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February 15, 2000

Mr. William Grimley / Ms Lara Autry  
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Room Number E-108 / E-128  
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RE: Mercury Flue Gas Measurements  
Presque Isle Power Plant  
Units 1-4 and 6

Dear Mr. Grimley and Ms Autry:

Enclosed are (3) copies of the test report for flue gas measurements performed on Units 1-4 and Unit 6 at the Presque Isle Power Plant. Reports for ICR-required measurements on Units 5 and 9 were sent to you via correspondence dated November 15, 1999. Wisconsin Electric and the US DOE decided to conduct additional voluntary measurements at this power plant to gain additional insights on the behavior of mercury in flue gases from units equipped with different particulate control devices ( Units 1-4 were outfitted with a fabric filter in early 1999; Unit 6 precipitator had been outfitted with an Environmental Elements Corporation Particulate Agglomerator in 1998 ). These measurements were conducted by the same US DOE-sponsored team of investigators as were used for the Units 5 & 9 work; namely, the Energy & Environmental Research Center; CONSOL, R&D; and Roy F. Weston, Inc. This report was also prepared by Roy F. Weston, Inc.

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

Sincerely,

A handwritten signature in black ink, appearing to read 'Terry Coughlin'.

Terry Coughlin  
Air Quality Team Leader

cc: Tom Brown, US DOE ( w/o report )  
Jeff O'Neill, RFW ( w/o report )  
Dennis Laudal, EERC (w/o report )  
Matt Devito, CONSOL ( w/o report )

**CONTRACT NO. DE-AC-22-93PC93255  
WESTON PROJECT NO. 20009.011.006.0700  
EMISSIONS TEST REPORT  
WISCONSIN ELECTRIC POWER COMPANY  
PRESQUE ISLE POWER PLANT  
MARQUETTE, MICHIGAN**

**JANUARY 2000**

**DOE PROJECT:  
ASSESSMENT OF SPECIATED MERCURY  
EMISSIONS FROM COAL-FIRED BOILERS  
UNIT NO. 6 AND UNITS 1-4**

Prepared for:

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## EXECUTIVE SUMMARY

This test report presents the results of the speciated mercury test program performed on Unit No. 6 and Units 1-4 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 14 through 16 July 1999.

The WEPCO Presque Isle Power Station was selected by the U. S. Environmental Protection Agency (EPA), Office of Air Quality Planning and Standards (OAQPS) to satisfy the Information Collection Request (ICR) requirement. During the ICR test program, mercury speciation testing was performed on the inlets and outlets of the electrostatic precipitators (ESPs) serving Units No. 5 and 9. The results obtained during the ICR test program are provided in the Information Collection Request, Assessment of Speciated Mercury Emissions from Two Coal-Fired Boilers, test report dated November 1999. Additional testing and analysis (not required as part of the ICR) was performed on Unit No. 6 and 1-4 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 ESP serving Unit No. 6 and the newly installed baghouses serving Units 1, 2, 3, and 4. Representative samples of the coal, ESP and baghouse ash streams were sampled in conjunction with the emissions testing. Mercury CEMs measured mercury concentrations at the ESP outlet and baghouse outlet (stack) of Units 1-4.

Table ES-1 presents a summary of the average speciated mercury concentrations and mass rate results for the Unit No. 6 and Units 1-4 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. 6 and Units 1-4. No removal efficiencies are provided for Units 1-4, since baghouse inlet testing was not performed.

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 NO. 6 and UNITS 1-4**

PARAMETERS	Unit 6 Inlet		Unit 6 Outlet		Units 1-4 Outlet	
	Average of Test Runs	Average % of Total	Average of Test Runs	Average % of Total	Average of Test Runs	Average % of Total
<b>PROCESS DATA:</b>						
Unit Load, MW	74.7	--	74.7	--	130	--
Coal feed rate, lb/hr.	63,967		63,967		113,000	
Coal Btu content, Btu/lb. (as received)	12,097		12,097		12,200	
Heat Input, 10 <sup>6</sup> Btu/hr (F-Factor)	776		776		1,726	
<b>PARTICULATE BOUND MERCURY EMISSIONS:</b>						
Conc., ug/m <sup>3</sup>	2.57	77.3	0.035	1.7	< 0.005	0.0
Conc., ug/Nm <sup>3</sup>	2.76		0.038		< 0.005	
Emission rate, lbs/10 <sup>12</sup> Btu.	2.08		0.030		< 0.004	
Emission rate, lbs/hr.	1.62E-03		2.37E-05		< 7.09E-06	
<b>OXIDIZED MERCURY EMISSIONS:</b>						
Conc., ug/m <sup>3</sup>	0.58	17.5	0.72	50.5	0.20	80.6
Conc., ug/Nm <sup>3</sup>	0.62		0.78		0.22	
Emission rate, lbs/10 <sup>12</sup> Btu.	0.47		0.62		0.18	
Emission rate, lbs/hr.	3.67E-04		4.84E-04		3.10E-04	
<b>ELEMENTAL MERCURY EMISSIONS:</b>						
Conc., ug/m <sup>3</sup>	0.18	5.2	0.68	47.8	0.05	19.4
Conc., ug/Nm <sup>3</sup>	0.19		0.73		0.06	
Emission rate, lbs/10 <sup>12</sup> Btu.	0.14		0.59		0.05	
Emission rate, lbs/hr.	1.11E-04		4.56E-04		7.78E-05	
<b>TOTAL MERCURY EMISSIONS:</b>						
Conc., ug/m <sup>3</sup>	3.33	--	1.43	--	0.25	--
Conc., ug/Nm <sup>3</sup>	3.57		1.53		0.27	
Emission rate, lbs/10 <sup>12</sup> Btu.	2.70		1.23		0.23	
Emission rate, lbs/hr.	2.10E-03		9.56E-04		3.88E-04	
<b>TOTAL MERCURY REMOVAL EFFICIENCY: MERCURY MATERIAL BALANCE <sup>(1)</sup>:</b>						
	--	--	54.5%	--	--	--
	--	--	72.9%	--	88.0%	--

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

Wisconsin Electric Power Company's (WEPCOs) Presque Isle Power Station, located near Marquette, Michigan, was selected as one of the ICR study sites. Mercury speciation sampling was performed at the Presque Isle facility using the Ontario Hydro method. During the ICR test program mercury speciation testing was performed on the inlets and outlets of the electrostatic precipitators (ESPs) serving Units No. 5 and 9. The results obtained during that test program are provided in the Information Collection Request, Assessment of Speciated Mercury Emissions from Two Coal-Fired Boilers, test report dated November 1999.

During the ICR Test Program, DOE and WEPCO requested that additional mercury speciation testing be performed on the Unit No. 6 ESP and the newly installed baghouses serving Units 1, 2, 3 and 4. This test report presents the test data and test results for the mercury speciation test program performed on Unit No. 6 and Units 1-4.

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 and EERC. 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 14 through 16, 1999.

This test report presents the test data and test results of the mercury speciation sampling program performed on Unit No. 6 and Units 1-4 at the Presque Isle Power Station and contains all test results and discussions. 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. This report format follows EPA's Emissions Measurement Center (EMC) guideline document (GD-043) titled, Preparation and Review of Emission Test Reports which is required for ICR report submittals and satisfies the data reporting requirements of this program.

## **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 precipitator (ESP) serving Unit No. 6 and on the common stack of the baghouses (A and B) serving Units 1-4. Mercury continuous emission monitoring systems (CEMS) measured mercury concentrations at the ESP outlet and baghouse outlet (stacks) of Units 6 and 1-4. 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 coal fired boilers.
- Simultaneously measure concentrations and mass rates of speciated mercury at the inlet and outlet of the ESP on Unit 6.
- Obtain and analyze representative samples of the coal and 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 ash streams.
- Perform mercury measurements using mercury CEMS in conjunction with mercury testing at the Unit 6 ESP outlet and Units 1-4 outlet.

- Document corresponding boiler and ESP or baghouse operations along with facility CEMS data.

Although not a specific objective of the original program, the major ash elements for both the coal and ash samples were determined to provide additional characterization of the coal and ash samples.

A Site-Specific Sampling/Testing, Analytical and QA/QC Plan and Quality Assurance Project Plan (QAPP) dated May 1999 were developed for the ICR test program performed on Units 5 and 9 at Presque Isle. The sampling, analytical and Quality Assurance (QA) procedures used during testing on Unit No. 6 and Units 1-4 were based on those documents.

### **1.3 SAMPLE LOCATIONS**

Representative samples from the following solid streams were collected and analyzed during the test program:

- Clean Coal Feed.
- ESP Ash (Unit No. 6).
- Baghouse Ash (Units 1-4 baghouses A and B)

Flue gas stream emission samples were collected at the following locations:

- Unit 6 ESP Inlet and Outlet (Stack).
- Units 1-4 Baghouse 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:

<b>Contact Name</b>	<b>Project Responsibility</b>	<b>Telephone No.</b>	<b>Facsimile No.</b>
<b>WEPCO</b>			
Mr. Dave Michaud	Corporate Environmental Contact	(414) 221-2187	(414) 221-2169
Mr. Todd Rapavi	Plant Environmental Contact	(906) 226-5738	(906) 226-5750
<b>DOE</b>			
Mr. Tom Brown	Project Manager	(412) 386-4691	(412) 386-5917
<b>WESTON</b>			
Mr. Jeff O'Neill	Project Manager	(610) 701-7201	(610) 701-7401
<b>EERC</b>			
Mr. Dennis Laudal	Project Leader	(701) 777-5138	(701) 777-5181
<b>CONSOL</b>			
Mr. Matt Devito	Project Leader and Process Observer	(412) 854-6679	(412) 854-6613

**Table 1-1**

**Presque Isle Power Station  
Unit No. 6 and Units 1-4  
Process Solid and Flue Gas Streams with  
Pollutants/Parameters**

<b>Location/Stream Type</b>	<b>Pollutants or Parameters</b>	<b>Frequency</b>
Units 1-4 and Unit 6 Clean Coal Feed	Heating value Moisture Ultimate and proximate analyses Mercury (Hg) content Chlorine (Cl) content Major ash elements	One composite sample per run (total of 3) in conjunction with flue gas sampling on Unit No. 6. One composite sample collected on Units 1-4 in conjunction with flue gas sampling.
Units 1 – 4 Baghouse Ash Unit No. 6 ESP Ash	Mercury content Carbon content Major ash elements	Grab samples obtained in conjunction with flue gas sampling. One sample obtained per test run.
Units 1-4 Baghouse Outlet Unit No. 6 ESP Inlet and Outlet (Stack)	Particulate bound and vapor phase mercury (including oxidized and elemental mercury speciation of vapor phase). Particulate loading <sup>1</sup>	Outlet sampling by Ontario Hydro method on Units 1-4. Inlet and outlet sampling by Ontario Hydro method on Unit No. 6. Mercury measurements by CEMS on the Units 1-4 and Unit No. 6 stacks (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 the Unit No. 6 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 is controlled by two newly installed baghouses. Baghouse 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 is 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 6 Coal Sampling**

Representative pulverized coal samples from Unit 6 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 airtight 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 6 ESP Ash Hopper Sampling**

Unit 6 is equipped with a cold-side ESP. The ESP has two rows of hoppers. Each row has three collection hoppers (total of 6). 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 hopper samples were combined into one composite (front row sample). The three back-row of hopper 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 Units 1-4 Coal Sampling**

Units 1 through 4 are fired from a common coal supply. One daily representative sample was taken from the coal feed pipes feeding Unit 4 on the July 16, 1999. Sampling was completed as discussed for Unit No. 6 using the same sampling equipment. Twelve sample increments were composited into a single composite sample. The increments were taken at 35-minute intervals with the first interval at the onset of the flue gas measurements at the stack and the final increment near the completion of the stack testing. 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 increments were stored in a double-lined plastic bag that was further sealed in an airtight plastic bucket. The individual composite coal samples were shipped from the plant to the CONSOL R&D 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.

### **2.2.4 Units 1-4 Ash Sampling**

Fly ash from Units 1 through 4 was collected by baghouses A and B. Each baghouse is equipped with 8 hoppers (total of 16 hoppers for both baghouses). Three composite ash samples were collected on July 16, 1999 in conjunction with of the three-flue gas Hg measurements made at the stack location. Each composite sample consisted of a 16 individual grab samples, one from each of the 16 hoppers. These samples were collected near the completion of the mercury speciation flue gas sampling periods. Ash for Test No. 1 was collected from 0925-0945 hours. Ash for Test No. 2 was collected from 1240-1300 hours. Ash for Test No. 3 was collected from 1500-1540 hours. These samples were collected from the hopper access ports located on the side of each hopper using a sample thief. These samples were size-reduced on-site, transferred to pre-labeled glass sample bottles, and transported to the CONSOL R&D 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 6 ESP Inlet**

The test site at the ESP inlet is vertically oriented and is a 6' 10" deep by 22' 4" 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 6 ESP inlet.

