



GREAT RIVER  
ENERGY<sup>SM</sup>

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January 28, 2000

Lara P. Autry  
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RE: Speciated Mercury Emissions Data

Dear Ms Autry:

Enclosed with this letter is a copy of the **FINAL REPORT, MERCURY SPECIATION SAMPLING FOR THE ICR AT GREAT RIVER ENERGY, STANTON STATION UNIT 1** and the **FINAL REPORT, MERCURY SPECIATION SAMPLING FOR THE ICR AT GREAT RIVER ENERGY, STANTON STATION UNIT 10**. These documents serve as the final reports for the activities resulting from measuring the speciated mercury emissions at the Great River Energy's Stanton Station Units 1 and 10, as required by the EPA mercury ICR. The documents contain a plant description, sampling location information, unit operating information, descriptions of the sampling and analytical methods, quality assurance/quality control (QA/QC) activities, data from the mercury speciation sampling, and a summary and discussion of results.

Please contact me at 701 745-3387 if you have any questions or comments concerning the enclosed documents.

Sincerely,

Steve Smokey  
Environmental Engineer

Enc.: Unit 1 and Unit 10 reports



**Energy &  
Environmental  
Research  
Center**

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**FINAL REPORT  
MERCURY SPECIATION SAMPLING FOR THE ICR AT GREAT RIVER  
ENERGY STANTON STATION UNIT 10**

**JANUARY 2000**

Prepared for:

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## **ACKNOWLEDGMENT**

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## 1.0 INTRODUCTION

### 1.1 SUMMARY OF TEST PROGRAM

The U.S. Environmental Protection Agency (EPA) has implemented an Information Collection Request (ICR) aimed at characterizing mercury emissions from coal-fired power plants in the United States. As part of this ICR, the operators of selected coal-fired boilers are required to collect and analyze flue gas samples for particulate, elemental, and oxidized mercury. Flue gas samples are to be collected at the inlet of the boiler's last air pollution control device and at the unit stack using the Ontario Hydro (OH) mercury speciation method. Additionally, fuel samples are to be collected and analyzed for mercury and chlorine content.

This document serves as the final report for the activities resulting from measuring the speciated mercury emissions at Great River Energy's Stanton Station Unit 10, as required by the EPA mercury ICR. The document includes a plant description, sampling location information, unit operating information, descriptions of the sampling and analytical methods, quality assurance/quality control (QA/QC) activities, data from the mercury speciation sampling, and a summary and discussion of results.

The test program was performed to meet the requirements of the EPA mercury ICR. EPA selected the test unit based on plant configuration to provide speciated mercury emissions data, which is to be used to develop emission factors for boilers in its class. The intent of the ICR is to provide the EPA with data that can be used to develop speciated mercury emission factors for coal-fired boilers in the United States and to provide information on particulate and SO<sub>2</sub> control device efficiency for collecting mercury. The units to be tested have been selected on the basis of the type of coal burned, type of SO<sub>2</sub> control, and type of particulate control device(s).

As the owner of one of the selected units, Great River Energy, through a tailored collaboration with EPRI, the U.S. Department of Energy (DOE), and the North Dakota Industrial Commission,

contracted the Energy & Environmental Research Center (EERC) to conduct the required tests at its Stanton Station.

Responsible organizations for this project are:

- Test site operator: Great River Energy (Stanton Station)
- Sampling and analytical team: University of North Dakota EERC
- QA/QC oversight: RMB Consulting and Research

The test unit was Stanton Station Unit 10. This unit is operated by Great River Energy and is located near Stanton, North Dakota, in EPA Region 8. The unit was selected by EPA as part of the following category:

- Fuel type: North Dakota lignite
- SO<sub>2</sub> control type: spray dryer
- Particulate control type: baghouse

The Stanton Station consists of two pulverized coal-fired boilers. Unit 10 generates a gross output of approximately 60 MW. The Unit 10 boiler was manufactured by Combustion Engineering and is a wall-fired boiler with a gross heat input of 642 10<sup>6</sup> Btu/hr. It is equipped with a combination of a spray dryer followed by a reverse-gas fabric filter (baghouse) for control of both SO<sub>2</sub> and particulate matter. The coal burned at the Stanton Station is a North Dakota lignite from the Freedom mine.

The dates of the testing were August 23–28, 1999. Measurements using the OH speciation method were done to collect speciated mercury emissions at the inlet of the spray dryer (the combination spray dryer and baghouse were considered one pollution control device for the purposes of the ICR) and at the stack (outlet duct from the baghouse) and fuel mercury and chlorine content. In addition, fly ash samples were collected from the baghouse hopper to verify the concentration of particulate-bound mercury.

The program included the following tests performed during three separate runs. Sampling was performed simultaneously at four sampling locations (inlet to the spray dryer, outlet from the baghouse, coal feed, and baghouse ash) for each run to meet the ICR requirements. Note: the baghouse ash samples were collected to verify the particulate mercury concentration and were not directly required for the ICR. In addition, mercury CEMs (continuous emission monitors) were operated at the stack location. The CEMs were operated to verify the gas-phase mercury concentration at the stack location, but were not directly required for the ICR.

Samples were taken to generate the following data:

- Particulate-bound, oxidized, and elemental mercury emissions at the baghouse exhaust duct using the OH speciation method.
- Particulate-bound, oxidized, and elemental mercury concentrations at the inlet to the spray dryer using the OH mercury speciation method.
- Mercury and chlorine content of representative coal samples collected from the coal feeders simultaneously with the OH mercury speciation method sampling.
- Mercury content of composite fly ash samples collected from the baghouse hoppers simultaneously with the OH mercury speciation method sampling.
- Gas-phase mercury concentrations at the stack location using mercury CEMs concurrently with the OH mercury speciation method sampling.

## **1.2 KEY PERSONNEL**

Table 1-1 lists the test program organization and key individuals with responsibilities, phone numbers, and e-mail addresses. Table 1-2 shows the responsibilities assigned to each position.

The Principal Investigator for the project is Mr. Dennis Laudal from the EERC. He reports directly to Mr. Paul Chu, Project Manager, of EPRI.

