

**WESTON PROJECT NO. 11634.001.002
EMISSIONS TEST REPORT
SOUTH MISSISSIPPI ELECTRIC
POWER ASSOCIATION
R.D. MORROW GENERATING STATION
HATTIESBURG, MISSISSIPPI**

27-28 OCTOBER 1999

**INFORMATION COLLECTION REQUEST
ASSESSMENT OF SPECIATED MERCURY
EMISSIONS FROM A COAL-FIRED BOILER**

Prepared for:

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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.

The South Mississippi Electric Power Association (SMEPA), R.D. Morrow Generating Station located in Hattiesburg, Mississippi was selected as one of the ICR study sites. Mercury speciation sampling was performed on Unit No. 2 at the Morrow Station facility using the Ontario Hydro method. During the ICR test program mercury speciation testing was performed on the inlet (ESP outlet) and outlet of the dry flue gas desulfurization unit (FGD) serving Unit No. 2.

The mercury speciation sampling activities were performed by Roy F. Weston, Inc. (WESTON®) and the analysis of the process and Ontario Hydro method samples were performed by Philip Analytical Services. The test program was performed during the period of 27-28 October 1999.

This test report presents the test data and test results of the mercury speciation sampling program performed on Unit No. 2 at the Morrow Station facility 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.

1.2 TEST PROGRAM OBJECTIVES

During the test program, mercury emissions testing was performed on the inlet and outlet of the FGD serving Unit No. 2 using the Ontario Hydro method. FGD inlet sampling was conducted at

the outlet of the electrostatic precipitator serving Unit No. 2. Representative samples of the coal were collected 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 boiler.
- Simultaneously measure concentrations and mass rates of speciated mercury at the inlet and outlet of the FGD on Unit No. 2.
- Obtain and analyze representative samples of the coal for the purpose of determining mercury, heating value, ash content, sulfur and chlorine levels.
- Document corresponding boiler, FGD and ESP operations along with facility continuous emission monitoring system (CEMs) data.

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 Unit No. 2.

1.3 SAMPLE LOCATIONS

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

- Coal Feed.

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

- Unit No. 2 FGD Inlet (ESP outlet).
- Unit No. 2 FGD 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.

Table 1-1

**SMEPA – R.D. Morrow Generating Plant
Unit No. 2
Process Solid and Flue Gas Streams with
Pollutants/Parameters**

Location/Stream Type	Pollutants or Parameters	Frequency
Unit No. 2 Coal Feed	Heating value Ash content Moisture Mercury (Hg) content Chlorine (Cl) content Sulfur content	One composite sample per run (total of 3) in conjunction with flue gas sampling on Unit No. 2.
Unit No. 2 FGD Inlet and Outlet (Stack)	Particulate bound and vapor phase mercury (including oxidized and elemental mercury speciation of vapor phase).	Inlet and outlet sampling by Ontario Hydro method on Unit No. 2.

1.5 TEST PROGRAM KEY PERSONNEL

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

Contact Name	Project Responsibility	Telephone No.	Facsimile No.
SMEPA Mr. Joey Ward	Facility Environmental Contact	(601) 261-2321	(601) 261-2395
EPA Mr. William Grimley	ICR Program Manager	(919) 541-1065	(919) 541-1039
WESTON Mr. Greg Sims Mr. Jack Mills	Project Manager Test Team Leader	(334) 887-0622 (610) 701-7245	(334) 826-0611 (610) 701-7401
PHILIP Mr. Vaughn O'Neill	Laboratory Analyst	(610) 921-8833	(610) 921-9667

2. PLANT AND SAMPLING LOCATION DESCRIPTIONS

2.1 SMEPA – R.D. MORROW GENERATING PLANT UNIT NO. 2 OVERVIEW

South Mississippi Electric Power Association operates Unit No. 2, which is a 1934 MMBtu/hr pulverized coal-fired boiler, at their R.D. Morrow Generating Station located in Purvis, Mississippi. The facility operates as a fossil fuel-fired electric power plant. Steam generated by burning bituminous coal is used to produce electricity in a steam turbine. The Unit No. 2 is designed to operate at a full load of 200 megawatts (mw).

Acid gas and particulate emissions are controlled using a FGD lime spray drying system and an electrostatic precipitator (ESP).

A continuous emission monitoring system (CEMS) measures the effluent concentration of nitrogen oxides (NO_x), sulfur dioxide (SO₂), carbon dioxide (CO₂), volumetric flow rate and opacity in the gas stream at the outlet stack location.

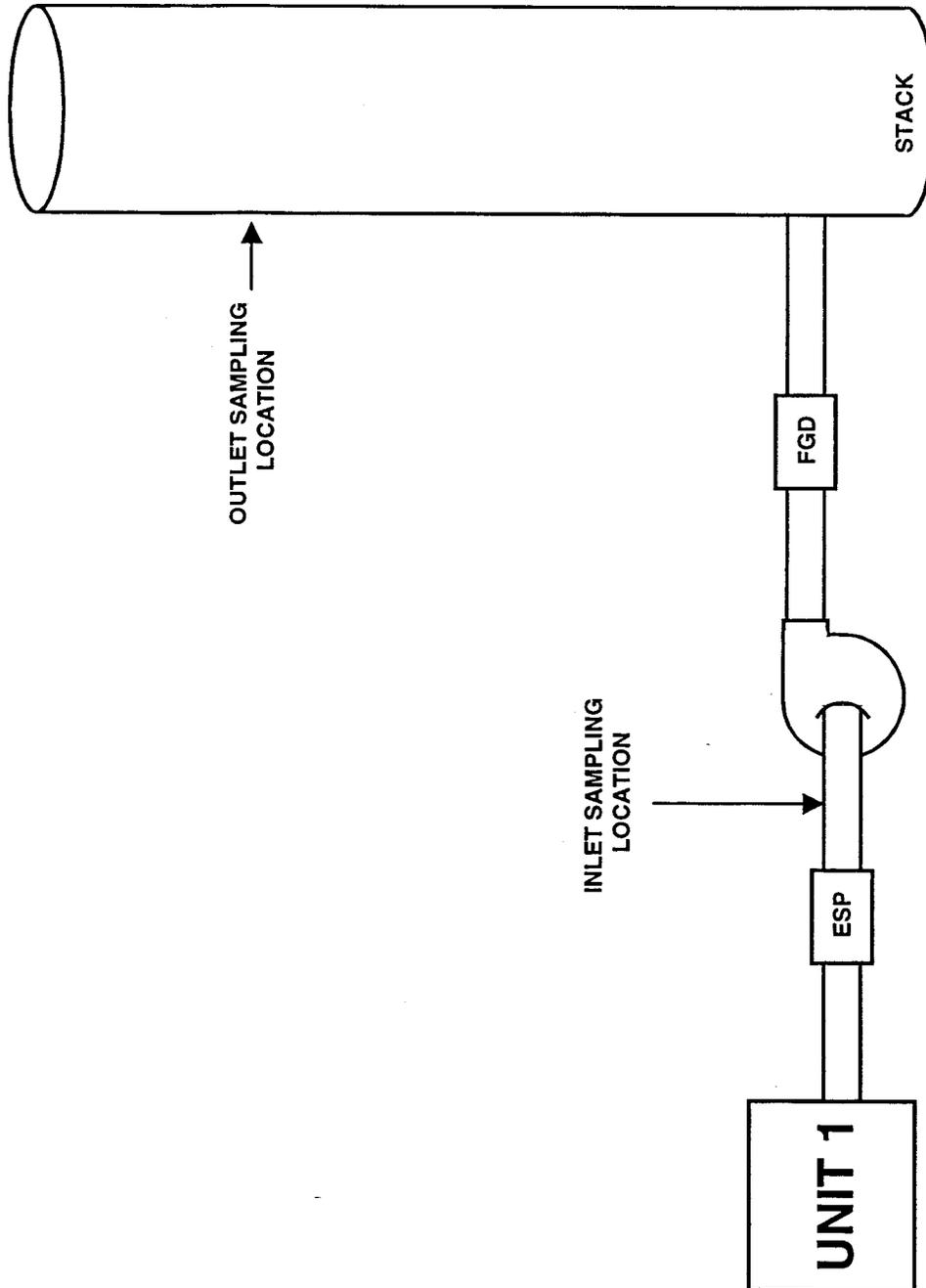
Figure 2-1 presents a schematic of the Unit No. 2 boiler and pollution control equipment.