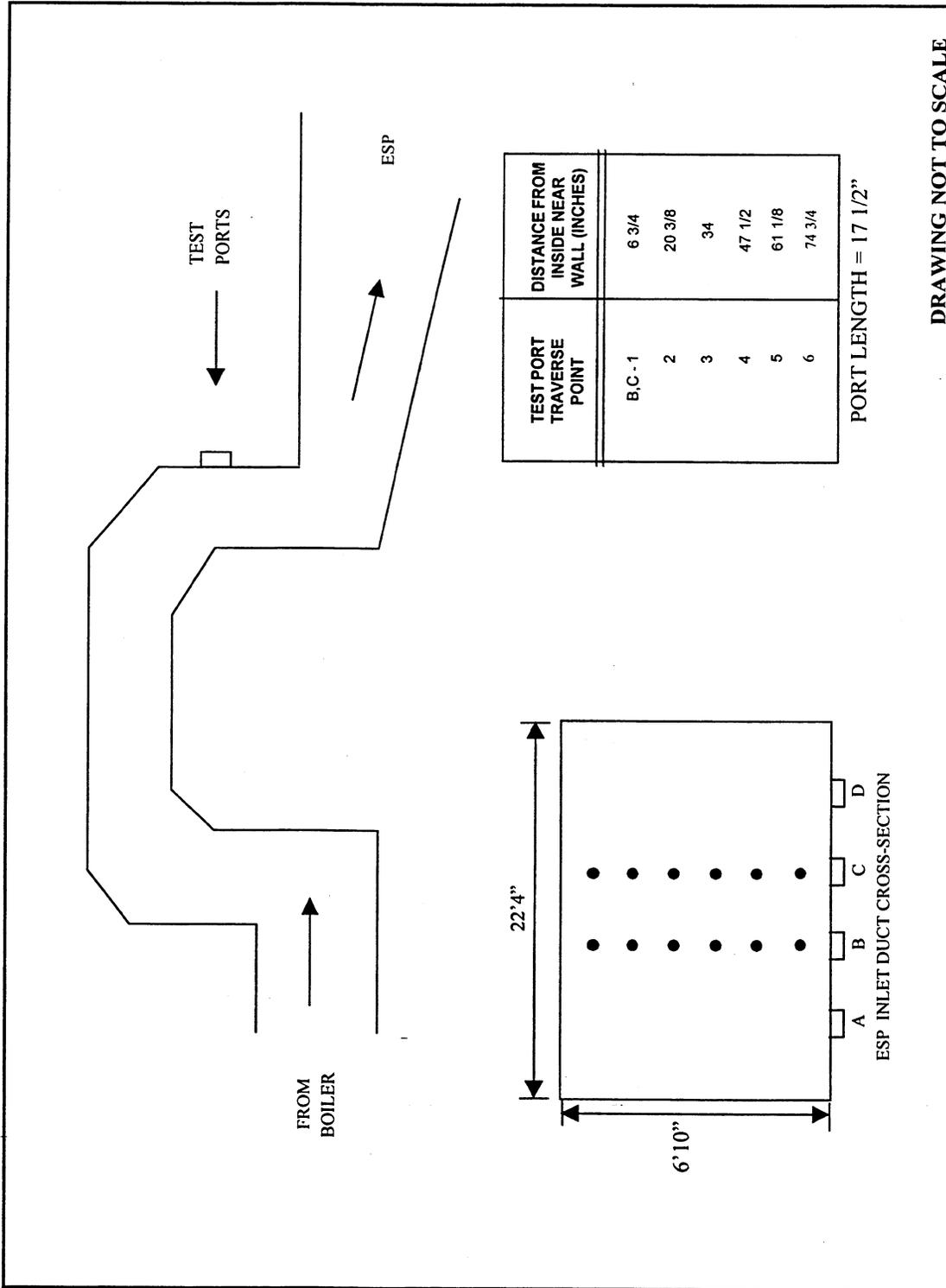
### **2.3.2 Unit 6 ESP Outlet (Stack)**

A total of four (4) 6" test ports are in place on the 9' ID flue, which serves Unit 6. 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. 6 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. 6 stack test location.

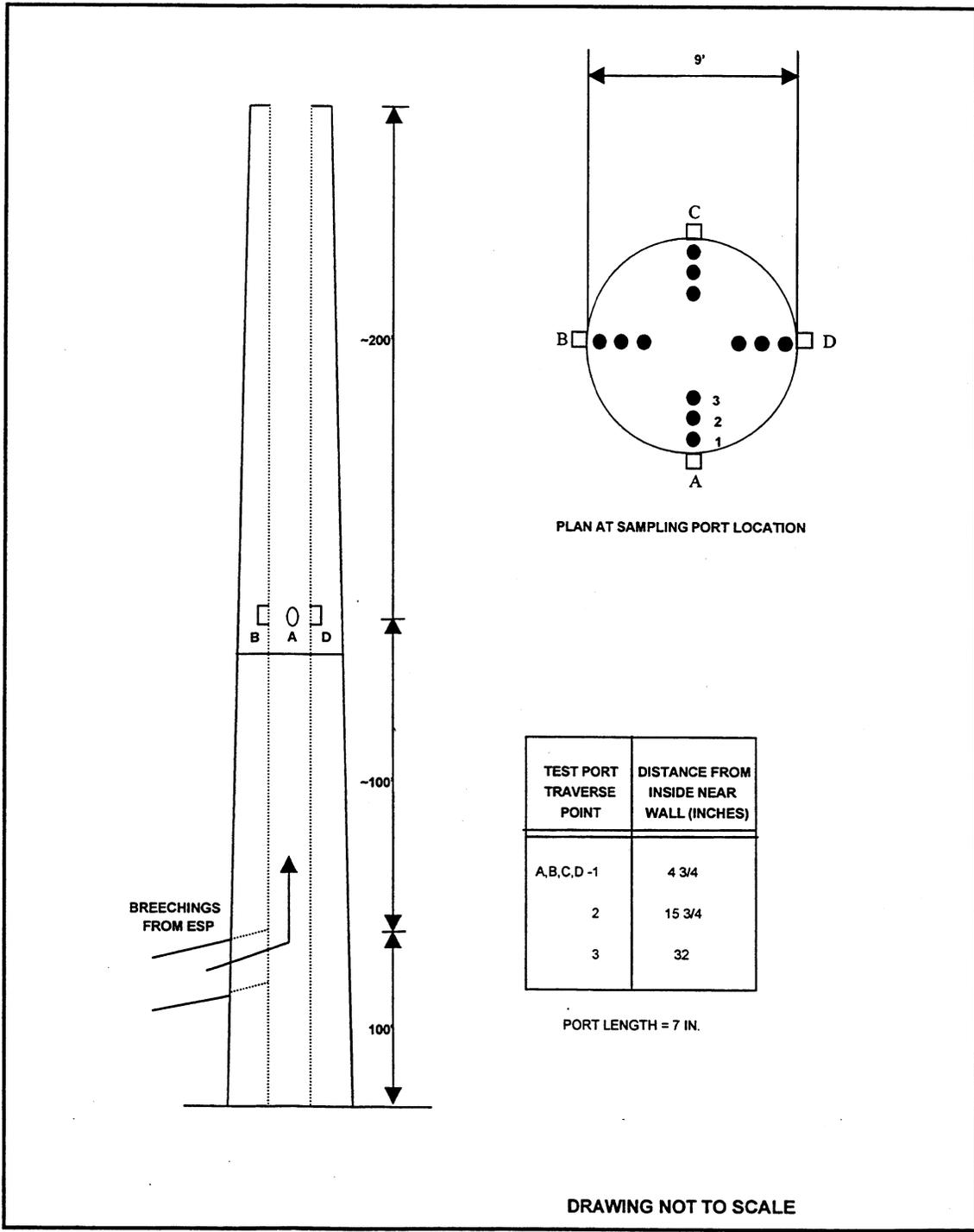
### **2.3.3 Units 1-4 Baghouse**

A total of four (4) 6" ID test ports are in place on the 11' ID flue which serves Units 1-4. The test ports are located ~100' (9 diameters) from the nearest downstream disturbance and ~200' (18 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 Units 1-4 stack is an ideal test location and satisfies all EPA Method 1 criteria for test port location. See Figure 2-3 for a schematic of the Units 1-4 stack test location.

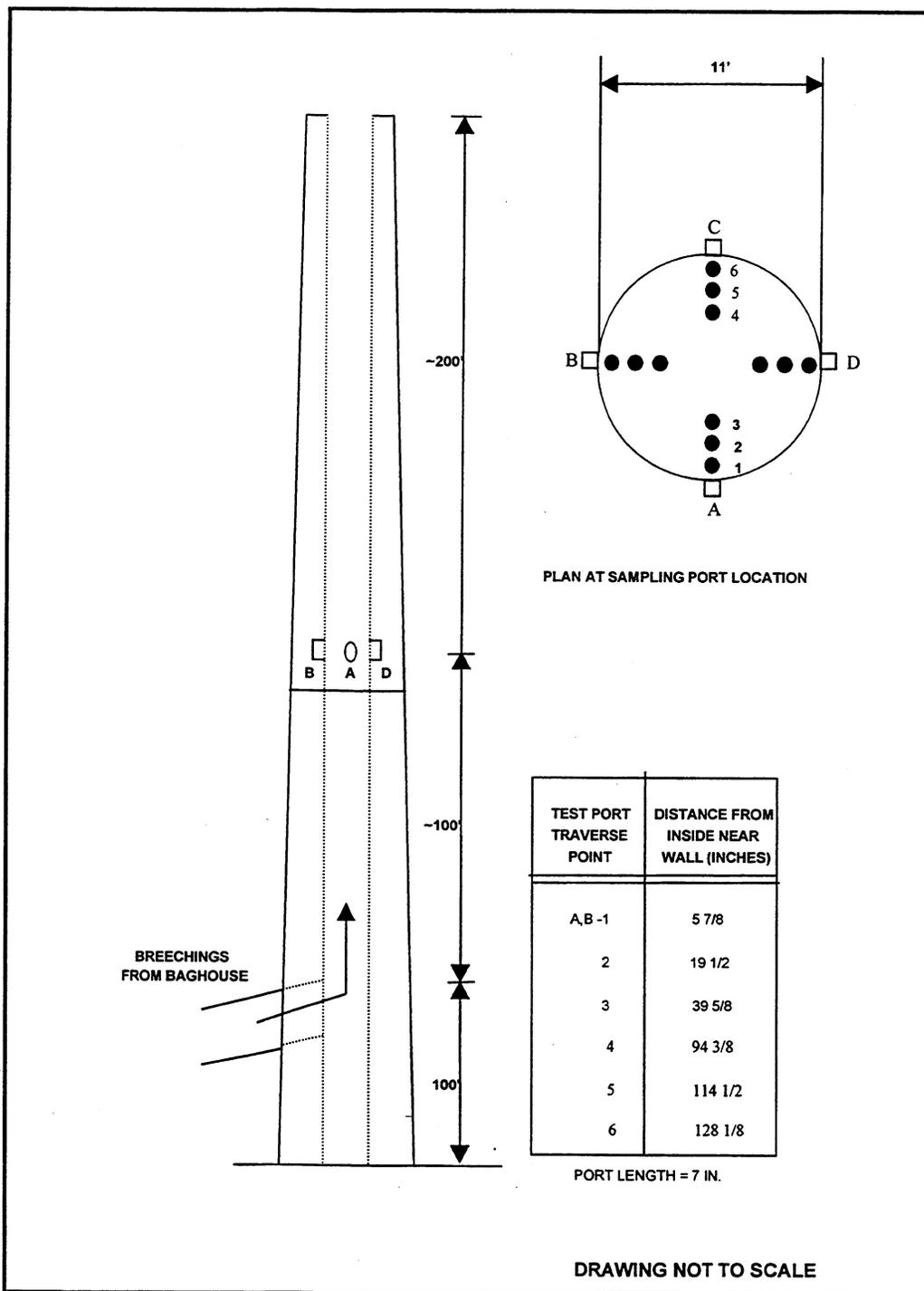


**FIGURE 2-1**  
**UNIT 6 ESP INLET DUCT TEST SITE**  
**PORT AND TRAVERSE POINT LOCATIONS**



**FIGURE 2-2  
UNIT 6 STACK TEST SITE  
PORT AND TRAVERSE POINT LOCATIONS**

DOE1\_03-D20



**FIGURE 2-3**  
**UNITS 1-4 STACK TEST SITE**  
**PORT AND TRAVERSE POINT LOCATIONS**

DOE1\_03-D20

### **3. SUMMARY AND DISCUSSION OF TEST RESULTS**

#### **3.1 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, 3-3 and 3-4 for the coal, 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.
- 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.