**TABLE 1-1**  
**TEST PROGRAM ORGANIZATION AND RESPONSIBILITIES**

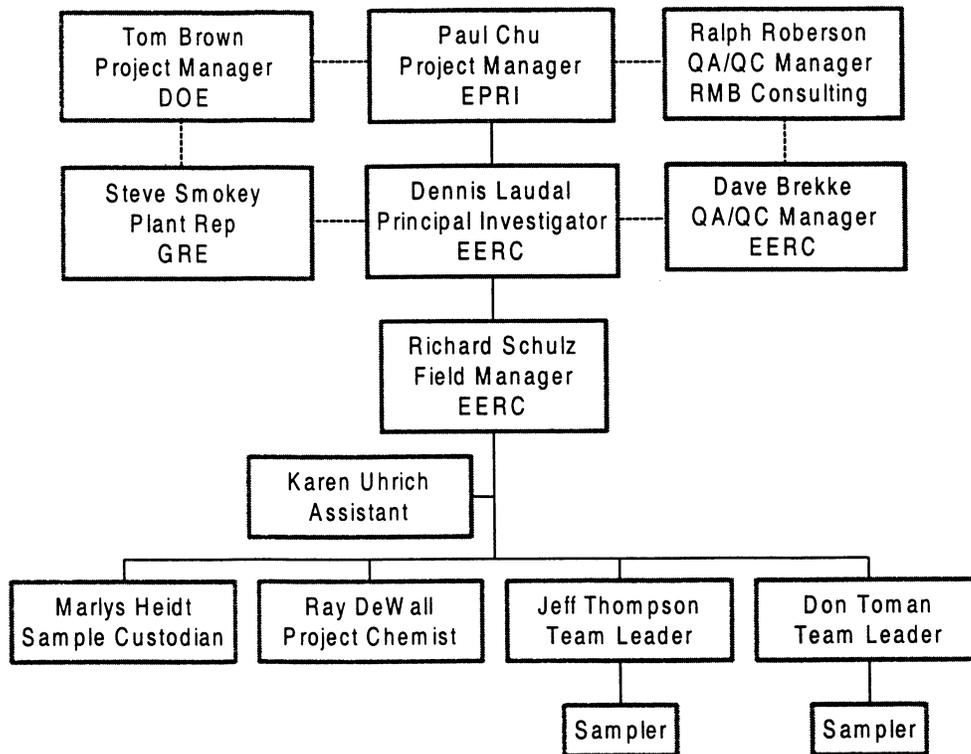
Organization	Individual	Responsibility	Reports To	Phone Number	Fax Number	E-mail Address
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EERC	Don Toman	Team Leader	Richard Schulz	(701) 777-5227	(701) 777-5181	dtoman@eerc.und.nodak.edu
EERC	Craig Eken	Assistant Sampler	Jeff Thompson	(701) 777-5000	(701) 777-5181	---
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EERC	Dave Brekke	EERC OA Manager	EERC Director	(701) 777-5154	(701) 777-5181	dbrekke@eerc.und.nodak.edu

**TABLE 1-2**  
**TEST PERSONNEL AND RESPONSIBILITIES**

<b>Staff Assignment</b>	<b>Responsibilities</b>
1. Project Manager	Maintains contact with EPA as to requirements and provides updates on any new information. Helps to maintain communication between plant representative and test participants. Reviews data and input on all reports. Assists in other activities as required.
2. Principal Investigator	Coordinates all test activities. Maintains communication between all test participants. Maintains custody of data sheets and reduced data. Reviews all data. Prepares necessary reports. Assists in other activities as required.
3. Field Manager	Coordinates or performs all sample train loading and recovery activities. Maintains sample custody records. Ships samples to laboratory. Assists in other activities as required. Also coordinates or performs all sample train recovery and analytical activities. Maintains sample custody records. Transfers custody of samples to on-site laboratory. Assists in other activities as required.
4. Sample Team Leaders	Prepare and operate OH trains. Record and reduce data. Assist in sample recovery and other activities as required.
5. Sampling Assistants	Assist in preparation and operation of OH trains. Assist in sample recovery and other activities as required. •
6. Project Chemist	Performs all analytical activities at on-site laboratory. Maintains sample custody records. Ships samples to off-site laboratory if necessary.
7. Sample Custodian	Maintains sample custody records. Transfers samples to on-site laboratory. Assists in sample recovery and other activities as required.

The Project QA Manager is Mr. Dave Brekke of the EERC, who is also the EERC's QA Manager. Mr. Brekke has no specific project technical assignments other than QA. As Project QA Manager, he was independent and reported only to the Office of the Director at the EERC.

Mr. Ralph Roberson of RMB Consulting and Research provided outside QA services under separate direct contract with EPRI. Activities included review of test plans and procedures as well as provision of blind spike materials for analysis. Mr. Tom Brown from DOE also provided technical review for the project. Figure 1-1 shows the organizational chart.



**Figure 1-1. Project organizational chart.**

## 2.0 SOURCE AND SAMPLING LOCATION DESCRIPTIONS

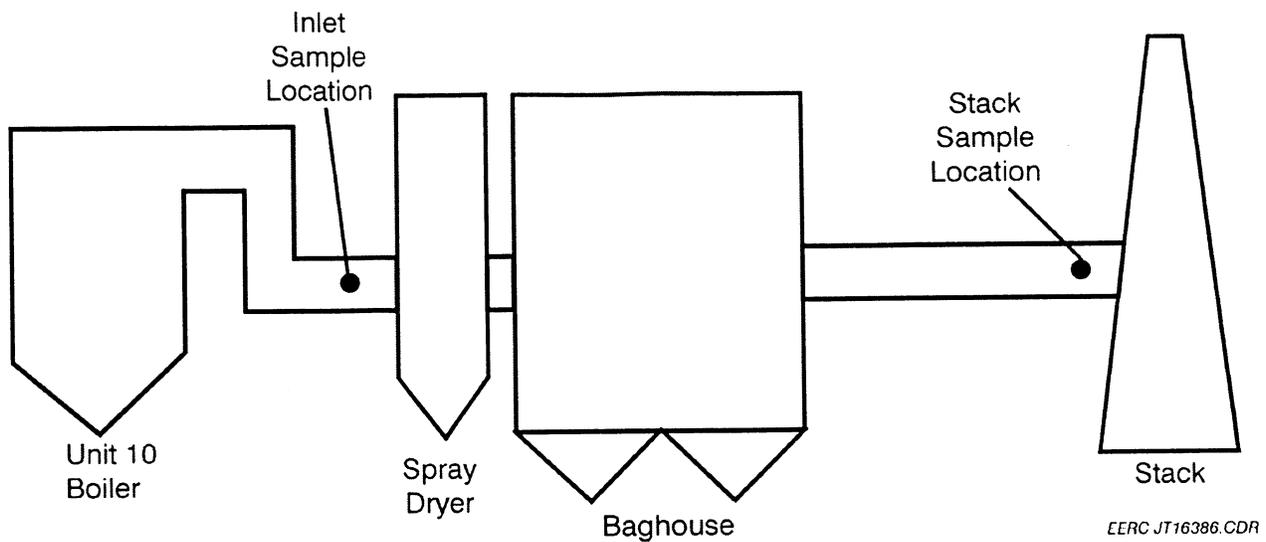
### 2.1 PROCESS DESCRIPTION

The Stanton Station consists of two pulverized coal-fired boilers. Both Units 1 and 10 burn North Dakota lignite to generate steam for electricity production. Unit 10 generates a gross output of approximately 60 MW. The Unit 10 boiler was manufactured by Combustion Engineering and is a wall-fired boiler with a gross heat input of  $642 \times 10^6$  Btu/hr. The flue gas from the Unit 10 boiler is directed to a spray dryer followed by a baghouse for SO<sub>2</sub> and particulate removal before being ducted to the stack. Figure 2-1 shows a schematic of the Unit 10 boiler, spray dryer, baghouse, and stack, including flue gas sampling locations.