2.2 PROCESS SOLID SAMPLING LOCATIONS AND SAMPLING PROCEDURES

2.2.1 Unit No. 2 Coal Sampling

Samples of the coal feed streams were collected and composited during each test run. The coal is introduced to the boiler by six (6) coal feeder tubes from three mills. Coal samples were obtained from each operating feeder tube into the boiler. Samples were collected once every 30-45 minutes from each of the operating feeder tubes throughout the duration of each of the three test runs.

Figure 2-1
Process Schematic and Sampling/Testing Location Unit No. 2



2.3 FLUE GAS SAMPLING LOCATIONS

2.3.1 Unit No. 2 FGD Inlet

The test site at the FGD inlets are located on a horizontal 8' 6" deep by 12' high rectangular duct. A total of five (5) 4" ID test ports are located vertically across the long side of each duct. The ports are located 39' (4.0 diameters) downstream from the ESP outlet and 4' (0.41 diameters) upstream of a flow disturbance in the duct prior to the induced draft (ID) fan leading to the FGD.

During each test run a total of five traverse points in each of the five ports (total of 25 points) on one of the two inlet ducts were sampled as recommended in the EPA Test Plan review and agreed to by WESTON.

See Figure 2-2 for a schematic of the FGD inlet test site.

2.3.2 Unit No. 2 FGD Outlet (Stack)

A total of four (4) 6" ID test ports are in place on the 201" ID flue. The test ports are located approximately 133' (7.9 diameters) downstream from the nearest disturbance and approximately 137' (8.2 diameters) from the nearest upstream distance (stack exit).

A total of 3 points per port (12 total) were sampled. See Figure 2-3 for a schematic of the Unit No. 2 stack test location.

Figure 2-2
Unit No. 2 FGD Inlet Duct Test Site - Port and Traverse Point Locations

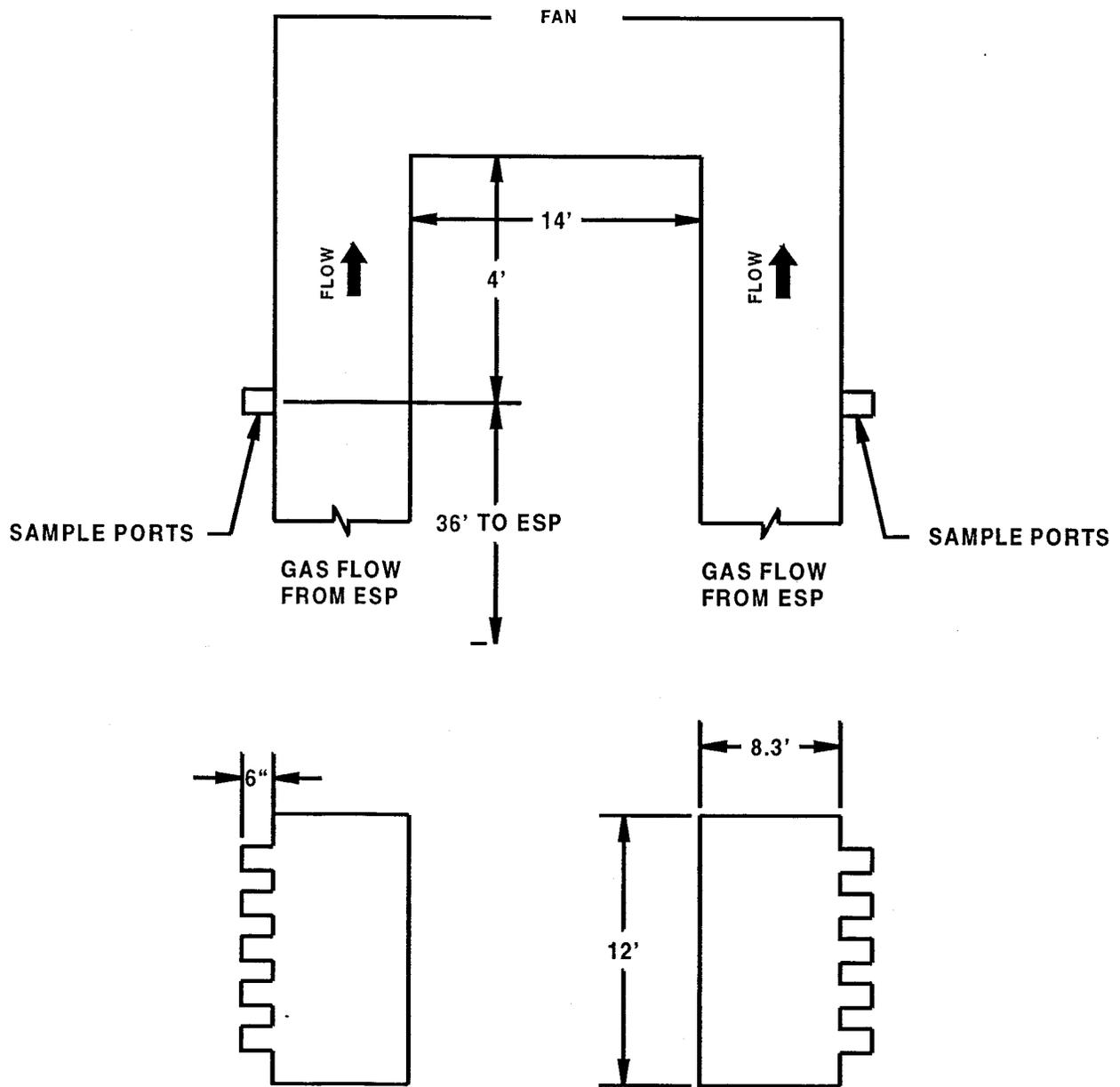
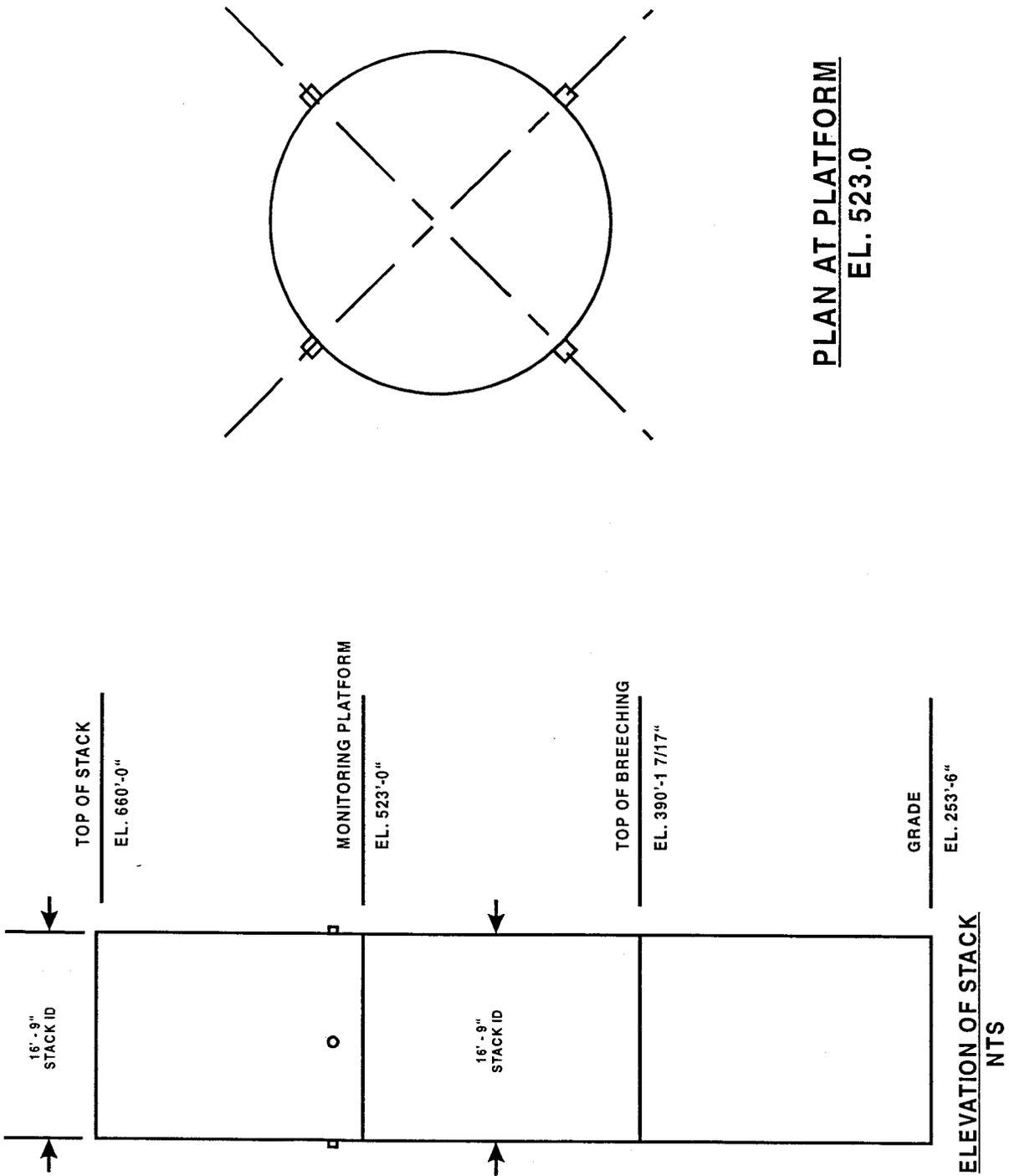


Figure 2-3
 Unit No. 2 Stack Test Site – Port and Traverse Point Locations



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 and 3-2 for the coal, 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 3-3, 3-4, and 3-5 for Unit No. 2.