#### **3.2 PRESENTATION OF RESULTS**

##### **3.2.1 Mercury Speciation Test Results**

A summary of the Ontario Hydro method mercury speciation test results are presented on Tables 3-5, 3-6, and 3-7 for Unit 6 and Units 1-4, respectively.

Table 3-5 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 3-6 and 3-7 presents the mercury concentrations and mass rate values for particulate, oxidized, elemental and total mercury for each individual test runs along with the measured

**Table 3-1**  
**Sampling/Testing, Analytical, and QC Plan**  
**Unit No. 6 and Units 1-4 Coal**

No. of Test Runs: 3 composites on Unit 6; 1 composite on Units 1-4  
 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
<b>Sampling or Monitoring Design:</b>							
Total No. of Samples	3 for Unit 6; 1 for Units 1-4	3 for Unit 6; 1 for Units 1-4	3 for Unit 6; 1 for Units 1-4	3 for Unit 6; 1 for Units 1-4	3 for Unit 6; 1 for Units 1-4	3 for Unit 6; 1 for Units 1-4	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 <sup>2</sup>	0	0	0	0	0	0	NA
Replicates <sup>3</sup>	All samples	All samples	All samples	All samples	All samples	All Samples	NA
QC Spikes <sup>4</sup>	1/batch <sup>1</sup>	1/batch	1/batch	1/batch	1/batch	1/batch	NA
Approximate No. of Samples Analyzed	7 for Unit 6; 3 for Units 1-4	7 for Unit 6; 3 for Units 1-4	7 for Unit 6; 3 for Units 1-4	7 for Unit 6; 3 for Units 1-4	7 for Unit 6; 3 for Units 1-4	7 for Unit 6; 3 for Units 1-4	NA
Analytical Laboratory:	CONSOL	CONSOL	CONSOL	CONSOL	CONSOL	CONSOL	NA

Notes: <sup>1</sup>A batch consists of a maximum of 20 samples.

<sup>2</sup>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 sample batch.

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

<sup>4</sup>A 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  
Unit 6 ESP Ash and Units 1-4 Baghouse 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:				NA
Total No. of Samples	6 for Unit 6 ; 3 for Units 1-4	6 for Unit 6 ; 3 for Units 1-4	6 for Unit 6 ; 3 for Units 1-4	NA
Site Blanks	0	0	0	NA
Trip Blanks	0	0	0	NA
Lab Blanks	0	0	0	NA
Blank Spikes <sup>2</sup>	0	0	0	NA
Replicates <sup>3</sup>	All samples	All samples	All Samples	NA
QC Spikes <sup>4</sup>	1/batch <sup>1</sup>	1/batch	1/batch	NA
Approximate No. of Samples Analyzed	13 for Unit 6; 7 for Units 1-4	13 for Unit 6; 7 for Units 1-4	13 for Unit 6; 7 for Units 1-4	NA
Analytical Laboratory:	CONSOL	CONSOL	CONSOL	NA

Notes: <sup>1</sup>A batch consists of a maximum of 20 samples.  
<sup>2</sup>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 sample batch.  
<sup>3</sup>This indicates that a duplicate analysis is made on one or more samples as a QC mechanism to measure analytical precision.  
<sup>4</sup>A 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  
Unit 6 – ESP Inlet and Outlet

No. of Test Runs: 3  
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 <sup>(1)</sup>
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 <sup>3</sup> (2)	NA
Total No. of Samples	3 at inlet and outlet	3 at outlet of ESP
Site/Reagent Blanks	Minimum of 1 per sample type	NA
Train Blanks	1 per test location (total of 2)	NA
Lab Blanks	1 per batch <sup>3</sup>	NA
Blank Spike Audit <sup>4</sup>	1 per day	NA
Blank Spikes <sup>5</sup>	1 per batch	NA
Replicates <sup>6</sup>	1 per batch	NA
QC spikes <sup>7</sup>	1 per batch	NA
Approximate No. of Samples Analyzed <sup>8</sup>	~70	3
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 know 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 (SO<sub>2</sub>), oxides of nitrogen (NO<sub>x</sub>), carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>) and opacity on the ESP outlet (stack) of Unit 6.

Table 3-4

Sampling/Testing, Analytical, and QC Plan  
Units 1-4 Baghouse Outlet

No. of Test Runs: 3.  
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 <sup>(1)</sup>
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 <sup>3</sup>	NA
Total No. of Samples	3 at outlet	3 at outlet
Site/Reagent Blanks	Minimum of 1 per sample type	NA
Train Blanks	1 per test location	NA
Lab Blanks	1 per batch <sup>2</sup>	NA
Blank Spike Audit <sup>3</sup>	1 per day	NA
Blank Spikes <sup>4</sup>	1 per batch	NA
Replicates <sup>5</sup>	1 per batch	NA
QC spikes <sup>6</sup>	1 per batch	NA
Approximate No. of Samples Analyzed <sup>7</sup>	~40	3
Analytical Laboratory:	EERC	NA

- 1 Mercury CEMS at ESP Outlet (stack only)
- 2 A batch consists of a maximum of 10 samples.
- 3 A blank spike audit sample is independently prepared and unknown to the analyst.
- 4 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
- 5 This indicates that a duplicate or triplicate analysis is made on one or more samples as a QC mechanism to measure analytical precision.
- 6 A sample of similar matrix is spiked with a known amount of the analyte(s) of interest to determine percent recovery.
- 7 Approximate number of total samples and individual fractions, duplicates and other QC samples.

Note: The facility CEMS measured sulfur dioxide (SO<sub>2</sub>), oxides of nitrogen (NO<sub>x</sub>), carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>) and opacity on the outlet (stack) of Units 1-4.

**TABLE 3-5**  
**COMPARISON OF MERCURY SPECIATION TO TOTAL MERCURY RESULTS**  
**UNIT NO. 6 AND UNITS 1-4**

Mercury Species	Unit 6 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	2.44	77.37	2.66	75.41	2.61	79.03	77.27
Oxidized Mercury Emissions	0.56	17.82	0.65	18.31	0.54	16.32	17.48
Elemental Mercury Emissions	0.15	4.81	0.22	6.28	0.15	4.65	5.25
<b>Total Mercury Emissions</b>	<b>3.16</b>	<b>100.00</b>	<b>3.53</b>	<b>100.00</b>	<b>3.30</b>	<b>100.00</b>	

Mercury Species	Unit 6 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	0.05	3.69	0.022	1.33	< 0.004	0.00	1.67
Oxidized Mercury Emissions	0.70	52.77	0.85	51.42	0.62	47.42	50.54
Elemental Mercury Emissions	0.58	43.54	0.78	47.25	0.68	52.58	47.79
<b>Total Mercury Emissions</b>	<b>1.32</b>	<b>100.00</b>	<b>1.66</b>	<b>100.00</b>	<b>1.30</b>	<b>100.00</b>	

Mercury Species	UnitS 1-4 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	< 0.004	0.00	< 0.005	0.00	< 0.005	0.00	0.00
Oxidized Mercury Emissions	0.25	85.09	0.17	62.65	0.19	94.20	80.65
Elemental Mercury Emissions	0.04	14.91	0.10	37.35	0.01	5.80	19.35
<b>Total Mercury Emissions</b>	<b>0.29</b>	<b>100.00</b>	<b>0.27</b>	<b>100.00</b>	<b>0.20</b>	<b>100.00</b>	

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 3-6  
SUMMARY OF MERCURY SPECIATION TEST RESULTS  
UNIT NO. 6

TEST DATA:	1		2		3		1		2		3		STDEV	%RSD
	7/14/99	1415-1640	7/15/99	0825-1050	7/15/99	1250-1510	7/14/99	1415-1641	7/15/99	0825-1055	7/15/99	1250-1512		
Unit Load, MW	74.0	75.0	307	307	308	308	74.0	74.0	75.0	75.0	75.0	75.0	74.7	0.8
Coal feed rate, lb/hr.	63,700	64,200	39.1	39.1	39.8	39.8	63,700	63,700	64,200	64,200	64,000	64,000	63,967	0.4
Coal Btu content, Btu/lb. (as received)	12,038	12,132	356900	356900	363200	363200	12,038	12,038	12,132	12,132	12,121	12,121	12,097	0.4
Heat Input, 10 <sup>6</sup> Btu/hr. (F-Factor)	761	818	214200	214200	218200	218200	761	761	818	818	749	749	776	4.7
<b>PROCESS DATA:</b>														
Avg. temperature, deg. F	309	307	307	307	308	308	305	305	304	304	306	306	305	0.3
Avg. gas stream velocity, ft./sec.	40.4	39.1	39.1	39.1	39.8	39.8	75.6	75.6	76.6	76.6	74.6	74.6	76.2	2.7
Avg. gas stream volumetric flow, waf/min. (as measured)	368100	356900	356900	356900	363200	363200	288,400	288,400	299,900	299,900	284,800	284,800	291,000	2.7
Avg. gas stream volumetric flow, dsc/min. (as measured) <sup>(1)</sup>	218900	214200	214200	214200	218200	218200	176,500	176,500	184,000	184,000	174,800	174,800	178,400	2.7
Avg. gas stream outlet volumetric flow, dsc/min. (adjusted) <sup>(1), (2)</sup>	163300	173600	173600	173600	167000	167000	168,000	168,000	168,000	168,000	168,000	168,000	168,000	3.1
<b>GAS STREAM VELOCITY AND VOLUMETRIC FLOW DATA:</b>														
Conc., ug/m <sup>3</sup>	2.44	2.66	2.66	2.66	2.61	2.61	2.57	2.57	2.57	2.57	2.57	2.57	2.57	4.4
Conc., ug/Nm <sup>3(2)</sup>	2.62	2.85	2.85	2.85	2.79	2.79	2.76	2.76	2.76	2.76	2.76	2.76	2.76	4.4
Emission rate, lbs/10 <sup>2</sup> Btu. <sup>(4)</sup>	1.96	2.11	2.11	2.11	2.18	2.18	2.08	2.08	2.08	2.08	2.08	2.08	2.08	5.2
Emission rate, lbs/hr. <sup>(4)</sup>	1.50E-03	1.73E-03	1.73E-03	1.73E-03	1.63E-03	1.63E-03	1.62E-03	1.62E-03	1.62E-03	1.62E-03	1.62E-03	1.62E-03	1.62E-03	7.3
<b>PARTICULATE BOUND MERCURY EMISSIONS:</b>														
Conc., ug/m <sup>3</sup>	0.56	0.65	0.65	0.65	0.54	0.54	0.58	0.58	0.58	0.58	0.58	0.58	0.58	9.7
Conc., ug/Nm <sup>3(2)</sup>	0.60	0.69	0.69	0.69	0.58	0.58	0.62	0.62	0.62	0.62	0.62	0.62	0.62	9.7
Emission rate, lbs/10 <sup>2</sup> Btu. <sup>(4)</sup>	0.45	0.51	0.51	0.51	0.45	0.45	0.47	0.47	0.47	0.47	0.47	0.47	0.47	7.7
Emission rate, lbs/hr. <sup>(4)</sup>	3.44E-04	4.20E-04	4.20E-04	4.20E-04	3.36E-04	3.36E-04	3.67E-04	3.67E-04	3.67E-04	3.67E-04	3.67E-04	3.67E-04	3.67E-04	12.6
<b>OXIDIZED MERCURY EMISSIONS:</b>														
Conc., ug/m <sup>3</sup>	0.15	0.22	0.22	0.22	0.15	0.15	0.18	0.18	0.18	0.18	0.18	0.18	0.18	22.7
Conc., ug/Nm <sup>3(2)</sup>	0.16	0.24	0.24	0.24	0.16	0.16	0.19	0.19	0.19	0.19	0.19	0.19	0.19	22.7
Emission rate, lbs/10 <sup>2</sup> Btu. <sup>(4)</sup>	0.12	0.18	0.18	0.18	0.13	0.13	0.14	0.14	0.14	0.14	0.14	0.14	0.14	20.8
Emission rate, lbs/hr. <sup>(4)</sup>	9.30E-05	1.44E-04	1.44E-04	1.44E-04	9.59E-05	9.59E-05	1.11E-04	1.11E-04	1.11E-04	1.11E-04	1.11E-04	1.11E-04	1.11E-04	25.9
<b>ELEMENTAL MERCURY EMISSIONS:</b>														
Conc., ug/m <sup>3</sup>	3.16	3.53	3.53	3.53	3.30	3.30	3.33	3.33	3.33	3.33	3.33	3.33	3.33	5.6
Conc., ug/Nm <sup>3(2)</sup>	3.39	3.79	3.79	3.79	3.54	3.54	3.57	3.57	3.57	3.57	3.57	3.57	3.57	5.6
Emission rate, lbs/10 <sup>2</sup> Btu. <sup>(4)</sup>	2.54	2.80	2.80	2.80	2.75	2.75	2.70	2.70	2.70	2.70	2.70	2.70	2.70	5.2
Emission rate, lbs/hr. <sup>(4)</sup>	1.93E-03	2.29E-03	2.29E-03	2.29E-03	2.06E-03	2.06E-03	2.10E-03	2.10E-03	2.10E-03	2.10E-03	2.10E-03	2.10E-03	2.10E-03	8.7
<b>TOTAL MERCURY EMISSIONS:</b>														
Conc., ug/m <sup>3</sup>	3.81	4.35	4.35	4.35	4.03	4.03	4.06	4.06	4.06	4.06	4.06	4.06	4.06	6.3
Conc., ug/Nm <sup>3(2)</sup>	4.07	4.57	4.57	4.57	4.29	4.29	4.32	4.32	4.32	4.32	4.32	4.32	4.32	6.3
Emission rate, lbs/10 <sup>2</sup> Btu. <sup>(4)</sup>	3.17	3.59	3.59	3.59	3.33	3.33	3.37	3.37	3.37	3.37	3.37	3.37	3.37	10.9
Emission rate, lbs/hr. <sup>(4)</sup>	2.42E-03	2.81E-03	2.81E-03	2.81E-03	2.56E-03	2.56E-03	2.61E-03	2.61E-03	2.61E-03	2.61E-03	2.61E-03	2.61E-03	2.61E-03	10.9
<b>TOTAL MERCURY REMOVAL EFFICIENCY:</b>														
Removal efficiency, %	54.73%	50.13%	50.13%	50.13%	58.60%	58.60%	54.49%	54.49%	54.49%	54.49%	54.49%	54.49%	54.49%	4.24%

(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 6 outlet corrected for O<sub>2</sub> measured at the inlet location.  
 (4) Emission rates at the ESP inlet are based on the adjusted volumetric flow.  
 (5) Non-detects not included in total mercury catch or in average emission rates.