Key unit parameters include the following:

- Unit capacity: approximately 60 MW gross
- Boiler type: tangentially fired
- Fuel type: North Dakota lignite
- SO<sub>2</sub> control: spray dryer
- Particulate control: spray dryer/baghouse
- NO<sub>x</sub> control: low-NO<sub>x</sub> burners with overfire air

Fuel samples were collected at the coal feeders ahead of the boiler; inlet samples were collected at the inlet to the spray dryer; and stack samples were collected at the outlet from the baghouse. In addition, ash was collected from the baghouse hopper, and mercury CEMs were operated at the stack location. The stack samples were collected at the baghouse exhaust duct because the stack is common to Units 1 and 10 and there was no way to separate the flows at the stack. Unit operation during testing was at or near nominal full load at steady-state operation. Coal type, boiler operation, spray dryer operation, and baghouse operation were all within normal operating ranges.



**Figure 2-1. Unit 10 schematic.**

## **2.2 CONTROL EQUIPMENT DESCRIPTION**

SO<sub>2</sub> and particulate control for the Unit 10 boiler is accomplished using a combination spray dryer and baghouse. The baghouse is a reverse-gas fabric filter constructed of ten modules with 300 woven fiberglass bags in each module. The air-to-cloth ratio for the baghouse is 1.61 at full capacity.

## **2.3 FLUE GAS AND PROCESS SAMPLING LOCATIONS**

Table 2-1 presents a summary of key inlet and stack sample location parameters. Individual discussions of the sampling locations are presented below.

**TABLE 2-1**  
**SAMPLING LOCATION DESCRIPTIONS**

<b>Description</b>	<b>Spray Dryer Inlet</b>	<b>Baghouse Outlet</b>
Physical Access	Stairs/ladder	Stairs/ladder
Side or Top Access	Top	Side
Round or Rectangular	Rectangular	Rectangular
Number/Type of Ports	12	3
Port Length, ft	1.83	0.83
Port Diameter, ft	0.33	0.33
Inside Dimensions, ft	15 × 5	10 × 8
Equivalent Diameter, ft	7.5	8.9
Nearest Upstream Disturbance	Bend	Bend
Distance, ft	35.8	67
Distance, equivalent diameters	3.4	4.2
Nearest Downstream Disturbance	Bend	Expansion
Distance, ft	7	9
Distance, equivalent diameters	0.9	0.6
<b>Typical Flue Gas Conditions</b>		
Temperature, °F	353	201
Moisture, %	14.6	19.4
Flow rate, scfm	NA	163,000
O <sub>2</sub> , % dry	5.6	6.5
CO <sub>2</sub> , % dry	14.0	12.8
Particulate Concentration, gr/scf	3.2914	0.0047
SO <sub>2</sub> , lb/10 <sup>12</sup> Btu	NA	0.49
NO <sub>x</sub> , lb/10 <sup>12</sup> Btu	NA	0.42

### 2.3.1 Inlet Location

The inlet samples were collected at existing sample ports in the duct at the inlet to the spray dryer. A schematic and cross section of the inlet location are shown in Figure 2-2. The sampling location for the inlet to the spray dryer meets EPA Method 1 criteria. It is located 3.4 equivalent diameters downstream from a bend in the ductwork and 0.9 equivalent diameters upstream from a bend in the ductwork. The sampling ports are located in a horizontal section of steel ductwork that is 5 feet deep and 15 feet wide. Twelve sample ports (A through L) are aligned on the top of the duct 25.8-feet downstream and 7-feet upstream of the nearest gas stream flow disturbances.

Sample traverse points for the inlet location are also shown in Figure 2-2. Six of the twelve ports were used for mercury sampling. 24 traverse points with a 6 x 4 sample grid were used for this duct configuration. The flue gas temperature was 353°F, which is above the method specification of a minimum filtration temperature of 120°C (248°F); therefore, in-stack filtration according to Method 17 was used. This approach is considered to be consistent with the intent and data quality requirements of the ICR.

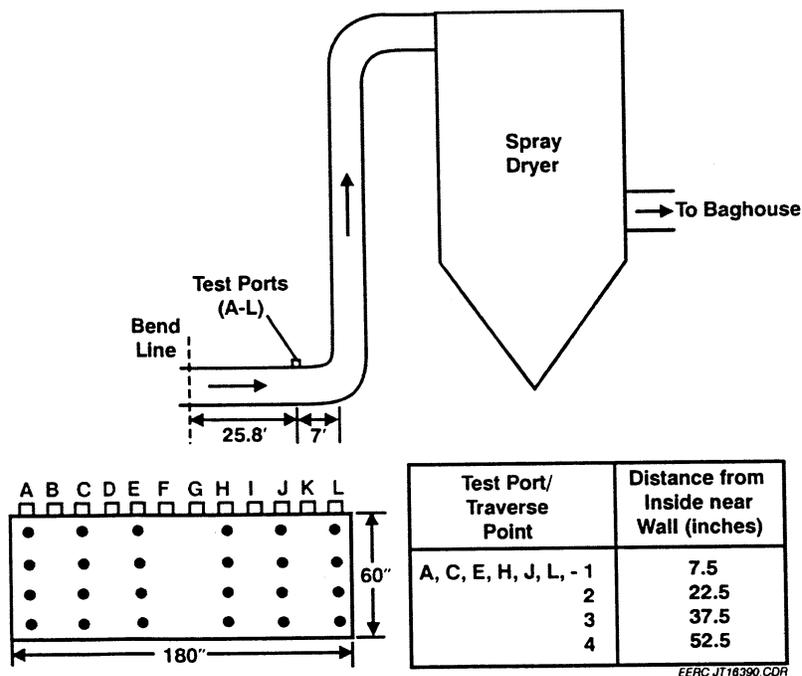


Figure 2-2. Illustration of inlet sampling location.