Table 3-3 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 3-4 and 3-5 presents the mercury concentrations and mass rate values for particulate, oxidized, elemental and total mercury for each individual test run along with the measured

**Table 3-1
Sampling/Testing, Analytical, and QC Plan
Unit No. 2 Clean Coal Feed**

No. of Test Runs: 3

Test Objective: Determine total mercury and chlorine content of as-fired coal.

Sampling Objective: Collect a representative sample.

Parameters to be Determined:	Mercury	Chlorine	Heating Value	Ash Content	Sulfur Content	Mass Flow Rate
Sampling or Monitoring Method:	Representative sample increments were obtained from the six (6) individual boiler coal feeder tubes once every 45 minutes during each test period. Samples stored in air-tight, plastic-lined bucket					
Sample Preparation/Extraction and Analysis Method(s):	ASTM D2013 and EPA Method 7471	ASTM E776 and EPA Method 300	ASTM D3286	ASTM D3174	ASTM D4239	Gravimetric feeder readings recorded in control room
Maximum Holding Time (days):	28	28	28	28	28	NA
Sampling or Monitoring Design:						
Total No. of Samples	3	3	3	3	3	NA
Site Blanks	0	0	0	0	0	NA
Trip Blanks	0	0	0	0	0	NA
Lab Blanks	1	1	1	1	1	NA
Blank Spikes ²	0	0	0	0	0	NA
Replicates ³	1/batch ¹	1/batch	1/batch	1/batch	1/batch	NA
QC Spikes ⁴	1/batch	1/batch	1/batch	1/batch	1/batch	NA
Total No. of Samples Analyzed	6	6	6	6	6	NA
Analytical Laboratory:	Philip Analytical Services					
Notes:	¹ A batch consists of a maximum of 20 samples. ² 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. ³ This indicates that a duplicate analysis is made on one or more samples as a QC mechanism to measure analytical precision. ⁴ 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 No. 2 FGD Inlet and Outlet**

No. of Test Runs: 3 per unit

Test Objective: Perform mercury speciation sampling at inlet and outlet of FGD.

Sampling Objective: Collect a representative sample.

Parameters to be Determined:	Speciated Mercury
Sampling or Monitoring and Preservation Method(s)	Ontario Hydro Method
Sample Preparation/Extraction and Analysis Method(s)	Ontario Hydro Method
Maximum Holding Time (days)	45
Sampling or Monitoring Design	
Length of Test	≥ 120 min
Sample Size	1 to 2.5 m ³
Total No. of Samples	3 at inlet and outlet
Site/Reagent Blanks	Minimum of 1 per sample type
Train Blanks	1 per test location (total of 2)
Lab Blanks	1 per batch ¹
Blank Spikes ²	1 per batch
Replicates ³	All samples
Total No. of Samples Analyzed ⁴	~90
Analytical Laboratory	Philip Analytical Services

¹A batch consists of a maximum of 10 samples.

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

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

⁴Approximate number of total samples and individual fractions, duplicates and other QC samples.

Note: The facility CEMs measured sulfur dioxide (SO₂), oxides of nitrogen (NO_x), carbon dioxide (CO₂), opacity and flow on the FGD outlet.

Table 3-3
Comparison of Mercury Speciation to Total Mercury Results
Unit No. 2

	Run 1		Run 2		Run 3		Mean	
	FGD Inlet	FGD Outlet						
Total Mercury Emissions								
Concentration, $\mu\text{g}/\text{M}^3$	11.63	5.29	9.73	4.85	8.14	4.38	9.83	4.84
Emission Rate, lb/hr	2.18E-2	1.01E-2	1.87E-2	9.51E-3	1.45E-2	8.03E-3	1.83E-2	9.23E-3
FGD ESP Removal Efficiency, %	53.7		49.1		44.6		49.1	
Particulate Bound Mercury Emissions								
Concentration, $\mu\text{g}/\text{M}^3$	0.04	<0.04	<0.01	<0.02	<0.02	<0.03	0.02	<0.03
% of Total Mercury	0.34	<0.76	<0.10	<0.41	<0.25	<0.68	0.23	<0.62
Oxidized Mercury Emissions								
Concentration, $\mu\text{g}/\text{M}^3$	8.2	1.5	6.5	1.4	5.5	0.86	6.8	1.3
% of Total Mercury	70.5	28.4	66.8	28.9	67.6	19.6	68.3	25.6
Elemental Mercury Emissions								
Concentration, $\mu\text{g}/\text{M}^3$	3.4	3.7	3.2	3.5	2.6	3.5	3.1	3.6
% of Total Mercury	29.2	69.9	32.9	72.2	31.9	79.9	31.5	74.4

Table 3-4
Summary of Mercury Speciation Test Data and Test Results
Unit No. 2 FGD Inlet

TEST DATA:				
Test run number	1	2	3	AVERAGE
Location		Unit No. 2 Inlet		
Test date	10/27/99	10/28/99	10/28/99	---
Test time period	1355-1623	902-1125	1232-1507	---
PROCESS DATA:				
Unit Load, MW	202	201	201	201
Coal feed rate, lb/hr.	158793	165648	154374	159605
Coal Btu content, Btu/lb.	12960	12760	12640	12787
Heat Input, 10 ⁶ Btu/hr	2058	2114	1951	2041
GAS STREAM VELOCITY AND VOLUMETRIC FLOW DATA:				
Avg. Stack gas stream volumetric flow, dscf/min. ⁽¹⁾	512020	523151	489723	508298
Stack O ₂ , % by volume, dry basis	7.6	7.2	7.2	7.3
Inlet O ₂ , % by volume, dry basis	7.3	6.9	6.8	7.0
Corrected volumetric flow rate, dscf/min ⁽⁴⁾	500808	512020	475928	496252
PARTICULATE BOUND MERCURY EMISSIONS:				
Conc., ug/m ³	0.04	< 0.01	< 0.02	0.02
Conc., ug/Nm ³⁽²⁾	0.05	< 0.01	< 0.02	0.03
Emission rate, lbs/10 ¹² Btu.	0.04	< 0.01	< 0.01	0.02
Emission rate, lbs/hr	8.32E-05	< 2.50E-05	< 2.82E-05	4.54E-05
OXIDIZED MERCURY EMISSIONS:				
Conc., ug/m ³	8.22	6.51	5.51	6.75
Conc., ug/Nm ³⁽²⁾	8.82	6.98	5.91	7.24
Emission rate, lbs/10 ¹² Btu.	7.49	5.91	5.03	6.14
Emission rate, lbs/hr	1.54E-02	1.25E-02	9.82E-03	1.26E-02
ELEMENTAL MERCURY EMISSIONS:				
Conc., ug/m ³	3.36	3.21	2.62	3.06
Conc., ug/Nm ³⁽²⁾	3.61	3.44	2.81	3.29
Emission rate, lbs/10 ¹² Btu.	3.07	2.91	2.39	2.79
Emission rate, lbs/hr	6.31E-03	6.15E-03	4.67E-03	5.71E-03
TOTAL MERCURY EMISSIONS: ⁽³⁾				
Conc., ug/m ³	11.63	9.73	8.14	9.83
Conc., ug/Nm ³⁽²⁾	12.47	10.44	8.74	10.55
Emission rate, lbs/10 ¹² Btu.	10.60	8.83	7.44	8.95
Emission rate, lbs/hr	2.18E-02	1.87E-02	1.45E-02	1.83E-02

(1) Standard conditions = 68 deg. F. (20 deg. C.) and 29.92 inches Hg (760mm Hg). These values are shown only to present the VFR measured at the selected Inlet duct (1 of 2) and were not used to calculate Inlet pollutant mass rates (lb/hr).