**TABLE 3-7  
SUMMARY OF MERCURY SPECIATION TEST RESULTS  
UNIT 1-4 OUTLET**

TEST DATA:		1	2	3		
Test run number			Units 1-4 Outlet			
Location			7/16/99	7/16/99		
Test date			1115-1338	1410-1641		
Test time period						
<b>PROCESS DATA:</b>						
Unit Load, MW		124	127	138		
Coal feed rate, lb/hr.		110,000	111,000	117,000		
Coal Btu content, Btu/lb. (as received)		12,200	12,200	12,200		
Heat Input, 10 <sup>6</sup> Btu/hr (F-Factor)		1784	1638	1755		
<b>GAS STREAM VELOCITY AND VOLUMETRIC FLOW DATA:</b>						
Avg. temperature, deg. F		285	299	310		
Avg. gas stream velocity, ft./sec.		111.9	107.1	114.9		
Avg. gas stream volumetric flow, wacf/min.		657,800	629,600	675,300		
Avg. gas stream volumetric flow, dscf/min. <sup>(1)</sup>		417,300	393,900	413,200		
<b>PARTICULATE BOUND MERCURY EMISSIONS:</b>						
Conc., ug/m <sup>3</sup>	<	0.004	<	0.005	<	0.005
Conc., ug/Nm <sup>3</sup> <sup>(2)</sup>	<	0.004	<	0.006	<	0.005
Emission rate, lbs/10 <sup>12</sup> Btu.	<	3.60E-03	<	4.63E-03	<	4.14E-03
Emission rate, lbs/hr	<	6.42E-06	<	7.58E-06	<	7.27E-06
<b>OXIDIZED MERCURY EMISSIONS:</b>						
Conc., ug/m <sup>3</sup>		0.25	0.17	0.19		
Conc., ug/Nm <sup>3</sup> <sup>(2)</sup>		0.27	0.18	0.20		
Emission rate, lbs/10 <sup>12</sup> Btu.		0.22	0.15	0.17		
Emission rate, lbs/hr		3.89E-04	2.46E-04	2.95E-04		
<b>ELEMENTAL MERCURY EMISSIONS:</b>						
Conc., ug/m <sup>3</sup>		0.04	0.10	0.01		
Conc., ug/Nm <sup>3</sup> <sup>(2)</sup>		0.05	0.11	0.01		
Emission rate, lbs/10 <sup>12</sup> Btu.		0.04	0.09	0.01		
Emission rate, lbs/hr		6.82E-05	1.47E-04	1.82E-05		
<b>TOTAL MERCURY EMISSIONS: <sup>(3)</sup></b>						
Conc., ug/m <sup>3</sup>		0.29	0.27	0.20		
Conc., ug/Nm <sup>3</sup> <sup>(2)</sup>		0.31	0.29	0.22		
Emission rate, lbs/10 <sup>12</sup> Btu.		0.26	0.24	0.18		
Emission rate, lbs/hr		4.58E-04	3.93E-04	3.13E-04		
					AVERAGE	%RSD
					130	5.7
					112,700	3.36
					12,200	0.00
					1726	4.48
					298.1	4.18
					111.3	3.53
					654,200	3.52
					408,100	3.06
					0.005	11.11
					0.005	11.11
					4.12E-03	12.48
					7.09E-06	8.45
					0.20	20.88
					0.22	20.88
					0.18	19.65
					3.10E-04	23.41
					0.05	86.06
					0.06	86.06
					0.05	87.30
					7.78E-05	83.44
					0.25	18.28
					0.27	18.28
					2.25E-01	18.25
					3.88E-04	18.61

(1) Standard conditions = 68 deg. F. (20 deg. C.) and 29.92 inches Hg (760mm Hg).  
 (2) Nm<sup>3</sup> = Normal cubic meter ( 32 deg. F. (0 deg. C.) and 29.92 inches Hg (760mm Hg)).  
 (3) Non-detects not included in total mercury catch or in average emission rates.

volumetric flow rates. Average values with the standard deviation (SDEV) and percent relative standard deviation (% RSD) have been calculated and are presented.

### **3.2.1.1 Unit 6**

For Unit 6 ESP Inlet an average of 77% of the total mercury measured is particulate bound mercury. On average the oxidized mercury was 18 percent of the total and the elemental mercury was approximately 5 percent of the total mercury collected. At the Unit 6 ESP outlet, oxidized mercury comprised the highest of the total at 50 percent. The elemental mercury was 48 percent of the total and the particulate bound mercury was less than two percent.

Based on the total mercury measurements the average removal efficiency for the ESP was 55 percent with an average mass emission rate of 0.00096 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 elevated carbon content (average of 17%) of the ash.

The average total mercury emission rates for Unit 6 are 1.4 ug/m<sup>3</sup>, 1.2 lbs/10<sup>12</sup> Btu and 0.00096 lb/hr.

Detailed test data and test results for the Unit 6 ESP inlet and outlet are provided on Tables A-1 and A-2 in Appendix A.

### **3.2.1.2 Units 1-4**

At the Units 1-4 stack location no particulate bound mercury was detected. On average the oxidized mercury was 81 percent of the total mercury with the balance of 19 percent attributed to elemental mercury.

The average total mercury emissions for Units 1-4 are 0.25 ug/m<sup>3</sup>, 0.22 lbs/10<sup>12</sup> Btu and 0.00039 lb/hr.

Detailed test data and test results for the Units 1-4 stack are provided on Table A-3 in Appendix A.

### 3.2.2 Mercury CEMS Comparison Results

A comparison of the gas phase mercury concentrations measured in  $\mu\text{g}/\text{m}^3$  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 Unit 6 and Units 1-4. 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  $100^\circ\text{F}$ . 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 ( $\text{Hg}^{2+}$ ) to elemental ( $\text{Hg}^0$ ), 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 Unit 6 stack the results showed that the two CEMs gave excellent results for both total mercury and  $\text{Hg}^0$ . The PS Analytical CEM gave more consistent results than the Semtech. The vapor-phase mercury concentration at the stack of Units 1-4 was very low ( $< 1 \mu\text{g}/\text{m}^3$ ). Both the Semtech and PS Analytical CEMS gave almost exactly the same results as the Ontario Hydro method. The PS Analytical CEM did appear to provide more consistent data with less noise than the Semtech. At this test location, no comparison was made for the elemental concentration because the vapor-phase mercury concentration was very low ( $0.05 \mu\text{g}/\text{m}^3$ ). The statistical results for the CEMS compared to the Ontario Hydro method for all tests are shown in Table 3-8.

Figure 3-1  
 Comparison of Mercury CEMS to Ontario Hydro Results  
 Unit 6 Stack

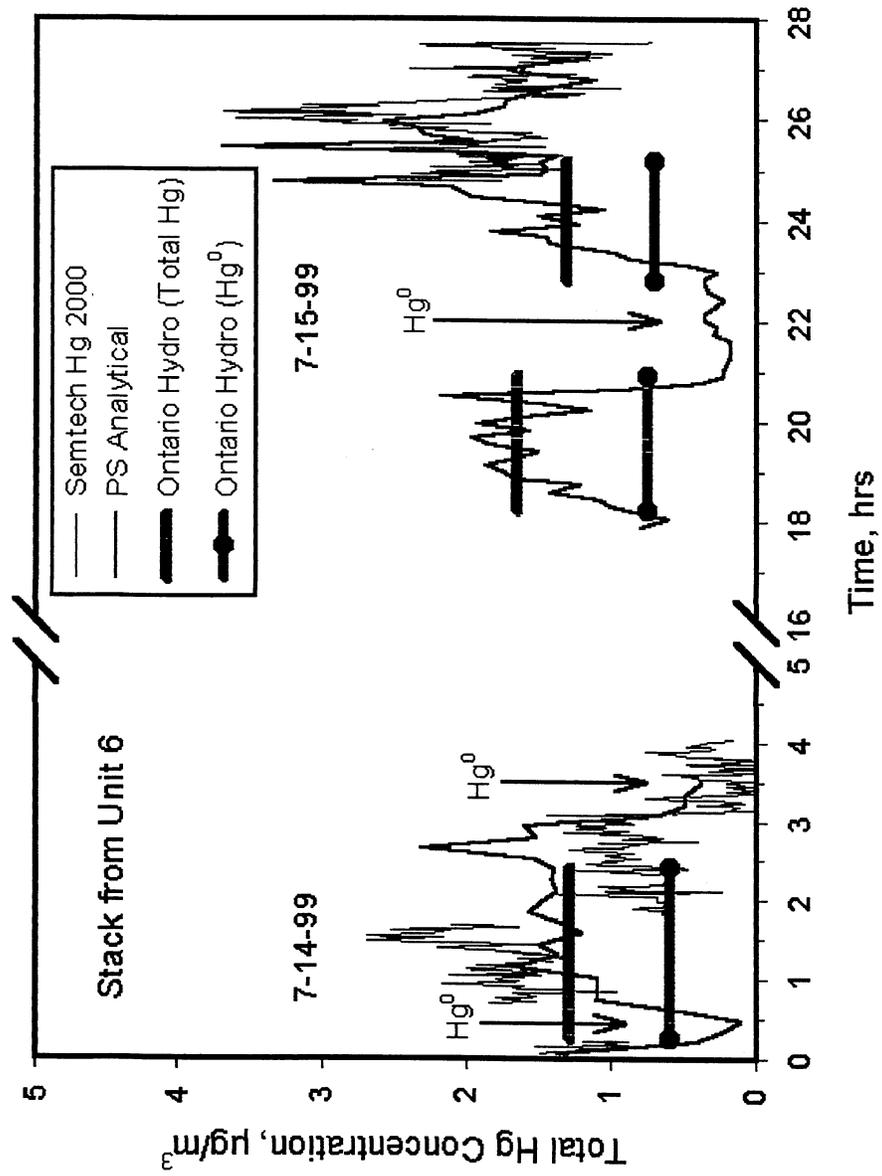
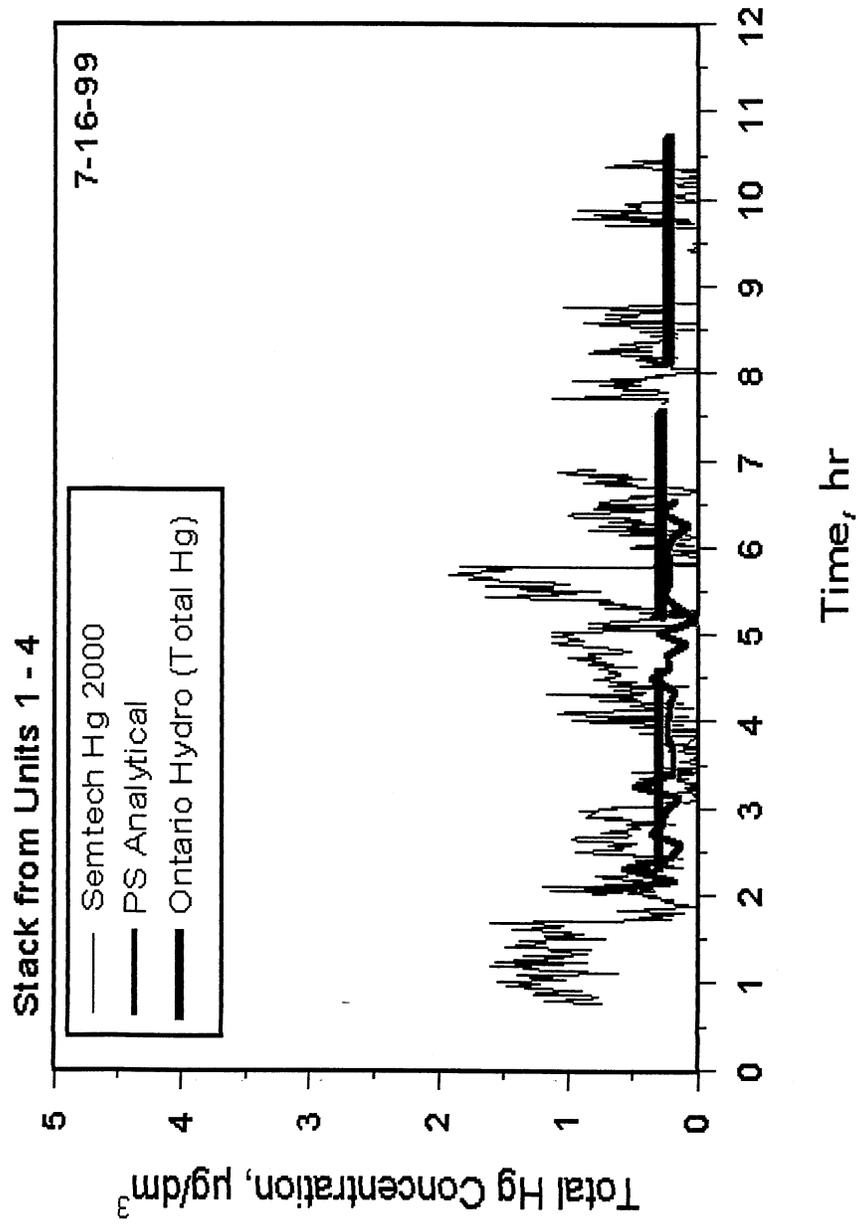