### 2.3.2 Stack Location

The stack samples were collected at existing sample ports in the outlet duct of the baghouse. A schematic and cross section of the stack location are shown in Figure 2-3. The sampling location for the stack meets EPA Method 1 criteria. The sampling ports are located in a horizontal section of steel ductwork that is 10 feet deep and 8 feet wide. Three sample ports (A through C) are aligned on the side of the duct 67 feet downstream and 9 feet upstream of the nearest gas stream flow disturbances.

A 3 × 8 traverse grid (24 traverse points) was used at the stack location. Sample traverse points for the stack location are illustrated in the schematic (Figure 2-3). The flue gas at the stack was 185°F, which is below the method specification of a minimum filtration temperature of 120°C (248°F); therefore, out-of-stack filtration according to EPA Method 5 was used.

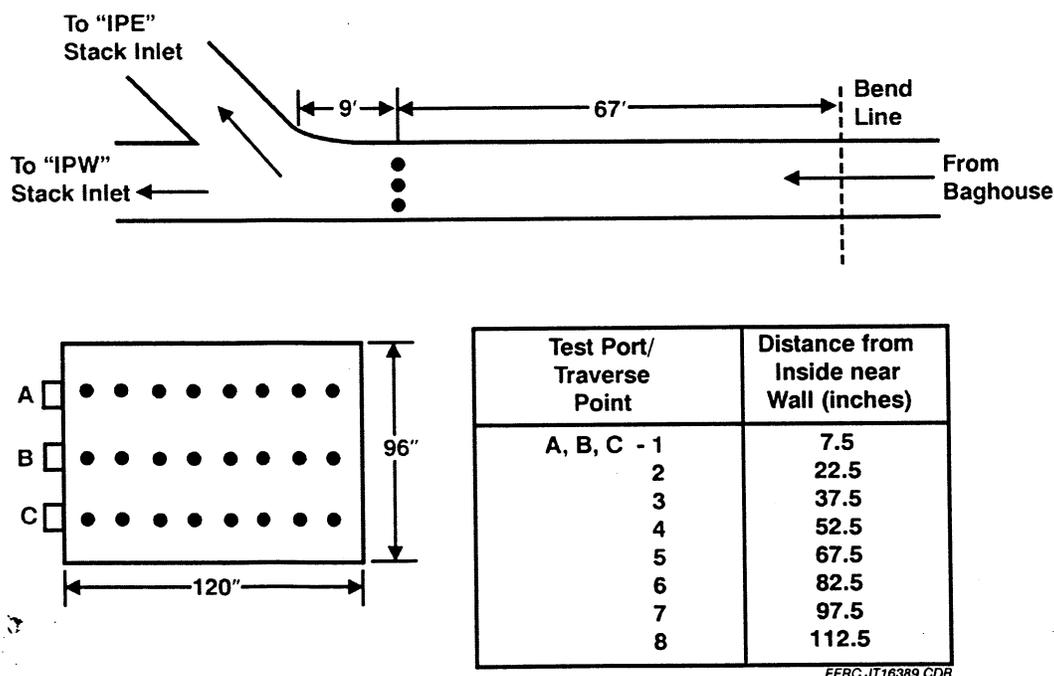


Figure 2-3. Illustration of stack sampling location.

## 2.4 PROCESS SAMPLING LOCATIONS

Coal samples were collected at the coal feeders to each individual mill. These samples were collected during each of the three flue gas sampling periods. In accordance with ASTM procedures, eight individual coal samples (four from each mill) were collected during each 2-hour sampling period. These samples were then composited prior to analysis.

Baghouse hopper ash was collected concurrently with the OH method sampling. The samples were mixed to create a composite sample for each test run.

Auxiliary gas analysis ( $O_2$ ,  $CO_2$ ) was done concurrently with the OH method using an additional gas sampling line in the probe.

A probe was placed in a port near the stack location to sample for the mercury CEMs.

### **3.0 SUMMARY AND DISCUSSION OF RESULTS**

#### **3.1 OBJECTIVES AND TEST MATRIX**

The objective of the program is to collect the information and measurements required by the EPA mercury ICR. Specific objectives are:

- Quantify speciated mercury emissions at the stack (outlet duct from the baghouse).
- Quantify speciated mercury concentrations in the flue gas at the inlet to the spray dryer.
- Quantify fuel mercury and chlorine content during the stack and inlet tests.
- Quantify baghouse hopper ash mercury content during the stack and inlet tests.
- Quantify gas-phase mercury emissions at the stack location using mercury CEMs.
- Provide the above information for use in developing boiler-, fuel-, and control device-specific mercury emission factors.

The test matrix is presented in Table 3-1. The table includes a list of test methods to be used. In addition to speciated mercury, the flue gas measurements will include moisture, stack gas flow, and O<sub>2</sub>/CO<sub>2</sub>. Testing at Great River Energy's Stanton Station Unit 10 was carried out over the 3-day period of August 26–28, 1999. Table 3-2 presents the date and time information for each individual run.

#### **3.2 FIELD TEST CHANGES AND PROBLEMS**

Field testing was carried out according to the test plan in all aspects except sampling times. Sampling times at the inlet location were changed for the fourth run to increase the total volume sampled and isokinetic sampling rate. Each traverse point was sampled for an additional minute for the fourth run. It was decided that the additional time beyond the 2-hour sampling time would not affect the intent of the ICR.

**TABLE 3-1**

**TEST MATRIX FOR MERCURY ICR TESTS AT STANTON STATION UNIT 10**

<b>Sampling Location</b>	<b>No. of Runs</b>	<b>Species Measured</b>	<b>Sampling Method</b>	<b>Sample Run Time</b>	<b>Analytical Method</b>
Stack	3	Speciated Hg	Ontario Hydro	~120 min	Ontario Hydro
Stack	3	Moisture	EPA 4	Concurrent	Gravimetric
Stack	3	Gas Flow	EPA 1/2	Concurrent	Pitot traverse
Stack	3	O <sub>2</sub> /CO <sub>2</sub>	Integrated batch sample	Concurrent	Portable O <sub>2</sub> /plant CEMs
Inlet	3	Speciated Hg	Ontario Hydro	~120 min	Ontario Hydro
Inlet	3	Moisture	EPA 4	Concurrent	Gravimetric
Inlet	3	Gas Flow	EPA 1/2	Concurrent	Pitot traverse
Inlet	3	O <sub>2</sub> /CO <sub>2</sub>	Integrated batch sample	Concurrent	Portable O <sub>2</sub> /plant CEMs
Coal Feeders	3	Hg, Cl in coal	Modified ASTM D2234	One grab sample per mill per run	EPA SW 846: modified 3051 (Hg) ASTM D2361, (Cl)
ESP Ash	3	Hg	Modified ASTM D2234	One composite sample per run	EPA 7473
Stack	3	Gas-phase Hg	CEMs	Concurrent	PS Analytical Sir Galahad, Semtech Hg 2000, Semtech Hg 2010

An error took place during the breakdown of the impinger samples for the second run. The impingers were not weighed. It was because of this that the fourth run was completed. The only data that were not recovered were the moisture values. Since the moisture does not fluctuate much and a small change in moisture value does not significantly affect the results, the data from Run 2 was included in this report. To replace the missing moisture value, 180 g was used as the moisture weight. This value is similar to the value obtained from Run 3, which was completed the same day. This value is italicized in Tables 3-4 and 3-5.