(2) Nm³ = Normal cubic meter (32 deg. F. (0 deg. C.) and 29.92 inches Hg (760mm Hg)).

(3) Non-detects included in total mercury catch value.

(4) Since only one of the two Inlet ducts were sampled, total Inlet volumetric flow rate was determined from the total Outlet flow rate corrected for oxygen concentrations determined at the Inlet and Outlet. The corrected volumetric flow rate was used to calculate Inlet pollutant mass rates (lb/hr).

**Table 3-5
Summary of Mercury Speciation Test Data and Test Results
Unit No. 2 FGD Outlet**

TEST DATA:				
	1	2	3	AVERAGE
Test run number				
Location		Unit No. 2 Outlet		
Test date	10/27/99	10/28/99	10/28/99	---
Test time period	1355-1623	902-1125	1232-1507	---
PROCESS DATA:				
Unit Load, MW	202	201	201	201
Coal feed rate, lb/hr.	158793	165648	154374	159605
Coal Btu content, Btu/lb.	12960	12760	12640	12787
Heat Input, 10 ⁶ Btu/hr	2058	2114	1951	2041
GAS STREAM VELOCITY AND VOLUMETRIC FLOW DATA:				
Avg. gas stream velocity, ft./sec.	54.4	55.6	52.6	54.2
Avg. gas stream volumetric flow, wacf/min.	719759	734581	696019	716786
Avg. gas stream volumetric flow, dscf/min. ⁽¹⁾	512020	523151	489723	508298
PARTICULATE BOUND MERCURY EMISSIONS:				
Conc., ug/m ³	< 0.04	< 0.02	< 0.03	< 0.03
Conc., ug/Nm ³ ⁽²⁾	< 0.04	< 0.03	< 0.03	< 0.03
Emission rate, lbs/10 ¹² Btu.	< 0.04	< 0.02	< 0.03	< 0.03
Emission rate, lbs/hr	< 7.87E-05	< 4.75E-05	< 4.94E-05	< 5.85E-05
OXIDIZED MERCURY EMISSIONS:				
Conc., ug/m ³	1.53	1.37	0.86	1.26
Conc., ug/Nm ³ ⁽²⁾	1.64	1.47	0.92	1.35
Emission rate, lbs/10 ¹² Btu.	1.43	1.27	0.81	1.17
Emission rate, lbs/hr	2.94E-03	2.69E-03	1.58E-03	2.40E-03
ELEMENTAL MERCURY EMISSIONS:				
Conc., ug/m ³	3.72	3.45	3.49	3.55
Conc., ug/Nm ³ ⁽²⁾	3.99	3.71	3.74	3.81
Emission rate, lbs/10 ¹² Btu.	3.46	3.20	3.28	3.32
Emission rate, lbs/hr	7.13E-03	6.77E-03	6.40E-03	6.77E-03
TOTAL MERCURY EMISSIONS: ⁽³⁾				
Conc., ug/m ³	5.29	4.85	4.38	4.84
Conc., ug/Nm ³ ⁽²⁾	5.68	5.21	4.70	5.19
Emission rate, lbs/10 ¹² Btu.	4.93	4.50	4.11	4.51
Emission rate, lbs/hr	1.01E-02	9.51E-03	8.03E-03	9.23E-03

(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) Non-detects included in total mercury catch value.

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 No. 2

For Unit No. 2 FGD Inlet an average of 0.23 percent of the total mercury measured is particulate bound mercury. On average the oxidized mercury was 68.3 percent of the total and the elemental mercury was approximately 31.5 percent of the total mercury collected. At the Unit No. 2 FGD outlet, elemental mercury comprised 74.4 percent of the total. The oxidized mercury was 25.6 percent of the total and the particulate bound mercury was <0.62 percent.

Based on the total mercury measurements the average removal efficiency for the FGD was 50.3 percent with an average outlet mass emission rate of 9.23 E-03 pounds per hour.

The average total mercury emission rates for Unit No. 2 outlet are 4.84 $\mu\text{g}/\text{m}^3$, 4.51 lbs/10¹² Btu and 9.23 E-03 lb/hr.

3.2.2 Process Solid Sample Stream Results

Table 3-6 provides a summary of the analytical results obtained on the coal feed samples collected on Unit No. 2.

For each parameter measured on the Unit No. 2 coal feed stream, the concentration or percent value is presented (on or as received basis) for each individual test run along with the average values.

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

Based on the mercury content of the coal and the measured coal feed rate, the mass rate of mercury introduced to the boiler averaged 0.016 lb/hr.

Table 3-6
Summary of Coal Sample Results
Unit No. 2 Coal Feed Samples

Parameter ¹	Test Run No.			Average
	1	2	3	
Mercury, ppm (mg/kg)	<0.10	<0.10	<0.10	<0.10
Chlorine, %	0.09	0.09	0.07	0.08
Heating value, Btu/lb	12960	12760	12640	12787
Ash, %	10.0	10.8	11.4	10.7
Sulfur, %	1.09	1.09	1.03	1.07
Moisture, %	1.41	1.71	1.83	1.65

(1) As received basis.

3.2.3 Unit Operation and Key Operational Parameters

This section describes the Unit No. 2 operations during the test program and provides the key operating parameters that were monitored and documented during testing.

3.2.3.1 Unit Operation During Testing

Operation of Unit No. 2 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 the boiler during testing.

3.2.3.2 Process Control Data

All key power generation process operating parameters and control data were recorded during each test period. FGD and ESP operational indicators data were recorded by a data acquisition system. The facilities CEMS data acquisition system provided concentration values.

A summary of the key operating data is provided in Table 3-7 for Unit No. 2. All additional boiler, FGD and ESP operations data and CEM data are provided in Appendix B.

3.3 TESTING PROBLEMS OR MODIFICATIONS

Per the Site-Specific Test Plan, an out-of-stack thimble holder was utilized at the inlet test location for test runs 1-3.

It should be noted that during the analysis of the Ontario Hydro samples, Philip Analytical Services noted some inconsistencies in the method equations. These inconsistencies were brought to the attention of EPA and the Energy & Environmental Research Center (EERC) for correction. The comments provided by Philip relating to the equations are provided in the laboratory report in Appendix D.

No further sampling or analytical problems were noted during the test program. No process problems were noted during any of the test periods.

**Table 3-7
Summary of Key Process Control Data
Unit No. 2**

Parameter	Units	Run No.		
		1	2	3
Gross Generation	MW	202	201	201
Net Generation	MW	188	188	189
Coal Total	lbs/hr	158,793	165,648	154,374
Main Steam Flow	10 ³ lb/hr	1418	1400	1402
Main Stream Pressure	psig	2580	2580	2580
Main Steam Temp.	°F	980	990	970
Scrubber Feed Rate (Make-Up)	gpm	34.96	33.07	29.55
Absorber PH (Recycle Tank)	----	5.4	5.4	5.3
Scrubber Module Differential Pressure	in. H ₂ O	9.1	8.4	8.4
Demist Section Differential Pressure	in. H ₂ O	1.0	2.2	2.1
Scrubber Module Outlet Temperature	°F	127.5	126.7	127.0
Stack gas flow (CEMs)	Kscfh	34,654	35,093	33,144
Stack Gas Exit Temperature	°F	179.5	----	----
Stack opacity	%	8.6	8.1	7.6
Stack CEMs (SO ₂)	ppm/v	238	248	240
Stack CEMs (SO ₂)	lb/MMBtu	0.655	0.657	0.649
Stack CEMs (NO _x)	ppm/v	258	252	250
Stack CEM (CO ₂)	%	11.2	11.0	11.0

