Sampling/Testing, Analytical and QA/QC Plan QAPP of greater than 90 percent.

Based on a review of all QA/QC results, no data has been lost or qualified as not satisfied the QC criteria for precision and accuracy. Therefore, a 100% completeness can be assigned for both sampling and analysis.