**TABLE 3-2**  
**RUN TIMES FOR MERCURY ICR TESTS AT STANTON STATION UNIT 10**

<b>Run No.</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>
Date	8/26/1999	8/27/1999	8/27/1999	8/28/1999
Starting Clock Time	1455	0938	1430	0908
Sampling Time, min				
Inlet	120.0	120.0	120.0	144.0
Stack	120.0	120.0	120.0	120.0

### 3.3 PRESENTATION OF RESULTS

The results of the testing at Stanton Unit 10 are included in the following subsections:

- Process Data
- Sampling Data
- Flue Gas Mercury Data
- Mercury CEM Data
- Baghouse Hopper Ash Data
- Coal Analysis Data
- Mercury Mass Rates and Removal Efficiencies
- Sample Calculations

### 3.3.1 Process Data

The unit process data were obtained from the plant for the test periods and are summarized in Table 3-3. All data were supplied by the plant. Documentation of these data fulfills the ICR requirements and shows that the unit operation was normal during the sampling activities.

### 3.3.2 Sampling Data

Sampling data for each of the four runs at the inlet and stack (outlet from the baghouse) are summarized in Tables 3-4 and 3-5. The tables also include the resulting calculated values according

**TABLE 3-3  
UNIT PROCESS DATA**

<b>Boiler</b>				
Unit Load, MW net	50	50	50	50
Steam Flow, klb/hr	370	368	375	375
Coal Mills in Service	2	2	2	2
Coal Flow, lb/hr	78,900	79,800	78,900	79,000
Exit Gas Temperature, °F	360	361	350	351
<b>CEM</b>				
CO <sub>2</sub> , %	11.27	11.22	11.34	11.43
SO <sub>2</sub> , lb/10 <sup>12</sup> Btu	0.52	0.48	0.45	0.50
NO <sub>x</sub> , lb/10 <sup>12</sup> Btu	0.42	0.43		0.41
Opacity, %	5.3	5.4	4.6	4.4
Stack Gas Flow, scfm	165,000	162,000	162,000	163,000
<b>Spray Dryer and Fabric Filter</b>				
Gas Inlet Temperature, °F	360	361	350	351
Gas Outlet Temperature, °F	181	181	181	181

**TABLE 3-4**  
**STANTON STATION UNIT 10 INLET SAMPLING DATA**

<b>Run No.</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>
Time, min	120.0	120.0	120.0	144.0
Ts, °F	337	342	368	364
Vm, dcf	40.175	53.990	55.895	54.725
Tm, °F	131	98	117	85
Pm, in. Hg	28.42	28.31	28.40	28.39
Ps, in. Hg	27.73	27.63	27.72	27.78
An, in. <sup>2</sup>	0.0299	0.0377	0.0377	0.0299
SQRT( $\Delta$ P)	0.612	0.757	0.778	0.752
H <sub>2</sub> O, g	120.6	180.0	181.5	176.7
Dust, g	14.33386	9.46975	10.57928	8.33767
CO <sub>2</sub> , %	14.0	13.8	14.1	13.9
O <sub>2</sub> , %	5.5	5.7	5.4	5.6
N <sub>2</sub> + CO, %	80.5	80.5	80.5	80.5
Cm	1.00	1.00	1.00	1.00
Vmc, dcf	40.175	53.990	55.895	54.725
Vm(std), dscf	34.099	48.317	48.559	50.264
Vw(std), scf	5.686	8.487	8.558	8.331
Bws	0.1429	0.1494	0.1498	0.1422
Md, lb/lb-mole	30.5	30.4	30.5	30.4
Ms, lb/lb-mole	28.7	28.6	28.6	28.7
Vs, ft/sec	44.0	54.8	57.1	54.9
I, %	99	91	90	100
<b>Vm*(std), Nm<sup>3</sup></b>	<b>0.832</b>	<b>1.163</b>	<b>1.192</b>	<b>1.218</b>

\* Corrected to 3% O<sub>2</sub>, dry, 68°F, 29.92 in. Hg.

to the appropriate method (EPA Method 2-5). The target sample volume of 1m<sup>3</sup> (35.31 ft<sup>3</sup>) was obtained as well as the isokinetic sampling rate range of 90% to 100%. The calculated sample volume used for subsequent mercury calculations is shown in bold. The sampling volume was corrected to standard conditions (68°F, 29.92 in. Hg, dry, and 3% O<sub>2</sub>).

**TABLE 3-5**  
**STANTON STATION UNIT 10 STACK SAMPLING DATA**

<b>Run No.</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>
Time, min	120.0	120.0	120.0	120.0
Ts, °F	196	203	201	205
Vm, dcf	44.978	41.851	41.159	43.897
Tm, °F	100	85	97	81
Pm, in. Hg	28.14	28.13	28.18	28.30
Ps, in. Hg	28.17	28.17	28.22	28.34
An, in. <sup>2</sup>	0.0299	0.0284	0.0284	0.0284
SQRT ( $\Delta P$ )	0.672	0.680	0.680	0.680
H <sub>2</sub> O, g	204.0	180.0	180.0	183.9
Dust, g	0.02622	0.01182	0.00987	0.00751
CO <sub>2</sub> , %	12.8	13.0	13.0	12.6
O <sub>2</sub> , %	6.5	6.4	6.4	6.8
N <sub>2</sub> + CO, %	80.7	80.6	80.6	80.6
Cm	0.939	0.939	0.939	0.939
Vmc, dcf	42.234	39.298	38.648	41.219
Vm(std), dscf	37.437	35.780	34.492	38.035
Vw(std), scf	9.619	8.487	8.487	8.671
Bws	0.2044	0.1917	0.1975	0.1856
Md, lb/lb-mole	30.3	30.3	30.3	30.3
Ms, lb/lb-mole	27.8	28.0	27.9	28.0
Vs, ft/sec	44.2	44.8	44.7	44.7
I, %	94	93	90	98
<b>Vm*(std), Nm<sup>3</sup></b>	<b>0.854</b>	<b>0.822</b>	<b>0.792</b>	<b>0.850</b>

\* Corrected to 3% O<sub>2</sub>, dry, 68°F, 29.92 in. Hg.