4. SAMPLING AND ANALYTICAL PROCEDURES

4.1 DESCRIPTION OF SAMPLING EQUIPMENT

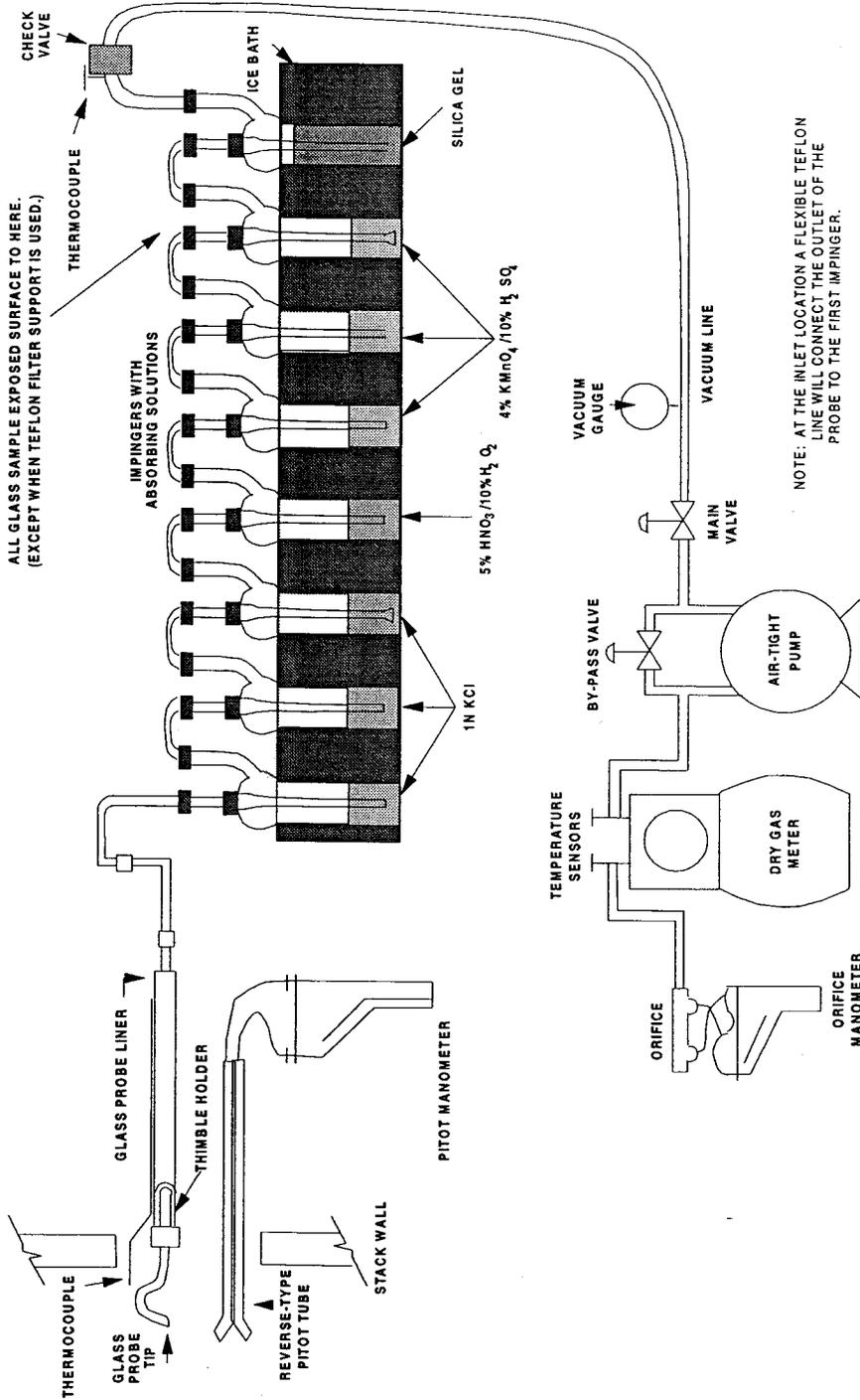
4.1.1 Ontario Hydro Mercury Speciation Method

The Ontario Hydro sampling train contained the following components:

- At the inlet location a calibrated borosilicate nozzle was attached to a heated borosilicate probe. The probe was attached to a thimble holder containing a high capacity quartz fiber thimble. A heated Teflon line connected the thimble holder outlet to the first impinger.
- At the inlet location the heated borosilicate probe was equipped with a calibrated thermocouple to measure flue gas temperature and a calibrated S-type pitot tube to measure flue gas velocity pressure.
- At the outlet location the heated borosilicate probe and nozzle was attached to a heated filter holder containing a 90-millimeter (mm) quartz fiber filter. The probe was equipped with a calibrated thermocouple to measure flue gas temperature and a calibrated S-type pitot tube to measure flue gas velocity pressure.
- An impinger train consisting of eight impingers. The first, second, and third impingers each contained 100 ml of 1 Normal (N) potassium chloride (KCl). The fourth impinger contained 100 ml of 5% nitric acid (HNO₃) and 10% hydrogen peroxide (H₂O₂). The fifth, sixth and seventh impingers each contained 100 ml of 4% potassium permanganate (KMnO₄) and 10% sulfuric acid (H₂SO₄). The eighth impinger contained 300 grams of dry preweighed silica gel. The third and seventh impingers were a Greenburg-Smith type; all other impingers were of a modified design. All impingers were maintained in a crushed ice bath.
- A vacuum line (umbilical cord) with adapter to connect the outlet of the impinger train to a control module.
- A control module containing a 3-cfm carbon vane vacuum pump (sample gas mover), a calibrated dry gas meter (sample gas volume measurement device), a calibrated orifice (sample gas flow rate monitor) and inclined manometers (orifice and gas stream pressure indicators).
- A switchable calibrated digital pyrometer to monitor flue and sample gas temperatures.

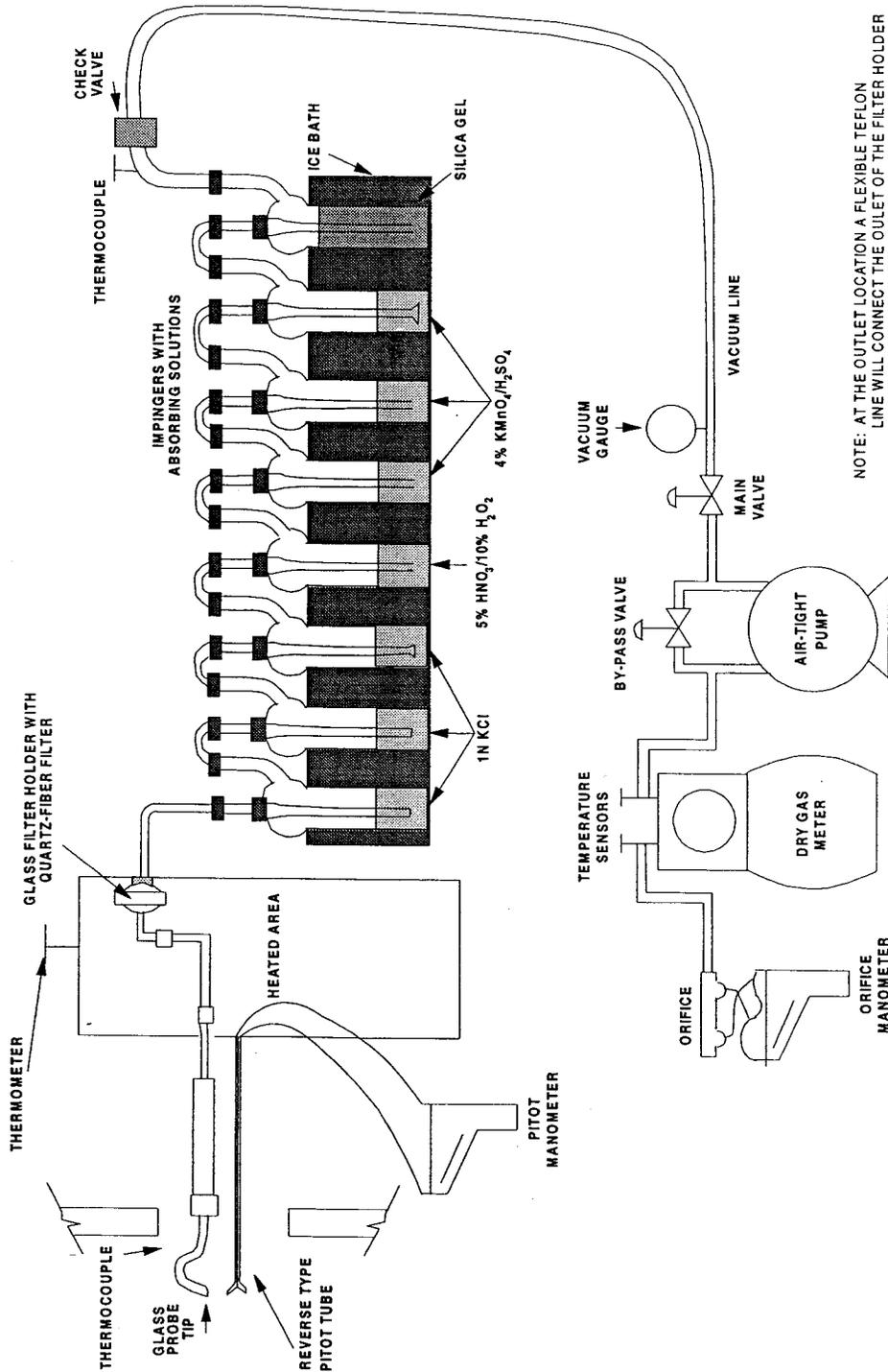
See Figures 4-1 and 4-2 for schematics of the Ontario Hydro test trains.