Figure 3-2  
 Comparison of Mercury CEMS to Ontario Hydro Results  
 Units 1-4 Stacks



**TABLE 3-8**

**SUMMARY OF MERCURY CEM RESULTS FOR UNIT 6 AND UNITS 1-4**

Test Location	Average Measured Values			
Unit 6 Stack	Hg <sup>0</sup> µg/m <sup>3</sup>	SDEV µg/m <sup>3</sup>	Total Hg µg/m <sup>3</sup>	SDEV µg/m <sup>3</sup>
PS Analytical	0.32	0.13	1.55	0.52
Semtech Hg 2000	0.27	0.21	1.64	0.65
Ontario Hydro Method	0.68	0.04	1.43	0.09
<b>Units 1-4 Stack</b>				
PS Analytical	---	---	0.24	0.14
Semtech Hg 2000	---	---	0.50	0.44
Ontario Hydro Method	0.05	0.05	0.25	0.44

### **3.2.3 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 are contained in this section.

#### **3.2.3.1 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 speciation train typically results in Hg-in-solution concentrations of 1 to 10  $\mu\text{g}/\text{L}$ . 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 units sampled. Because flue gas mercury measurements were completed at both the Unit No. 6 ESP inlet and stack locations, material balance closures were calculated at both locations. The material balance for Units 1-4 was based on the stack location only.

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 Unit No. 6 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 6 has a cold-side precipitator. Ash was obtained from both rows of hoppers 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 rows of ESP hoppers.

### **3.2.3.2 Discussion of Mercury Material Balance Results**

#### **Unit No. 6**

The average coal firing rate for Unit No. 6 using the F-factor calculation was 62,385 lb/h. This compares well with the feeder value of 63,967 lb/h. The average Hg concentration of the coal was 0.039 µg/g (ppm). This results in an average Hg mass flow rate of 18.39 mg/min into the

boiler. The average Hg mass flow rate at the ESP inlet as determined from the Ontario Hydro measurements was 15.85 mg/min. The average material balance closure at the ESP inlet for Unit No. 6 was 86.1% with the individual closures of 80.7%, 89.6%, and 88.0%. The mass flow rate of Hg in the flue gas at the ESP outlet was 7.22 mg/min. The Hg mass flow rate in the ESP ash was 6.23 mg/min. The average material balance closure at the ESP outlet for Unit No. 6 was 72.9% with the individual closures of 66.6%, 81.0%, 71.2%. These closures are biased low and could be a results of uncertainty of the coal Hg concentration. However, the balances are reasonable when again considering the low value for Hg in the ESP outlet gas ( $\sim 1.5 \mu\text{g}/\text{m}^3$ ) and the Hg concentration of the coal ( $\sim 0.04$  ppm). The ESP ash hopper samples show that 34% of the Hg released during combustion is captured with the ash.

#### **Units 1-4**

The only significant mercury output streams are the baghouse ash and the stack flue gas. The mass flow rate for the baghouse ash was estimated by assuming that in the boiler, 80% of coal ash partitions as fly ash. By knowing the ash content of the coal and the coal firing rate, the mass flow rate of fly ash into the baghouse can be estimated. This estimate was refined by accounting for the amount of un-burned carbon contained in the ash samples. The mass flow rate of mercury associated with this ash was then calculated from the mercury concentration of the ash. The mass flow rate of mercury in the stack flue gas was determined by a direct measurement using the Ontario Hydro flue gas sampling method.

The average combined coal firing rate of Units 1-4 using the F-factor calculation was 134,052 lb/hr. The average mercury concentration of the coal was  $0.02 \mu\text{g}/\text{g}$  (ppm). This results in an average mercury mass flow rate of 20.26 mg/min into the boiler. The mass flow rate of mercury in the flue gas at the stack location (baghouse outlet) was 2.92 mg/min or 15% of the total mercury in the coal. The mercury mass flow rate in the baghouse ash was 14.90 mg/min. The average material balance closure at the stack location was 88.0% with individual closures of 90.2%, 92.0% and 81.9%. These closures are excellent when considering the low value of mercury in the flue gas ( $\sim 0.2$  to  $2.0 \mu\text{g}/\text{m}^3$ ) and the uncertainty in the mercury-in-coal determination. The baghouse ash hopper samples show that 70% to 76% of the mercury released during combustion is captured with the ash.

### **3.2.3.3 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 Unit No. 6 ESP inlet and outlet and Units 1-4 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-4 and A-5 in Appendix A.

### **3.2.4 Process Solid Sample Stream Results**

Tables 3-9 through 3-13 provides a summary of the analytical results obtained on the coal feed and ash samples collected on Unit No. 6 and Units 1-4.

For each parameter measured on the Unit No. 6 coal and ash streams and the Units 1-4 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 single composite sample of the Units 1-4 coal was analyzed for this program.

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

**Table 3-9  
Summary of Process Solid Sample Stream Results  
Unit No. 6 Coal Feed Samples**

**As Determined Basis**

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Date	7/14/99	7/15/99	7/15/99			
Moisture	5.17	5.10	5.10	5.12	0.04	0.8%
Volatile Matter	35.87	36.16	36.10	36.04	0.15	0.4%
Ash	9.75	9.54	9.48	9.59	0.14	1.5%
Carbon	69.13	69.13	70.66	69.64	0.88	1.3%
Hydrogen	4.98	4.97	5.09	5.01	0.07	1.3%
Nitrogen	1.49	1.53	1.53	1.52	0.02	1.5%
Sulfur	0.97	0.99	0.99	0.98	0.01	1.2%
Oxygen	8.51	8.75	7.15	8.14	0.86	10.5%
Chlorine, ppm	190	199	171	187	14	7.6%
Mercury, ppb	25	46	41	37	11	29.3%
Btu/lb	12038	12132	12121	12097	51	0.4%
Total Moisture	5.17	5.10	5.10	5.12	0.04	0.8%

**Dry Basis**

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Volatile Matter	37.83	38.10	38.04	37.99	0.14	0.4%
Ash	10.28	10.05	9.99	10.11	0.15	1.5%
Carbon	72.90	72.84	74.46	73.40	0.91	1.2%
Hydrogen	4.64	4.64	4.76	4.68	0.07	1.5%
Nitrogen	1.57	1.61	1.61	1.60	0.02	1.5%
Sulfur	1.02	1.04	1.04	1.04	0.01	1.1%
Oxygen	9.04	9.80	8.12	8.99	0.84	9.3%
Chlorine, ppm	200	210	180	197	15	7.6%
Mercury, ppb	26	48	43	39	12	29.2%
Btu/lb	12694	12784	12772	12750	49	0.4%
F Factor, O <sub>2</sub>	9853	9749	10048	9883	151	1.5%
F Factor, CO <sub>2</sub>	1843	1829	1871	1848	21	1.2%

**Major Ash Elements: (% of Coal Ash)**

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
SiO <sub>2</sub>	55.57	55.59	57.17	56.11	0.91	1.6%
Al <sub>2</sub> O <sub>3</sub>	24.78	25.29	25.89	25.32	0.55	2.2%
TiO <sub>2</sub>	0.80	0.82	0.83	0.82	0.02	1.9%
Fe <sub>2</sub> O <sub>3</sub>	5.42	5.10	5.00	5.17	0.22	4.2%
CaO	3.51	3.27	3.31	3.36	0.13	3.8%
MgO	1.78	1.69	1.75	1.74	0.05	2.6%
Na <sub>2</sub> O	2.12	2.08	2.15	2.12	0.03	1.7%
K <sub>2</sub> O	1.05	1.02	1.05	1.04	0.02	1.7%
P <sub>2</sub> O <sub>5</sub>	0.46	0.46	0.46	0.46	0.00	0.0%
SO <sub>3</sub>	3.69	3.20	3.07	3.32	0.33	9.8%
Undetermined	0.82	1.48	-0.68	0.54	1.10	---

Table 3-10

Summary of Process Solid Sample Stream Results  
Unit No. 6 ESP Ash Samples (Front-Row of Hoppers)

As Determined Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Date	7/14/99	7/15/99	7/15/99			
Moisture	0.34	0.32	0.27	0.31	0.04	11.6%
Ash	83.13	81.54	80.95	81.87	1.12	1.4%
Carbon	15.52	17.33	17.60	16.82	1.13	6.7%
Sulfur	0.62	0.65	0.66	0.64	0.02	3.2%
Mercury, ppb	67	69	80	72	7	9.7%

Dry Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Ash	83.41	81.80	81.17	82.13	1.15	1.4%
Carbon	15.57	17.39	17.65	16.87	1.13	6.7%
Sulfur	0.62	0.65	0.66	0.65	0.02	3.2%
Mercury, ppb	67	69	80	72	7	9.6%

Major Ash Elements:

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
SiO <sub>2</sub>	49.67	50.01	48.72	49.47	0.67	1.3%
Al <sub>2</sub> O <sub>3</sub>	21.12	20.67	20.38	20.72	0.37	1.8%
TiO <sub>2</sub>	0.66	0.66	0.66	0.66	0.00	0.0%
Fe <sub>2</sub> O <sub>3</sub>	4.09	4.13	4.09	4.10	0.02	0.6%
CaO	2.56	2.75	2.78	2.70	0.12	4.4%
MgO	1.37	1.38	1.37	1.37	0.01	0.4%
Na <sub>2</sub> O	1.67	1.75	1.72	1.71	0.04	2.3%
K <sub>2</sub> O	0.80	0.81	0.81	0.81	0.01	0.7%
P <sub>2</sub> O <sub>5</sub>	0.35	0.35	0.36	0.35	0.01	1.6%
SO <sub>3</sub>	0.38	0.37	0.37	0.37	0.01	1.5%
Undetermined	17.33	17.12	18.74	17.73	0.88	4.9%

**Table 3-11**

**Summary of Process Solid Sample Stream Results  
Unit No. 6 ESP Ash Samples (Back-Row of Hoppers)**

**As Determined Basis**

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Date	7/14/99	7/15/99	7/15/99			
Moisture	0.41	0.36	0.22	0.33	0.10	29.7%
Ash	79.39	79.83	80.05	79.76	0.33	0.4%
Carbon	18.56	18.20	18.32	18.36	0.18	1.0%
Sulfur	0.86	0.82	0.81	0.83	0.03	3.2%
Mercury, ppb	228	280	264	257	27	10.3%

**Dry Basis**

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Ash	79.72	80.12	80.23	80.02	0.27	0.3%
Carbon	18.64	18.27	18.36	18.42	0.19	1.0%
Sulfur	0.86	0.82	0.81	0.83	0.03	3.3%
Mercury, ppb	229	281	265	258	27	10.3%