### 3.3.3 Flue Gas Mercury Data

The speciated mercury data for the inlet and stack locations are shown in Tables 3-6 and 3-7. The raw mercury data obtained from the analytical lab are included in the appendices. The raw data were used to calculate the absolute  $\mu\text{g}$  of mercury for each fraction (particulate, oxidized, and elemental) of the sample. The mercury speciation data along with the sample volume data were used to calculate

**TABLE 3-6  
STANTON STATION UNIT 10 INLET MERCURY DATA**

Run No.	1	2	3	4	Average	Std. Dev.
<b>Particulate</b>						
$\mu\text{g}$	0.16	0.27	0.52	0.13		
$\mu\text{g}/\text{Nm}^3$	0.190	0.228	0.435	0.110	0.241 <sup>a</sup>	0.139
$\text{lb}/10^{12}$ Btu	0.163	0.187	0.370	0.091	0.203	0.119
% Total	2.1	2.6	4.7	1.4	2.7	
<b>Oxidized</b>						
$\mu\text{g}$	0.18	0.36	0.72	0.18		
$\mu\text{g}/\text{Nm}^3$	0.21	0.31	0.60	0.14	0.32 <sup>a</sup>	0.20
$\text{lb}/10^{12}$ Btu	0.18	0.25	0.51	0.12	0.27	0.17
% Total	2.3	3.5	6.5	1.9	3.5	
<b>Elemental</b>						
$\mu\text{g}$	7.33	9.75	9.76	8.98		
$\mu\text{g}/\text{Nm}^3$	8.81	8.38	8.19	7.37	8.19	0.61
$\text{lb}/10^{12}$ Btu	7.60	6.87	6.96	6.14	6.89	0.60
% Total	95.7	94.0	88.8	96.7	93.8	
<b>Total</b>						
$\mu\text{g}/\text{Nm}^3$	<b>9.21</b>	<b>8.92</b>	<b>9.22</b>	<b>7.62</b>	<b>8.74</b>	<b>0.76</b>
$\text{lb}/10^{12}$ Btu	<b>7.94</b>	<b>7.31</b>	<b>7.84</b>	<b>6.35</b>	<b>7.36</b>	<b>0.73</b>

<sup>a</sup> The variation of mercury concentration between runs was greater than 25%. Because of the low level of mercury in the particulate (less than 3% of total mercury) and the low level of oxidized mercury (less than 4% of total mercury), this is not considered to affect the results of the ICR speciation data.

**TABLE 3-7**  
**STANTON STATION UNIT 10 STACK MERCURY DATA**

<b>Run No.</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>Average</b>	<b>Std. Dev.</b>
<b>Particulate</b>						
$\mu\text{g}$	0.016	0.0050	0.0090	0.0080		
$\mu\text{g}/\text{Nm}^3$	0.0187	0.0061	0.0114	0.0094	0.0114 <sup>a</sup>	0.0054
lb/10 <sup>12</sup> Btu	0.014	0.005	0.009	0.007	0.008	0.004
% Total	0.2	0.1	0.1	0.1	0.1	
<b>Oxidized</b>						
$\mu\text{g}$	0.27	0.12	<0.02	0.05		
$\mu\text{g}/\text{Nm}^3$	0.32	0.14	<0.01	0.06	0.13 <sup>a</sup>	0.14
lb/10 <sup>12</sup> Btu	0.24	0.10	<0.008	0.04	0.10	0.10
% Total	3.7	1.6	<0.2	0.8	1.5	
<b>Elemental</b>						
$\mu\text{g}$	6.98	7.05	6.95	6.48		
$\mu\text{g}/\text{Nm}^3$	8.17	8.58	8.77	7.62	8.29	0.51
lb/10 <sup>12</sup> Btu	6.12	6.38	6.59	5.56	6.16	0.45
% Total	96.1	98.3	99.9	99.1	98.3	
<b>Total</b>						
$\mu\text{g}/\text{Nm}^3$	<b>8.51</b>	<b>8.72</b>	<b>8.78</b>	<b>7.69</b>	<b>8.43</b>	<b>0.50</b>
lb/10 <sup>12</sup> Btu	<b>6.37</b>	<b>6.49</b>	<b>6.60</b>	<b>5.61</b>	<b>6.27</b>	<b>0.45</b>

<sup>a</sup> The variation of mercury concentration between runs was greater than 25%. Because of the low level of mercury in the particulate (less than 1% of total mercury) and the low level of oxidized mercury (less than 2% of total mercury), this is not considered to affect the results of the ICR speciation data.

the mercury concentration in  $\mu\text{g}/\text{Nm}^3$  for each fraction. The coal feed rate and coal analysis, detailed in Section 3.3.6, were used along with the stack gas flow to calculate the emission rates for mercury in lb/10<sup>12</sup> Btu. Sample calculations are included in Section 3.3.8. The average and standard deviation data are also shown in the tables and show the consistency of the test results. The results show that, typical of lignite coals, the mercury was primarily in elemental form.

### 3.3.4 Mercury CEM Data

Concurrently with the ICR sampling, a total of three mercury CEMs were also used to obtain gas-phase mercury concentration data at the stack location. The instruments used included two Semtech analyzers and a PS Analytical instrument. The data obtained with these instruments verify the results obtained with the OH method. 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 OH method is provided in Figure 3-1.

### 3.3.5 Baghouse Hopper Ash Data

Baghouse hopper ash was collected concurrently with the ICR runs for verification of the particulate mercury concentrations. A composite sample from each run was analyzed for mercury. The results along with the comparative OH data are shown in Table 3-8. The low level of mercury in the particulate was confirmed with these results.