Figure 4-1
FGD Inlet Test Location Ontario Hydro Sampling Train



03/07/00

Figure 4-2
FGD Outlet Test Location Ontario Hydro Sampling Train



1:141634/001/002/SMPEA/SMEPARPT.wmf

4.2 CO₂ AND O₂ SAMPLING EQUIPMENT

The fixed gases sampling train (Figure 4-3) used at the Unit No. 2 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. 2, the CO₂ and O₂ concentrations of each bag were determined using Orsat analysis.

4.3 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.

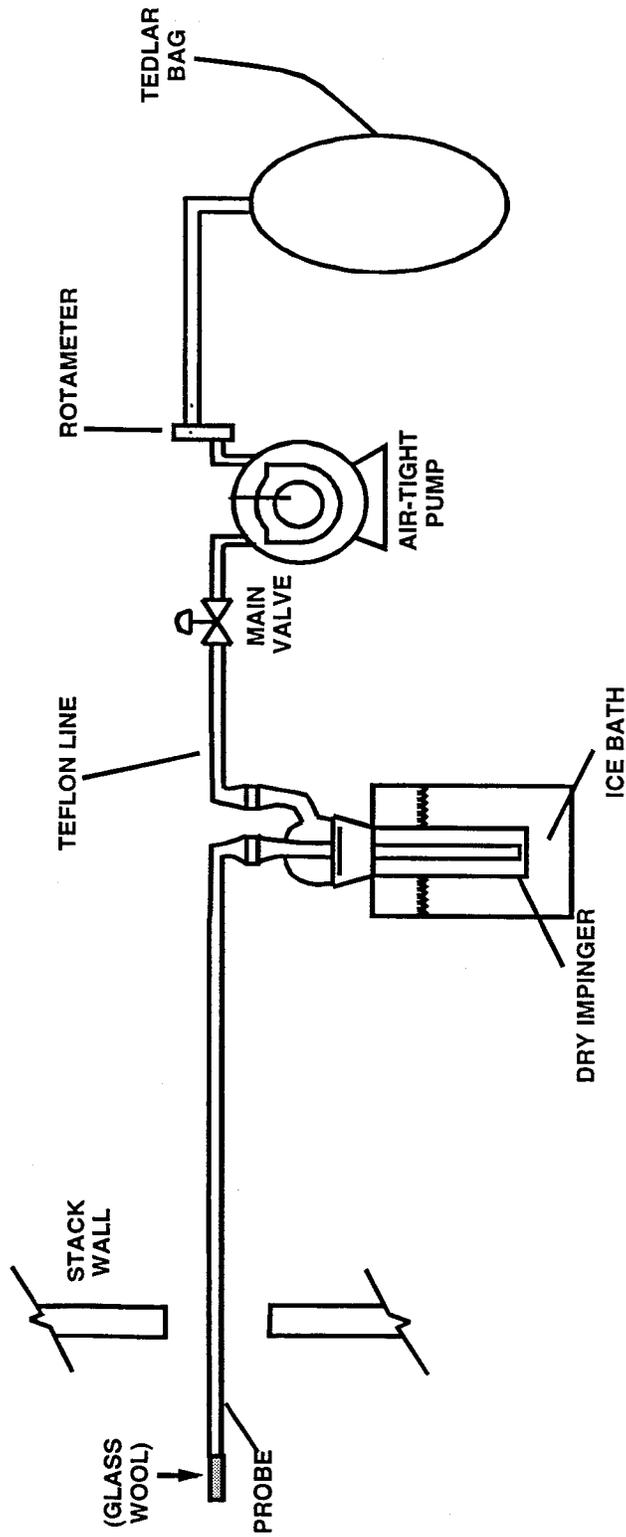
4.3.1 Preliminary Tests

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 both 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

Figure 4-3
EPA Method 3 – Dry Gas Stream Composition Sampling Train



in the Site-Specific Sampling/Testing, Analytical and QA/QC plan. Each inlet test run was 125 minutes in duration with readings taken at each of the 25 traverse points once every 5 minutes. The outlet tests were 120 minutes in length and each of the 12 traverse points were sampled for 10 minutes with readings taken every 5 minutes. 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 4-4 illustrates the train preparation. Figure 4-5 illustrates the sampling procedures. Figure 4-6 illustrates the sample recovery procedures.

4.4 ANALYTICAL PROCEDURES

4.4.1 Sample Analyses

4.4.1.1 Ontario Hydro Sample Analyses

Figure 4-7 presents a schematic of the analytical procedures used during analysis of the Ontario Hydro samples.

4.4.1.2 Coal Sample Analyses

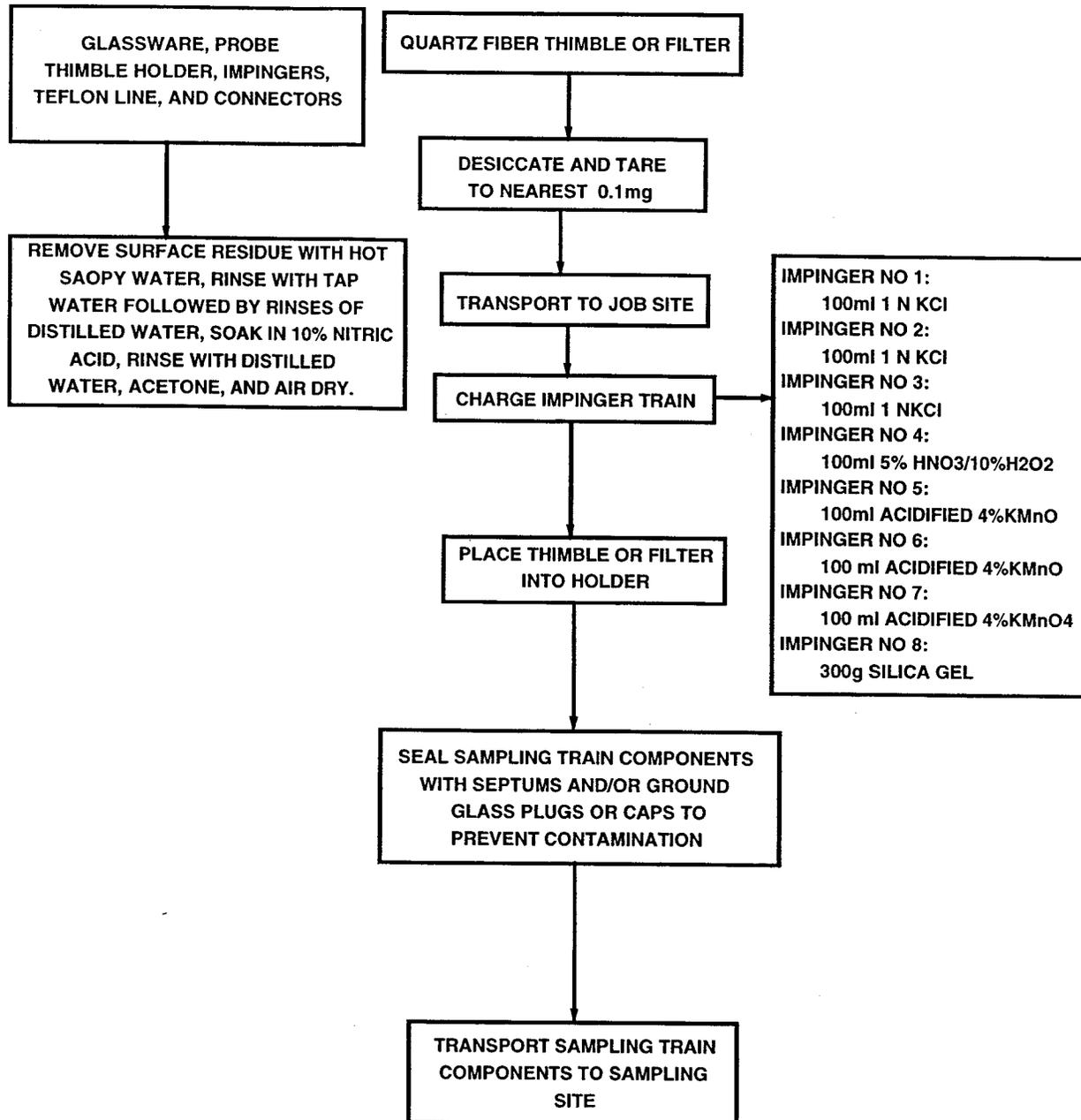
4.4.1.2.1 Preparation

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

4.4.1.2.2 Chlorine

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 determined by ion-chromatography (IC) using EPA Method 300 procedures.