Table 3-12

Summary of Process Solid Sample Stream Results  
Unit No. 1-4 Coal Feed Samples

As Determined Basis

Test ID	T-1			AVG	SDEV	PRSD
Date	7/16/99					
Moisture	5.38					
Volatile Matter	34.02					
Ash	9.26					
Carbon	69.93					
Hydrogen	5.11					
Nitrogen	1.48					
Sulfur	0.93					
Oxygen	7.91					
Chlorine, ppm	199					
Mercury, ppb	22					
Btu/lb	12180					
Total Moisture	5.38					

Dry Basis

Test ID	T-1			AVG	SDEV	PRSD
Volatile Matter	35.95					
Ash	9.79					
Carbon	73.90					
Hydrogen	4.76					
Nitrogen	1.56					
Sulfur	0.98					
Oxygen	8.99					
Chlorine, ppm	210					
Mercury, ppb	23					
Btu/lb	12872					
F Factor, O <sub>2</sub>	9870					
F Factor, CO <sub>2</sub>	1843					

Major Ash Elements: (% of Coal Ash)

Test ID	T-1			AVG	SDEV	PRSD
SiO <sub>2</sub>	56.30					
Al <sub>2</sub> O <sub>3</sub>	25.05					
TiO <sub>2</sub>	0.81					
Fe <sub>2</sub> O <sub>3</sub>	4.75					
CaO	3.19					
MgO	1.67					
Na <sub>2</sub> O	2.19					
K <sub>2</sub> O	0.95					
P <sub>2</sub> O <sub>5</sub>	0.47					
SO <sub>3</sub>	2.92					
Undetermined	1.70					

Table 3-13

Summary of Process Solid Sample Stream Results  
Unit No. 1-4 Baghouse Ash Samples

As Determined Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Date	7/16/99	7/16/99	7/16/99			
Moisture	0.35	0.25	0.36	0.32	0.06	18.9%
Ash	69.42	74.40	71.63	71.82	2.49	3.5%
Carbon	28.82	24.13	26.74	26.56	2.34	8.8%
Sulfur	0.91	0.92	0.92	0.92	0.01	0.6%
Mercury, ppb	131	145	129	135	9	6.4%

Dry Basis

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
Ash	69.66	75.58	71.88	72.37	2.98	4.1%
Carbon	28.91	24.19	26.84	26.65	2.36	8.8%
Sulfur	0.92	0.92	0.92	0.92	0.00	0.2%
Mercury, ppb	131	145	129	135	9	6.4%

Major Ash Elements:

Test ID	T-1	T-2	T-3	AVG	SDEV	PRSD
SiO <sub>2</sub>	43.21	46.85	46.07	45.38	1.91	4.2%
Al <sub>2</sub> O <sub>3</sub>	15.55	16.64	15.25	15.81	0.73	4.6%
TiO <sub>2</sub>	0.44	0.49	0.41	0.45	0.04	9.0%
Fe <sub>2</sub> O <sub>3</sub>	4.35	5.15	5.15	4.88	0.46	9.4%
CaO	2.41	2.56	2.44	2.47	0.08	3.2%
MgO	1.17	1.33	1.25	1.25	0.08	6.4%
Na <sub>2</sub> O	1.38	1.51	1.39	1.43	0.07	5.1%
K <sub>2</sub> O	0.74	0.79	0.76	0.76	0.03	3.3%
P <sub>2</sub> O <sub>5</sub>	0.25	0.28	0.24	0.26	0.02	8.1%
SO <sub>3</sub>	0.40	0.42	0.34	0.39	0.04	10.7%
Undetermined	30.10	23.98	26.70	26.93	3.05	11.3%

### **3.2.5 Unit Operation and Key Operational Parameters**

This section describes the Unit 6 and Units 1-4 operations during the test program and provides the key operating parameters that were monitored and documented during testing.

#### **3.2.5.1 Unit Operation During Testing**

Operation of Unit 6 and Units 1-4 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.

#### **3.2.5.2 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 Unit 1-4 baghouse temperature and pressure drops were recorded once per hour alternating between the A and B baghouse. The facilities CEMS data acquisition system provided concentration values at 1-minute intervals.

A summary of the key operating data is provided in Tables 3-14 and 3-15 for Unit 6 and Units 1-4. The boiler operating data provided on Table 3-15 is for Units 3 and 4 only. No boiler operation data were recorded for Units 1 and 2 during the test periods. The total gross generation provided on Table 3-15 is for all four units, the CEMS data was obtained on the common stack which serves all four units. The total coal feed rate was calculated based on gravimetric feeder readings obtained on all four units during a 24 hour period that bracketed the test periods. A summary of the individual gross generation for each of the four units is provided in Appendix B. All additional process, ESP and baghouse operations data and CEMS data are provided in Appendix B.

**Table 3-14**  
**Summary of Key Process Control Data**  
**Unit No. 6**

Parameter	Units	Run No.		
		1	2	3
Gross Generation	MWH	74.0	75.0	75.0
Station Service	MWH	4.7	5.6	5.8
Net Generation	MWH	69.3	69.4	69.2
Coal Total	lbs/hr	63,700	64,150	64,300
Lbs. Coal/Net KWH	lbs	0.92	0.93	0.93
Lbs. Steam/Net KWH	lbs	6.5	6.7	6.8
Control Valve Position	%	88	87.5	88
Main Steam Flow	lbs/hr	446,700	467,500	473,300
Feed Water Flow	lbs/hr	469,700	485,000	480,000
Feed Water Pressure	psig	1530	1610	1590
First Stage Pressure	psig	980	975	980
Cold Reheat Pressure	psig	346	350	349
Hot Reheat Pressure	psig	323	330	330
Feed Water	Loading %/Temp °F	46% / 397	46% / 398	47% / 398
Main Steam Temp.	°F	990	1000	1000
Cold Reheat Temp.	°F	642	648	637
Hot Reheat Temp.	°F	963	980	987
Superheat Spray Flow	lbs/hr	0	0	0
Reheat Spray Flow	lbs/hr	0	0	0
Air Flow	acfm	515,000	520,000	518,000
Excess Oxygen	%	3.1	3.2	3.1
Inlet Air Temp.	°F	131	130	130
Gas Outlet Temp.	°F	281	280	280
Stack Opacity	%	14	12	14
Stack CEMs (SO <sub>2</sub> )	ppm/v	530	546	539
Stack CEMs (NO <sub>x</sub> )	ppm/v	447	453	449
Stack CEM (CO)	ppm/v	2.3	2.8	4.1
Stack CEM (CO <sub>2</sub> )	%	11.7	11.8	11.8
Induced Draft Fan	(Loading %/Amps)	63% / 156	64% / 160	64% / 160
Forced Draft Fan	(Loading %/Amps)	62% / 85	75% / 90	87% / 90

**Table 3-15  
Summary of Key Process Control Data  
Units 1-4**

Parameter	Units	Run No.					
		Unit Designation					
		1		2		3	
		3	4	3	4	3	4
Total Gross Generation (All Units)	MWH	124		127		138	
Gross Generation	MWH	48.6	49.0	50.0	48.9	48.4	47.8
Station Service	MWH	3.3	3.0	3.0	3.0	3.0	3.0
Net Generation	MWH	45.2	46.0	47.0	45.9	45.4	44.8
Coal Total (units 3 and 4)	lbs/hr	43,200	50,800	48,000	44,000	39,600	45,200
Lbs. Coal/Net KWH	lbs	0.95	1.11	0.97	0.96	0.87	1.01
Coal Total (all units) <sup>1</sup>	lb/hr	110,000		111,000		117,000	
Lbs. Steam/Net KWH	lbs	6.6	8.2	6.6	6.9	5.7	7.5
Main Steam Flow	lbs/hr	297,000	378,000	310,000	320,000	257,000	338,000
Feed Water Flow	lbs/hr	387,000	423,000	405,000	360,000	338,000	374,000
Feed Water Pressure	psig	1810	1850	1800	1850	1800	1850
First Stage Pressure	psig	825	900	825	900	825	900
Cold Reheat Pressure	psig	349	339	349	339	349	335
Hot Reheat Pressure	psig	315	305	315	305	315	305
Feed Water	Loading %/Temp °F	50/438	51/442	50/439	51/442	50/439	51/492
Main Steam Temp.	°F	955	974	957	975	951	975
Cold Reheat Temp.	°F	675	685	675	685	675	685
Hot Reheat Temp.	°F	1010	1009	1009	1009	1009	1009
Superheat Spray Flow	lbs/hr	0	0	0	0	0	0
Reheat Spray Flow	lbs/hr	0	0	0	0	0	0
Air Flow	acfm	360,000	390,000	360,000	390,000	360,000	390,000
Excess Oxygen	%	3.3	3.5	3.4	3.5	3.4	3.5
Stack Opacity	%	1		1		1	
Stack CEMs (SO <sub>2</sub> )	ppm/v	501		514		549	
Stack CEMs (NO <sub>x</sub> )	ppm/v	304		313		361	
Stack CEM (CO)	ppm/v	28		19		11	
Stack CEM (CO <sub>2</sub> )	%	11.4		11.7		12.2	
Induced Draft Fan	Amps	239	270	236	271	249	271
Forced Draft Fan	(Loading %/Amps)	84/64	84/67	84/64	84/67	84/64	84/67
ND = Not Determined (data not available).							

Notes: Boiler data not available for Units 1 and 2; CEMS data measured on common stack of all four units.

<sup>1</sup> Calculated from gravimetric feeder readings for 24 hour period between midnight 7-15-99 to midnight 7-16-99.

## 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 all test locations.
- 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 (HNO<sub>3</sub>) and 10% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). The fifth, sixth and seventh impingers each contained 100 ml of 4% potassium permanganate (KMnO<sub>4</sub>) and 10% sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). 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.

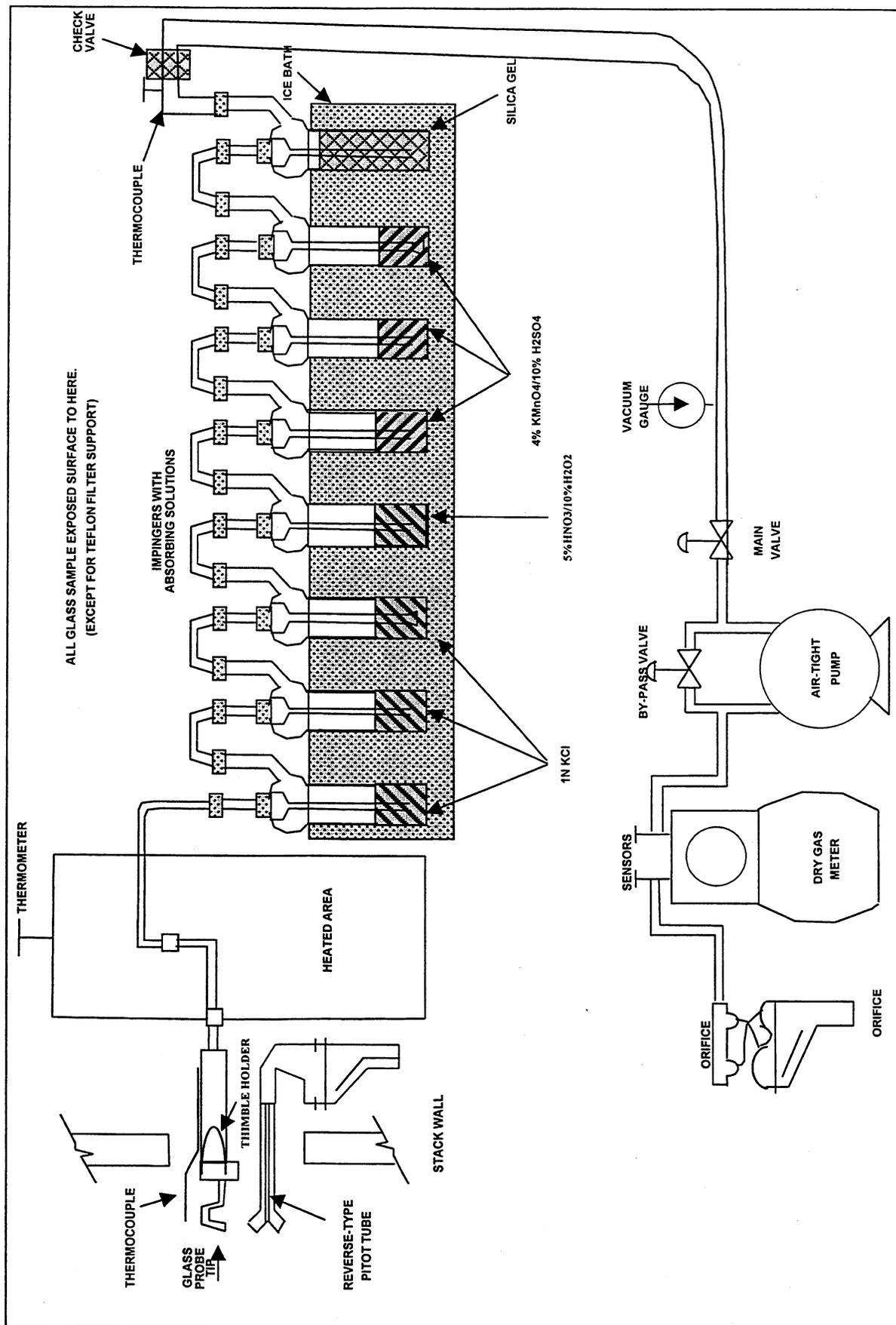


FIGURE 4 - 1  
ONTARIO HYDRO SAMPLING TRAIN

## 4.2 CO<sub>2</sub> AND O<sub>2</sub> SAMPLING EQUIPMENT

The fixed gases sampling train (Figure 4-2) used at the Unit No. 6 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.