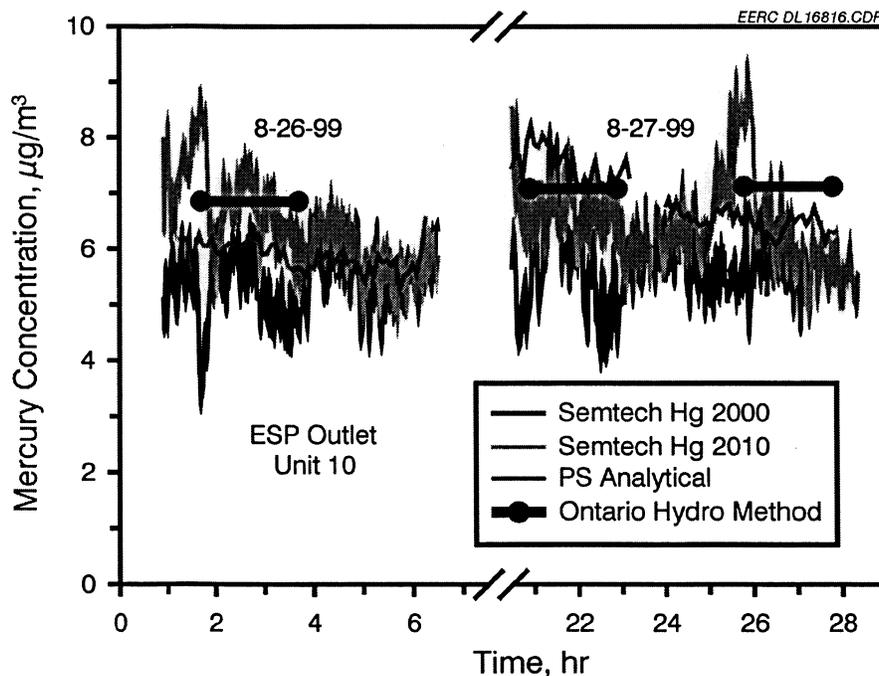


Figure 3-1. Mercury CEM data.

**TABLE 3-8**  
**BAGHOUSE HOPPER ASH DATA**

<b>Run</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>Average</b>
Baghouse Hopper Ash Hg, $\mu\text{g/g}$	0.0055	0.0076	0.0063	0.0057	0.0063
OH Particulate Hg, $\mu\text{g/g}$	0.011	0.028	0.049	0.016	0.026

### 3.3.6 Coal Analysis Data

The composite coal samples, one for each run, were submitted to the lab for mercury, chlorine, heating value, and proximate/ultimate analysis. The results of these analyses are summarized in Table 3-9.

### 3.3.7 Mercury Mass Rates and Removal Efficiencies

Mercury flow rates were calculated for each run, in lb/hr, at each location. The results of these calculations are included in Table 3-10. The removal efficiencies for each species of mercury were also calculated across the spray dryer/baghouse combination. The particulate-bound mercury is shown to be removed (>90%) across the spray dryer/baghouse, but because of the small percentage of mercury in the particulate phase, the percentage of total mercury removed is small (2.3%). Gas-phase mercury was also shown to be removed across the spray dryer/baghouse, with total removal averaging almost 15%.

**TABLE 3-9**  
**COAL ANALYSIS, <sup>1</sup>**

<b>Mercury and Chlorine</b>				
Mercury, ppm (dry)*	0.0865	0.101	0.0630	0.0422
Chlorine, ppm (dry)*	<60	<60	<50	<70
<b>Proximate Analysis</b>				
Moisture, %	36.90	36.80	36.40	37.50
Volatile Matter, %	30.36	28.87	29.65	29.26
Fixed Carbon, %	26.12	27.07	26.08	27.52
Ash, %	6.62	7.26	7.87	5.72
<b>Ultimate Analysis</b>				
Hydrogen, %	6.67	6.56	6.54	6.65
Carbon, %	38.72	38.78	38.48	38.36
Nitrogen, %	0.85	0.87	0.87	0.89
Sulfur, %	0.84	0.82	0.70	0.60
Oxygen, %	46.29	45.71	45.55	47.78
<b>Heating Value</b>				
Btu, Btu/lb	6705	6691	6648	6822

<sup>1</sup> As-received unless otherwise noted (\*).

A summary of the results from this testing with regard to mercury partitioning and emissions are as follows:

- Mercury is primarily in the elemental form (>95%).
- Particulate-bound mercury was removed across the spray dryer/baghouse, but the percentage of total mercury removed was small (2.3% of total).
- Gas-phase mercury was removed across the spray dryer/baghouse, with total removal averaging 14.7%.

**TABLE 3-10**  
**MERCURY MASS RATES AND REMOVAL EFFICIENCY**

<b>Run No.</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>Average</b>	<b>Std. Dev.</b>
<b>Mercury Balance</b>						
Coal Feeder, lb/hr	0.0043	0.0081	0.0050	0.0033	0.0052	0.0020
Inlet, lb/hr	0.0042	0.0039	0.0041	0.0034	0.0039	0.0003
Stack, lb/hr	0.0034	0.0035	0.0035	0.0030	0.0033	0.0002
<b>Removal Efficiency</b>						
<b>Particulate</b>						
Inlet, lb/10 <sup>12</sup> Btu	0.163	0.187	0.370	0.091	0.203	0.119
Stack, lb/10 <sup>12</sup> Btu	0.014	0.005	0.009	0.007	0.008	0.004
Removal, % of total	1.9	2.5	4.6	1.3	2.6	
<b>Oxidized</b>						
Inlet, lb/10 <sup>12</sup> Btu	0.181	0.254	0.510	0.120	0.266	0.172
Stack, lb/10 <sup>12</sup> Btu	0.237	0.104	<0.008	0.043	0.096	0.103
Removal, % of total	-0.7	2.0	6.5	1.2	2.3	
<b>Elemental</b>						
Inlet, lb/10 <sup>12</sup> Btu	7.60	6.87	6.96	6.14	6.89	0.60
Stack, lb/10 <sup>12</sup> Btu	6.12	6.38	6.59	5.56	6.16	0.45
Removal, % of total	18.6	6.8	4.8	9.1	9.8	
<b>Total</b>						
Inlet, lb/10 <sup>12</sup> Btu	7.94	7.31	7.84	6.35	7.36	0.73
Stack, lb/10 <sup>12</sup> Btu	6.37	6.49	6.60	5.61	6.27	0.45
Removal, %	19.8	11.3	15.9	11.7	14.7	

### 3.3.8 Sample Calculations

Sample calculations are included for each of the calculated parameters. Data from the inlet location during Run 1 were used with data from the stack location from Run 1 where necessary.