**Figure 4-4
Preparation Procedures for Ontario Hydro Sampling train**



**Figure 4-5
Sampling Procedures for Ontario Hydro Train**

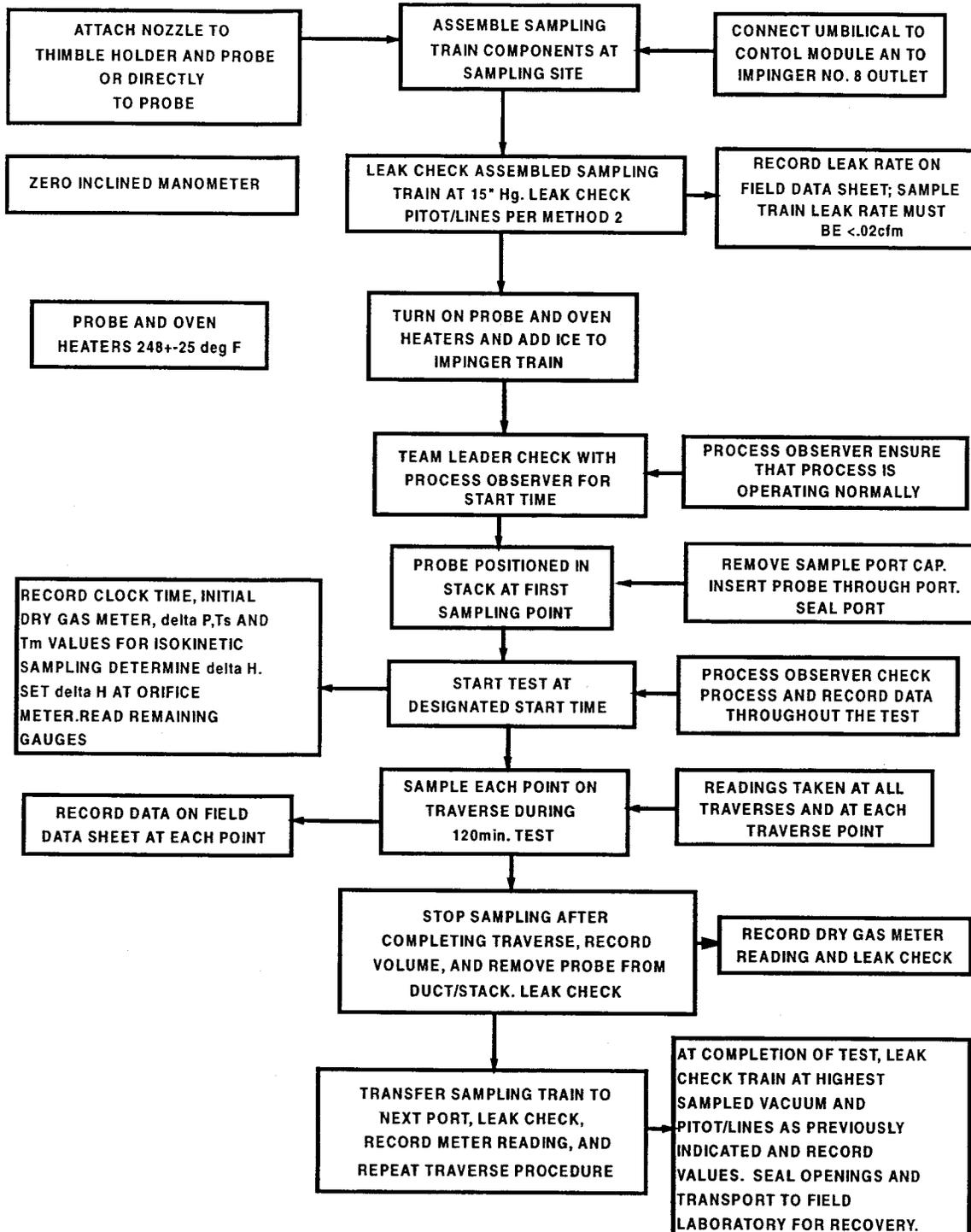
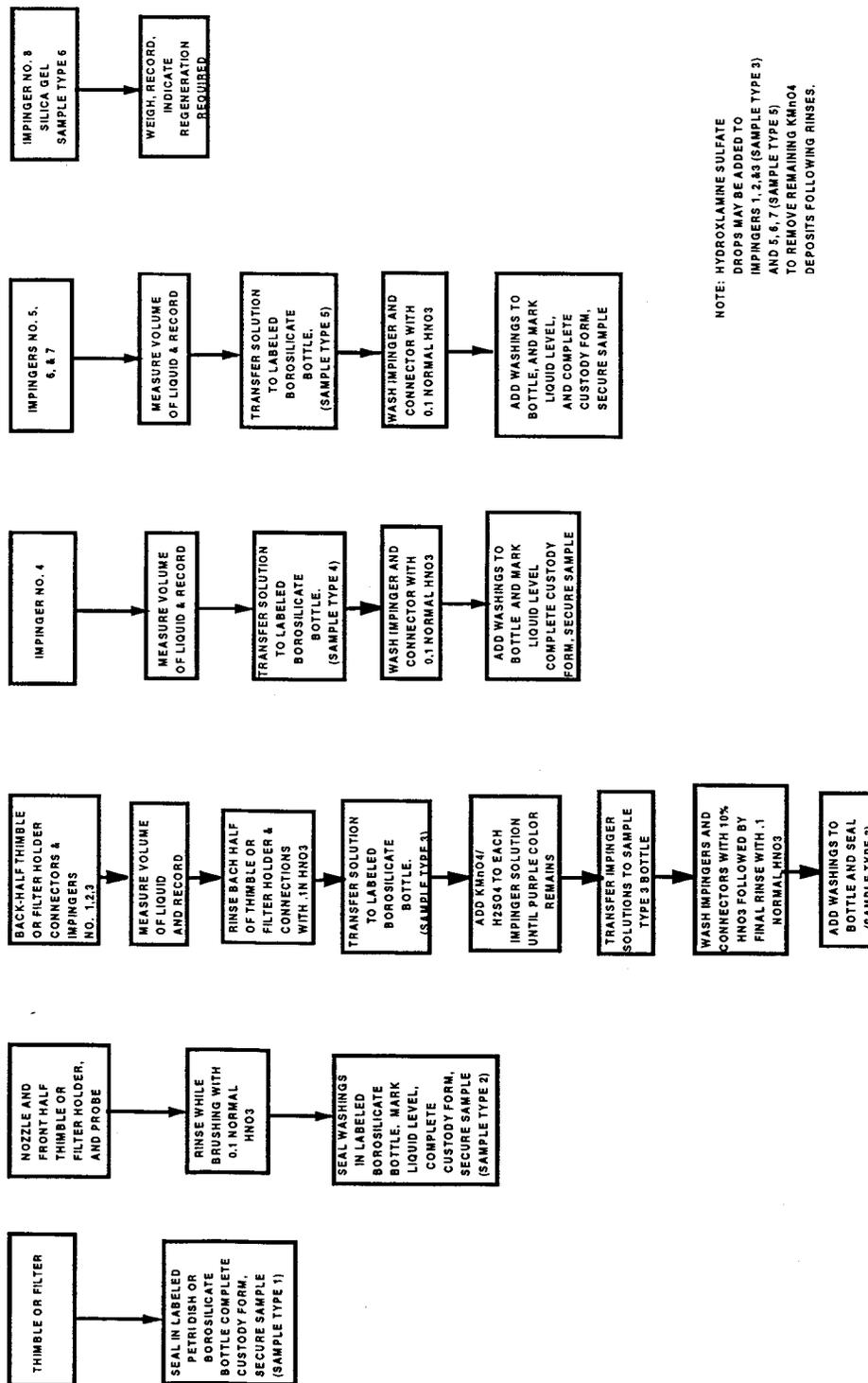
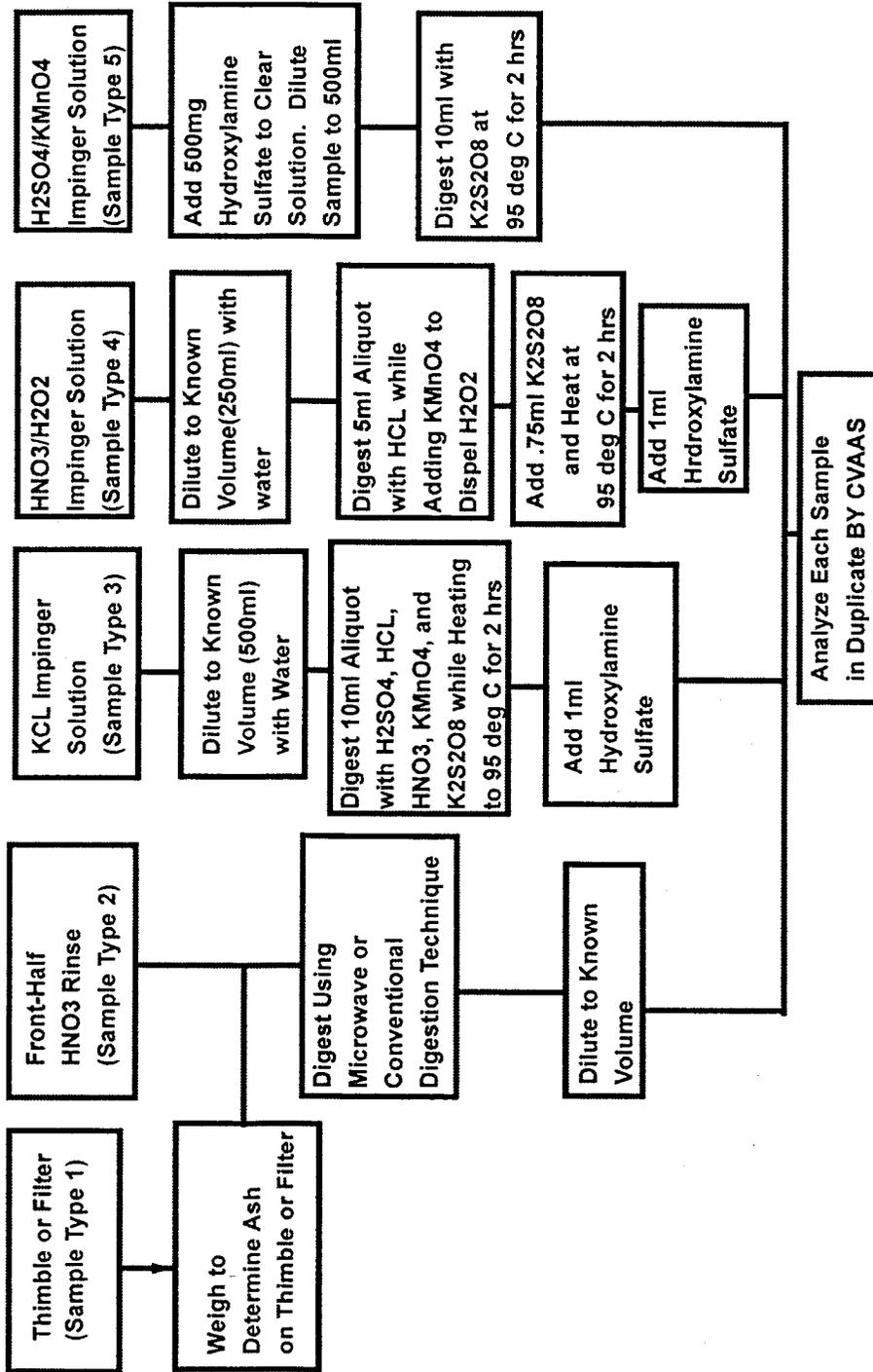