For Unit No. 6, the CO<sub>2</sub> and O<sub>2</sub> 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 CO<sub>2</sub> and O<sub>2</sub> gas standards with nitrogen used as the zero gas.

The CO<sub>2</sub> and O<sub>2</sub> concentrations of the Units 1-4 flue gas were measured using an ECOM® portable gas analyzer.

## 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 Hg<sup>0</sup> in fossil fuel combustion flue gas on a continuous or semi-continuous basis and can be equipped with converters for reducing Hg<sup>2+</sup> forms to Hg<sup>0</sup> to determine total mercury; the Hg<sup>2+</sup> 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.

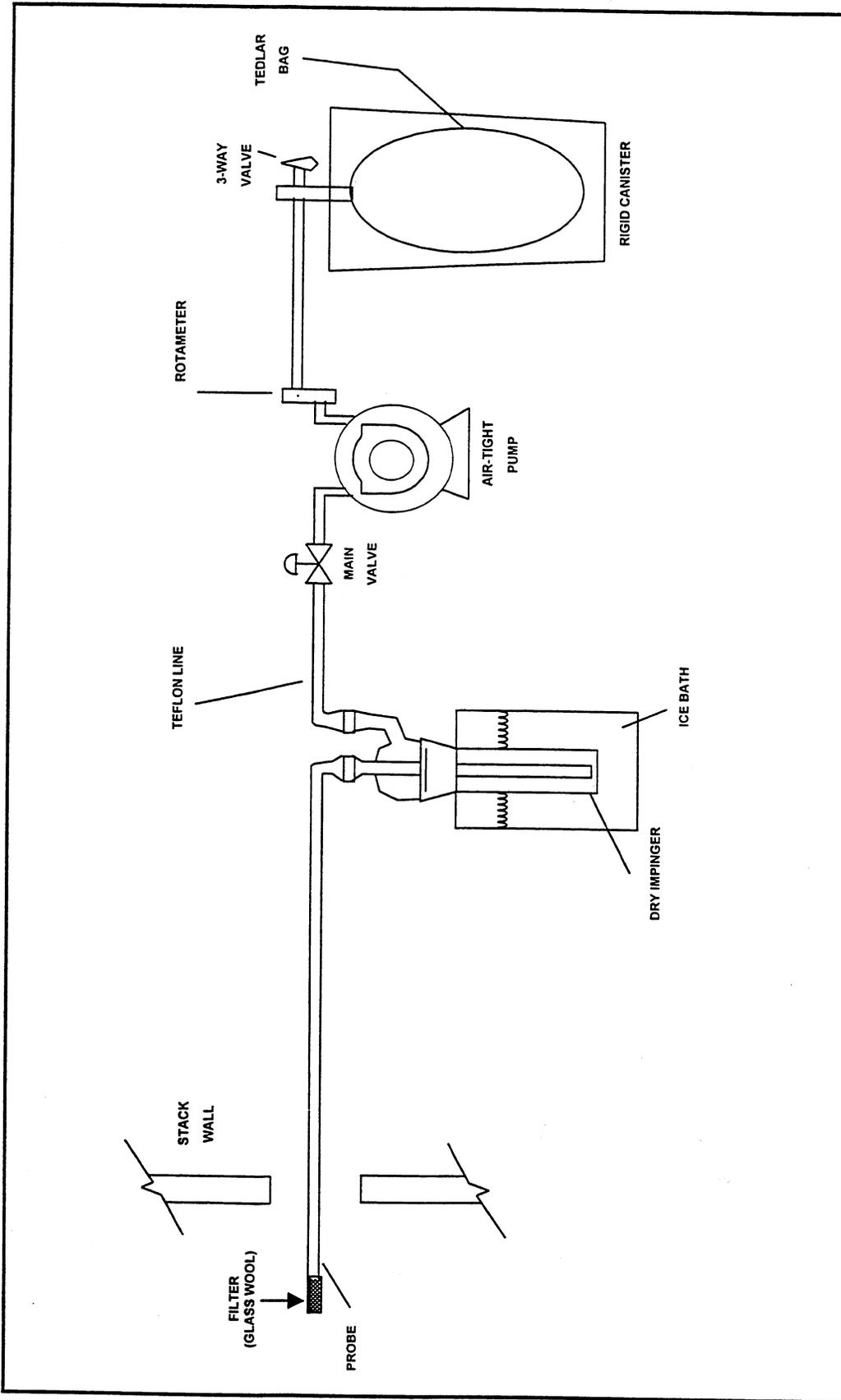


FIGURE 4-2  
EPA METHOD 3 - DRY GAS STREAM COMPOSITION SAMPLING TRAIN

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- An analyzer can provide information on the temporal variations of mercury emissions for a process that may be variable in its emission characteristics.

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  $\text{Hg}^0$  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 ( $\text{SnCl}_2$ ) or sodium borohydride ( $\text{NaBH}_4$ ) 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  $\text{Hg}^{2+}$  to  $\text{Hg}^0$ . The presence of  $\text{SO}_2$  (>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  $\text{Hg}^0$ , 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  $\text{SO}_2$ , 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  $\text{Hg}^0$ . However, by passing the gas through a solution such as  $\text{SnCl}_2$  which reduces all the mercury present in gas to  $\text{Hg}^0$ , total mercury can be measured. However, it has been shown that the presence of  $\text{SO}_2$  ( $>200 \text{ ppm}$ ) 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  $\text{Hg}^{2+}$  to  $\text{Hg}^0$ . The primary feature of this system is an acid gas trap that removes the  $\text{SO}_2$  without removing the mercury. The system can then be operated such that it can measure either total mercury or  $\text{Hg}^0$ .

**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  $500^\circ\text{C}$ . 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  $\text{Hg}^0$  as the primary standard. The  $\text{Hg}^0$  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  $\text{NO}_x$  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  $\text{SO}_2$ .

#### **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. All tests were 120 minutes in duration. Each of the twelve traverse points at the Unit No. 6 ESP inlet and outlet and Units 1-4 outlet were sampled for 5 minutes each resulting in a 120 minute test period. 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

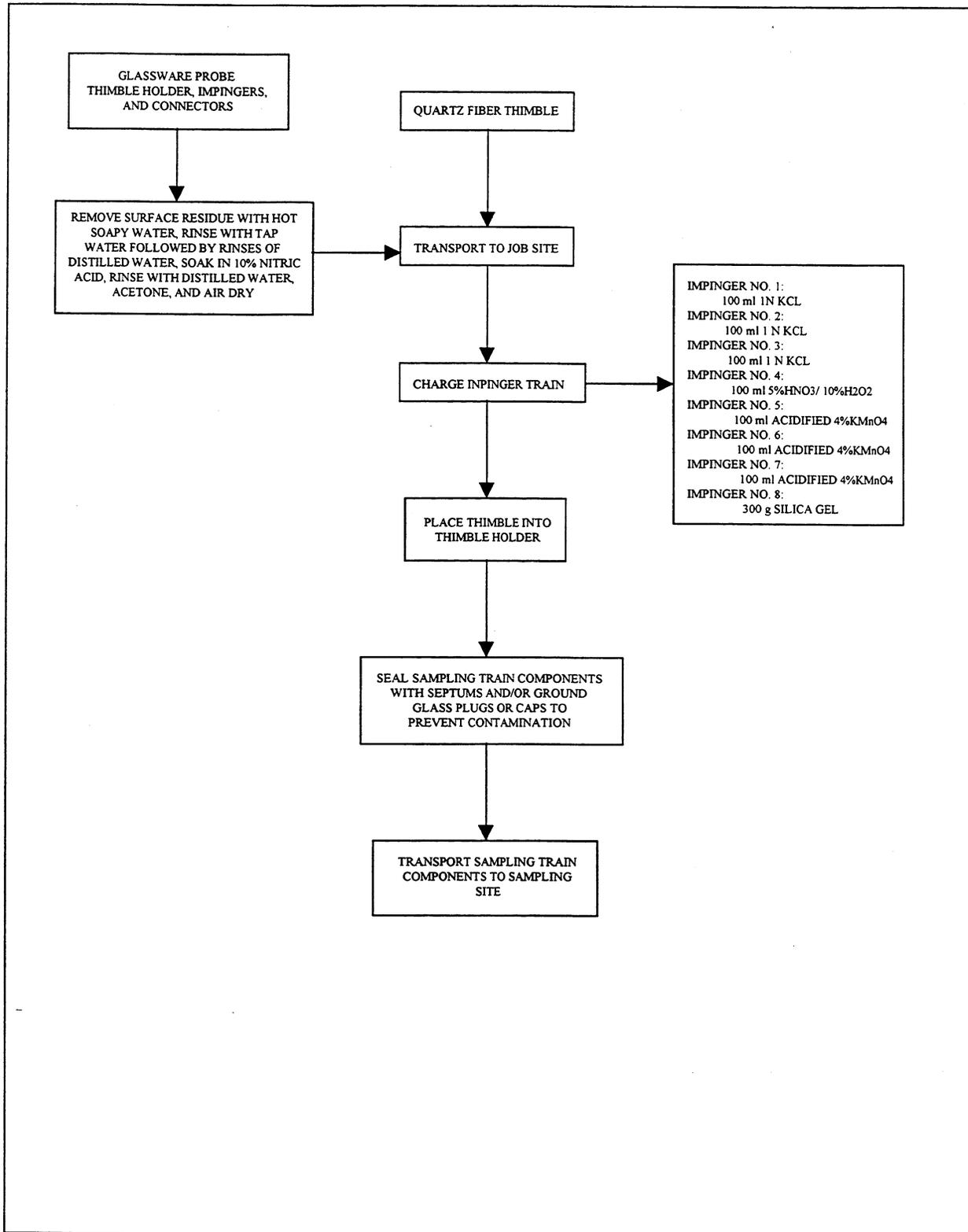


FIGURE 4-3  
PREPARATION PROCEDURES FOR ONTARIO HYDRO SAMPLING TRAIN

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.