Figure 4-6
Sample Recovery Procedures for Ontario Hydro Method



NOTE: HYDROXYLAMINE SULFATE DROPS MAY BE ADDED TO IMPINGERS 1,2,4,5 (SAMPLE TYPE 3) AND 5, 6, 7 (SAMPLE TYPE 5) TO REMOVE REMAINING KMNO4 DEPOSITS FOLLOWING RINSES.

Figure 4-7
Analytical Procedure for Ontario Hydro Sampling Train



NOTE: Blank Samples (types 7,8,9, 10,11, &12) and Blank Train Samples Will Be Analyzed the same as the Corresponding Source Sample Fraction.

4.4.1.2.3 Mercury

Following preparation the coal sample was weighed. The sample digested in sulfuric acid, nitric acid and potassium permanganate.

Following digestion the liquid sample was analyzed for total mercury content using cold vapor atomic absorption (CVAA) by EPA Method 7471 procedures.

4.4.1.2.4 Ash, Sulfur and Heating Value

The prepared coal samples were analyzed for ash and sulfur content plus heating value using ASTM Methods D3174, D4239 and D3286, respectively.

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 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 No. 2 during the period of 27-28 October 1999. Analyses were performed on these samples for speciated mercury.

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

5.1.1 Stack Sample Collection and Calculations

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

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

Proper field sampling procedures include sampling at 100% isokinetic $\pm 10\%$ and maintaining sample train leakage rates at ≤ 0.02 CFM. Table 5-1 contains a summary of all isokinetic

**Table 5-1
Stack Emission Sampling Field QA/QC Results**

Test Location	Test Run	Isokinetic Sampling Rate ¹	Initial Leak Check Rate ²	Final Leak Check Rate ²	Gas Meter Calibration Values ³	
					Pre	Post ⁽⁴⁾
Unit No. 2 FGD Inlet (ESP outlet)	1	104	0.009	0.006	0.9979	0.9795
	2	100	0.012	0.008	0.9979	0.9890
	3	98	0.008	0.008	0.9979	0.9714
Unit No. 2 ESP Outlet (stack)	1	103	0.019	0.010	0.9961	0.9795
	2	102	0.016	0.014	0.9961	0.9890
	3	104	0.012	0.005	0.9961	0.9714

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

Note: Silica gel impinger exit temperature maintained $< 68^{\circ}\text{F}$ 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 under chain of custody to representatives of Philip Analytical Services Laboratory located in Reading, Pennsylvania. 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 Philip 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 No. 2. Site blanks of the thimbles, filters, impinger train solutions and recovery solutions were retained and analyzed. No mercury above the analytical detection limit was present in any of the site blank samples collected for Unit No. 2.

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 45 days per the method.

5.1.5 Internal Field Audit Procedures

During the performance of the test program, the WESTON field team leader performed an audit of the field measurement activities. A field audit checklist (Technical System Audit) was used to document the internal audit. The audit included examination of field sampling records, field

instrument operating records, sample collection, recovery, handling and chain-of-custody procedures. A copy of the Technical System Audit is provided in Appendix G.

5.1.6 External Performance Evaluation Audits

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

5.1.7 Ontario Hydro Sampling QA/QC Conclusion

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.

5.1.8 Ontario Hydro Sample Analysis

Each Ontario Hydro sample was analyzed in duplicate and every 1 in 10 samples were analyzed in triplicate. The relative percent difference (RPD) for duplicate analysis is $\leq 20\%$. With the exception of a few samples which contained very low levels of mercury near the detection limit, the RPD criteria was satisfied.

The accuracy criteria for spike samples and laboratory control samples is 80 to 120%. This criteria was satisfied in all cases.

5.1.9 Ontario Hydro Sample Analysis QA/QC Conclusion

All source sample data and results 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.

All QA/QC analysis results are provided in Appendix D of this report. A brief summary of the results follows.

Analytical Precision

Analytical precision was determined by RPD obtained by the duplicate sample analyses. The RPD objective for the mercury and chlorine in coal was $\leq 20\%$. The RPD for ash, sulfur and heating value 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 80-120% for chlorine. The objectives for accuracy were satisfied in all cases.

5.2.1 Holding Times

All coal 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.