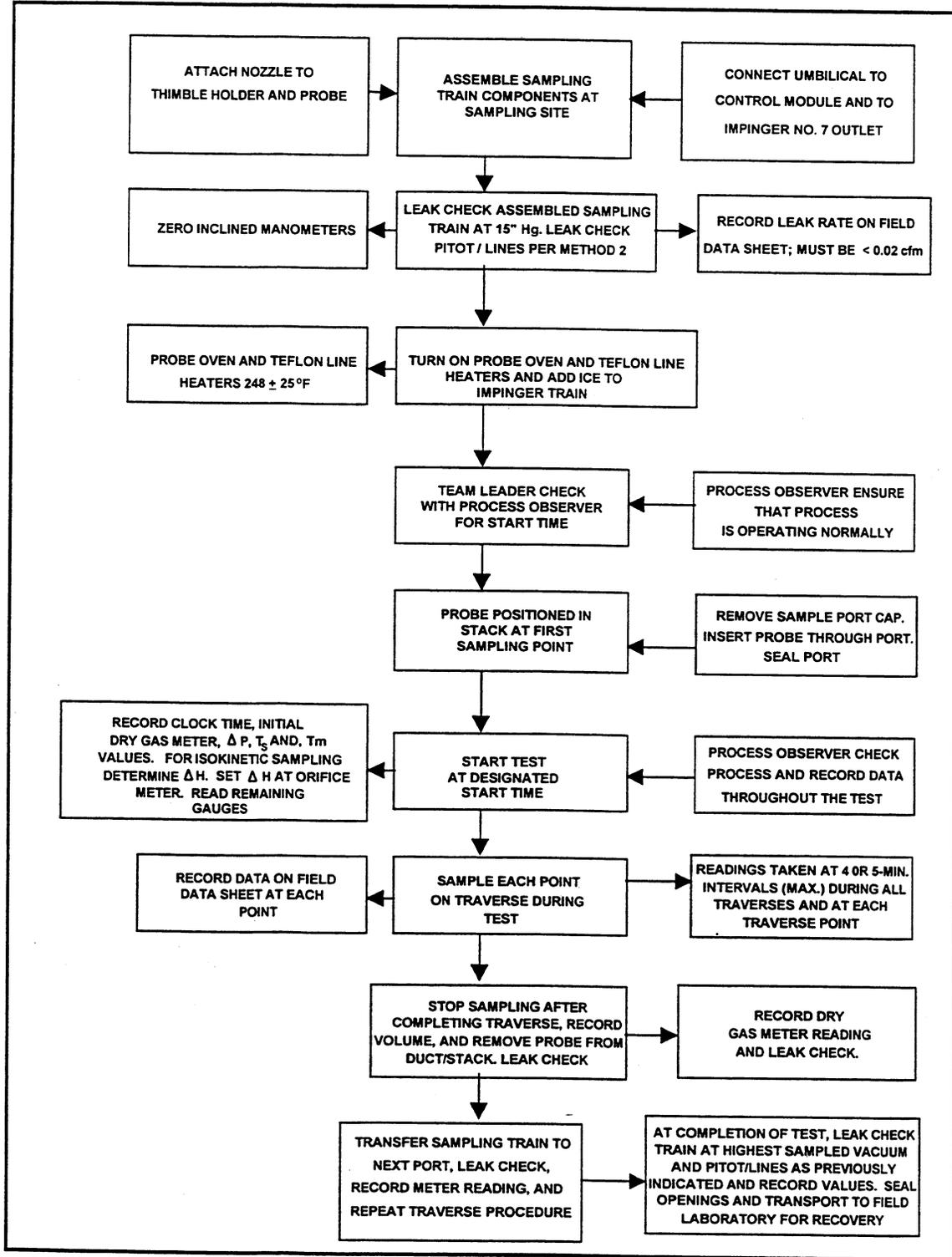
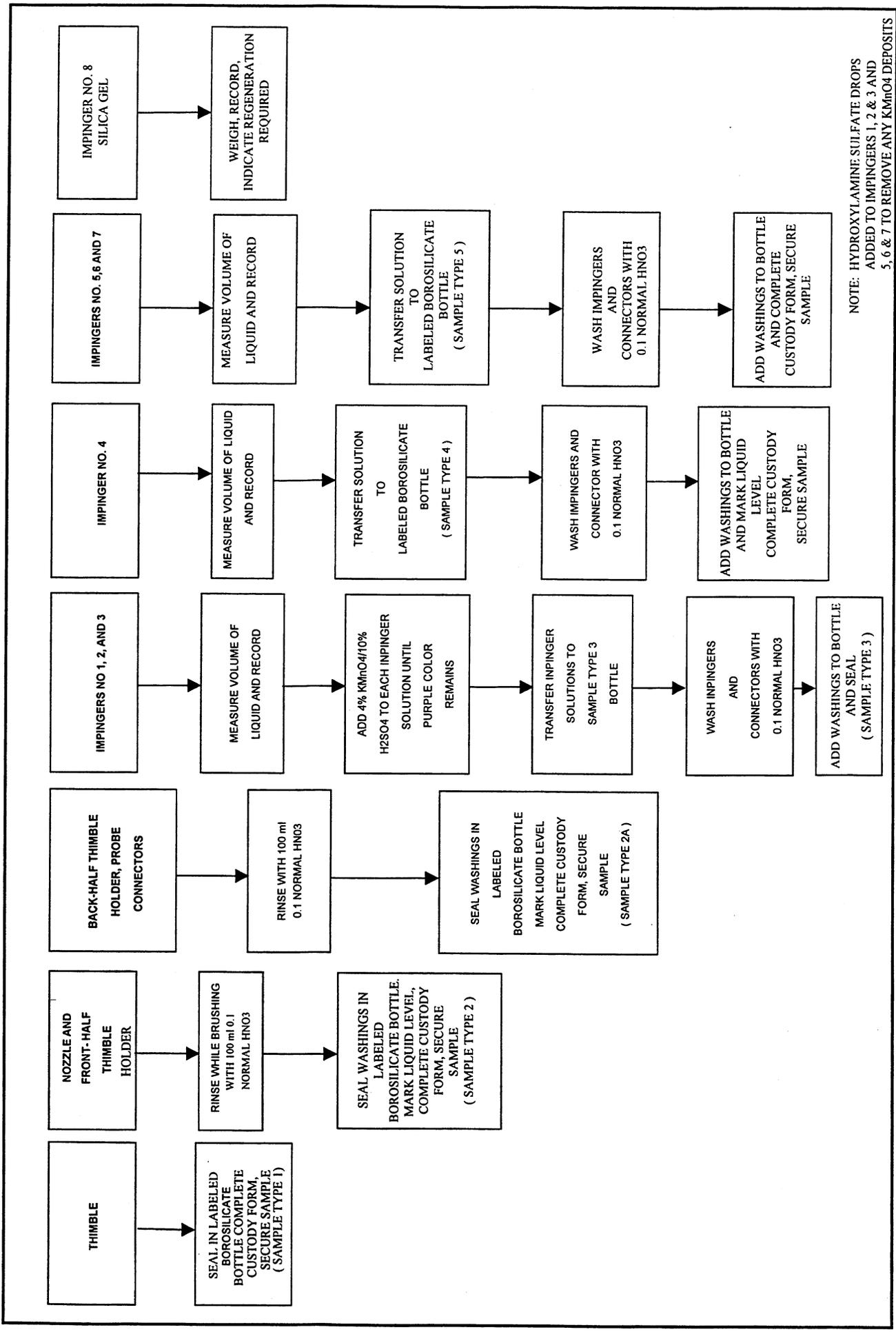


FIGURE 4 - 4  
SAMPLING PROCEDURES FOR ONTARIO HYDRO TRAIN

DOE21-D20



NOTE: HYDROXYLAMINE SULFATE DROPS ADDED TO IMPINGERS 1, 2 & 3 AND 5, 6 & 7 TO REMOVE ANY KMnO4 DEPOSITS

FIGURE 4 - 5  
SAMPLE RECOVERY PROCEDURES FOR ONTARIO HYDRO METHOD

**Table 4-1**

**Methods Used For the Analysis of  
Presque Isle Unit 6 and Units 1-4  
Process Solids Streams Samples**

<b>Analyte</b>	<b>Coal</b>	<b>ESP Ash</b>
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 Iso-peribol 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 HNO<sub>3</sub> 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 Na<sub>2</sub>O, K<sub>2</sub>O, MgO, CaO, Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and SO<sub>3</sub>.

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:

<b>Sample Fraction</b>	<b>Container No.</b>	<b>Mercury Form</b>
▪ 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 ( $\text{Hg}^{2+}$ )
▪ KCl impingers 1, 2, and 3 and rinses	Container No. 3	Oxidized ( $\text{Hg}^{2+}$ )
▪ $\text{HNO}_3/\text{H}_2\text{O}_2$ impingers and rinses	Container No. 4	Elemental ( $\text{Hg}^0$ )
▪ $\text{KMnO}_4/\text{H}_2\text{SO}_4$ impingers 5, 6, and 7 and rinses	Container No. 5	Elemental ( $\text{Hg}^0$ )

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 was tared and the total particulate collected in each thimble was measured. 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.

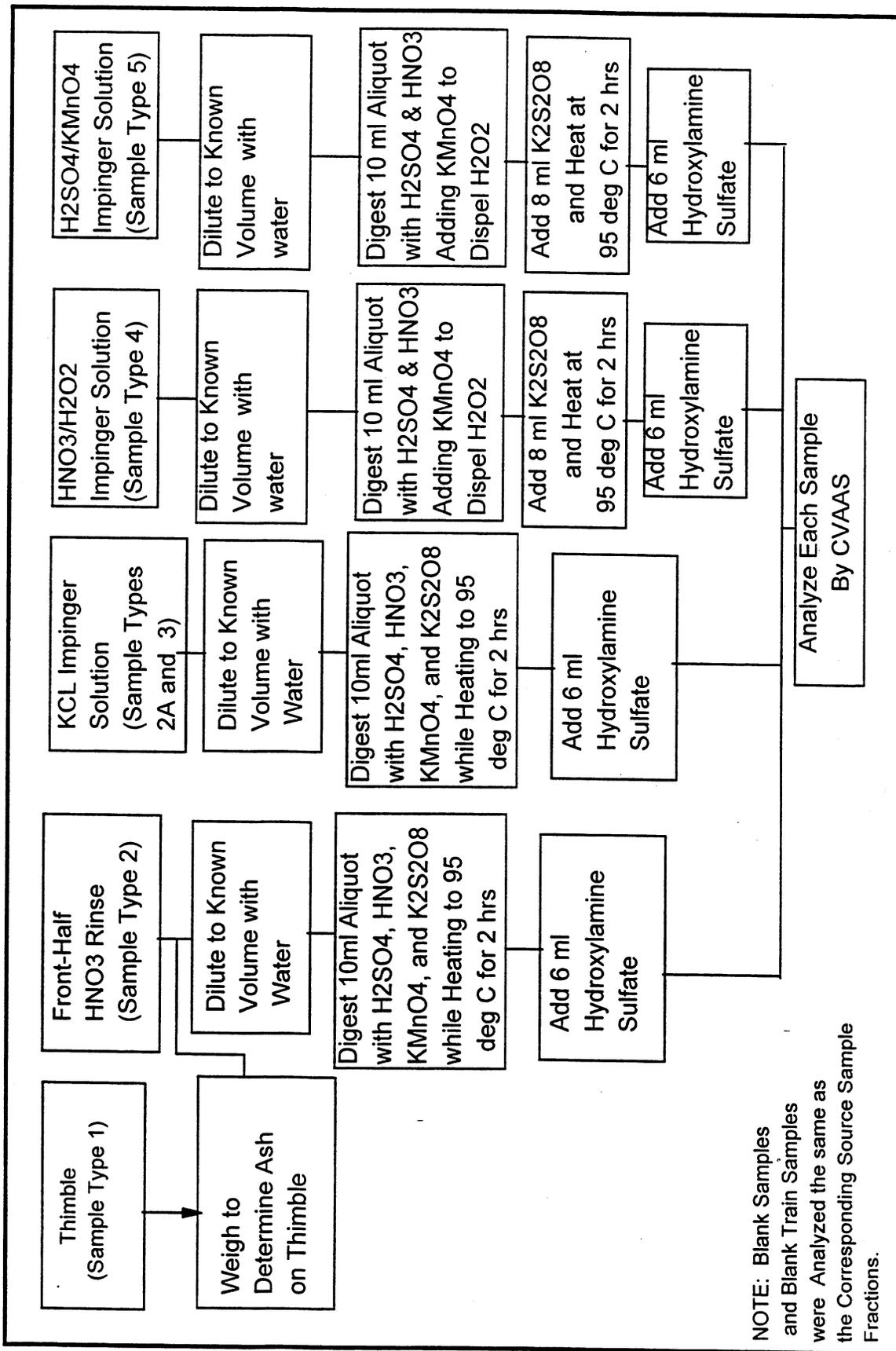


FIGURE 4 - 6  
ANALYTICAL PROCEDURE FOR  
ONTARIO HYDRO SAMPLING TRAIN

## **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 Unit 6 and Units 1-4 during the period of 14 through 16 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  $\leq 0.02$  CFM. Table 5-1 contains a summary of all isokinetic

Table 5-1

Stack Emission Sampling Field QA/QC Results

Test Location	Test Run	Isokinetic Sampling Rate <sup>1</sup>	Initial Leak Check Rate <sup>2</sup>	Final Leak Check Rate <sup>2</sup>	Gas Meter Calibration Values <sup>3</sup>	
					Pre	Post
Unit No. 6 ESP Inlet	1	103.9	0.010	0.008	1.009	0.9772
	2	100.6	0.010	0.011	1.009	0.9772
	3	101.5	0.013	0.014	1.009	0.9772
Unit No. 6 ESP Outlet	1	100.7	0.008	0.002	1.0098	1.0322
	2	98.1	0.010	0.008	1.0098	1.0322
	3	99.5	0.006	0.004	1.0098	1.0322
Units 1-4 Outlet	1	109.6	0.014	< 0.02	1.000	1.000
	2	90.4	0.011	< 0.02	1.000	1.000
	3	96.8	0.007	< 0.02	1.000	1.000

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

Note:

Silica gel impinger exit temperature maintained  $\leq 68\%$  during all test periods.

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 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 Unit 6 and Units 1-4. 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 Unit 6 and Units 1-4.

### **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 Units 5 and 9 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 10<sup>th</sup> 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 analyses 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 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 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.

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

SAMPLE ID	KCl Solution				H <sub>2</sub> O <sub>2</sub> Solution				KMnO <sub>4</sub> Solution			
	Sample No. 1				Sample No. 2				Sample No. 3			
	1	2	3	S.D.	1	2	3	S.D.	1	2	3	S.D.
B06-Out-2	3.96	3.99	3.91	0.0404	---	---	---	---	3.42	3.77	3.69	0.1834
B06-FBSPK	2.2	2.22	---	0.0141	1.73	1.55	---	0.1273	1.97	2	---	0.0212

**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

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.

## **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 107% 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.