### 3.3.8.1 Volume of Gas Sample

$V_m(\text{std})$  = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscf

$V_m(\text{std}) (\text{dscf}) = K_1 * V_{mc} * P_m / (T_m + 460)$

$V_m(\text{std}) = 17.64 * 40.175 * 1 * 28.423 / (130.73 + 460) = 34.099 \text{ dscf}$

$V_w(\text{std}) = 0.04715 * 120.6 = 5.686 \text{ scf}$

Where:

$K_1 = 17.64^\circ\text{R/in. Hg}$

$V_{mc} = V_m * C_m =$  volume of gas sample as measured by dry gas meter corrected for meter calibration ( $C_m =$  meter calibration coefficient) (dcf)

$P_m =$  Meter pressure (in. Hg)

$T_m =$  Meter temperature ( $^\circ\text{F}$ )

### 3.3.8.2 Volume of Water Vapor

$V_w(\text{std}) =$  Volume of water vapor in the gas sample, corrected to standard conditions, scf

$V_w(\text{std}) (\text{scf}) = K_2 * H_2\text{O}(\text{g})$

Where:

$K_2 = 0.04715 \text{ ft}^3/\text{g}$

$H_2\text{O}(\text{g}) =$  Mass of liquid collected in impingers and silica gel (g)

### 3.3.8.3 Water Vapor in the Gas Stream

$B_{ws} =$  Water vapor in the gas stream, proportion by volume

$B_{ws} = V_w(\text{std}) / (V_m(\text{std}) + V_w(\text{std}))$

$B_{ws} = 5.686 / (34.099 + 5.686) = 0.1429$

### 3.3.8.4 Dry Molecular Weight

Md = Dry molecular weight of stack gas, lb/lb-mole

$$\text{Md (lb/lb-mole)} = 0.440 * (\% \text{CO}_2) + 0.320 * (\% \text{O}_2) + 0.280 * (\% \text{N}_2 + \% \text{CO})$$

$$\text{Md} = 0.440 * 14.0 + 0.320 * 5.5 + 0.280 * 80.5 = 30.5 \text{ lb/lb-mole}$$

Where:

$\%(\text{CO}_2, \text{O}_2, \text{N}_2, \text{CO})$  = Percent ( $\text{CO}_2, \text{O}_2, \text{N}_2, \text{CO}$ ) by volume, dry basis

### 3.3.8.5 Molecular Weight

Ms = Molecular weight of stack gas, wet basis, lb/lb-mole

$$\text{Ms (lb/lb-mole)} = \text{Md} * (1 - \text{Bws}) + 18.0 * \text{Bws}$$

$$\text{Ms} = 30.5 * (1 - 0.1429) + 18.0 * 0.1429 = 28.7 \text{ lb/lb-mole}$$

### 3.3.8.6 Average Stack Gas Velocity

Vs = Average stack gas velocity, ft/sec

$$\text{Vs (ft/sec)} = K_3 * C_p * (\Delta p)^{1/2} \text{ avg} * [(T_s + 460) / (P_s * M_s)]^{1/2}$$

$$\text{Vs} = 85.49 * 0.84 * 0.612 * [(337 + 460) / (27.73 * 28.7)]^{1/2} = 44.0 \text{ ft/sec}$$

Where:

$$K_3 = 85.49 \text{ ft/sec} [((\text{lb/lb-mole})(\text{in. Hg}) / ((^\circ\text{R})(\text{in. H}_2\text{O})))]^{1/2}$$

Cp = Pitot tube coefficient, dimensionless

$\Delta p$  = Velocity head of stack gas (in. Hg)

Ts = Stack gas temperature ( $^\circ\text{F}$ )

Ps = Stack pressure (in. Hg)

### 3.3.8.7 Isokinetic Sampling Rate

I = Percent of isokinetic sampling, %

$I (\%) = K_4 * (T_s + 460) * V_m(\text{std}) / (P_s * V_s * A_n / 144 * \theta * (1 - B_{ws}))$

$I = 0.09450 * (337 + 460) * 34.099 / (27.73 * 44.0 * 0.0299 / 144 * 120 * (1 - 0.1429)) = 98.6\%$

Where:

$K_4 = 0.09450\% (\text{in. Hg})(\text{min}) / ((^\circ\text{R})(\text{sec}))$

$A_n = \text{Cross-sectional area of nozzle (in.}^2\text{)}$

$\theta = \text{Total sampling time (min)}$

### 3.3.8.8 Volume of Gas Sample Corrected to 3% O<sub>2</sub>

$V_m^*(\text{std}) = \text{Volume of gas sample measured by the dry gas meter (V}_m(\text{std})\text{), * corrected to 3\% oxygen, Nm}^3$

$V_m^*(\text{std}) = V_m(\text{std}) * (21 - \%O_2) / 18 * K_5$

$V_m^*(\text{std}) = 34.099 * (21 - 5.5) / 18 * 0.02832 = 0.832 \text{ Nm}^3$

Where:

$K_5 = 0.02832 \text{ m}^3/\text{ft}^3$

### 3.3.8.9 Mercury

$\text{Hg } (\mu\text{g}) = \mu\text{g/g} * \text{g or } \mu\text{g/L} * \text{mL} / 1000$

$\text{Hg} = 0.35 * 500 / 1000 = 0.175 \mu\text{g}$  (using the values for the oxidized mercury from Run 1)

$\text{Hg } (\mu\text{g/Nm}^3) = \mu\text{g} / V_m^*(\text{std})$

$\text{Hg} = 0.175 / 0.832 = 0.21 \mu\text{g/Nm}^3$

Particulate Hg = Sum of mercury from filter and nozzle rinse (note all nozzle rinse values were nondetects)

Oxidized Hg = Sum of mercury from KCl impingers

Elemental Hg = Sum of mercury from H<sub>2</sub>O<sub>2</sub> and KMnO<sub>4</sub> impingers

### 3.3.8.10 Mass Rates

$$\text{Hg (lb/hr) from coal} = C_f * (1 - B_c) * \text{Hg(ppm)} / 10^6$$

$$\text{Hg (lb/hr) from coal} = 78,900 * (1 - 0.3690) * 0.0865 / 10^6 = 0.0043 \text{ lb/hr}$$

Where:

C<sub>f</sub> = Coal feed rate (lb/hr)

Hg(ppm) = Mercury concentration in coal (ppm, dry)

B<sub>c</sub> = Coal moisture (fraction)

### 3.3.8.11 Emission Rates

$$\text{Hg (lb/10}^{12} \text{ Btu)} = \text{Hg} (\mu\text{g/Nm}^3) * S_f * (1 - B_{ws}) * (21 - \%O_2) / 18 / C_f / H_v * K_6$$

$$\text{Hg (lb/10}^{12} \text{ Btu)} = 9.21 * 165,000 * (1 - 0.1429) * (21 - 5.5) / 18 / 78,900 / 6705 * 3745 = 7.94 \text{ lb/10}^{12} \text{ Btu}$$

Where:

K<sub>6</sub> = 3745 (m<sup>3</sup>/ft<sup>3</sup>)(m/hr)(lb/μg)(Btu/10<sup>12</sup> Btu)

S<sub>f</sub> = Stack gas flow (scfm)

H<sub>v</sub> = Heating value (Btu/lb)

## 4.0 SAMPLING AND ANALYTICAL PROCEDURES

### 4.1 TEST METHODS

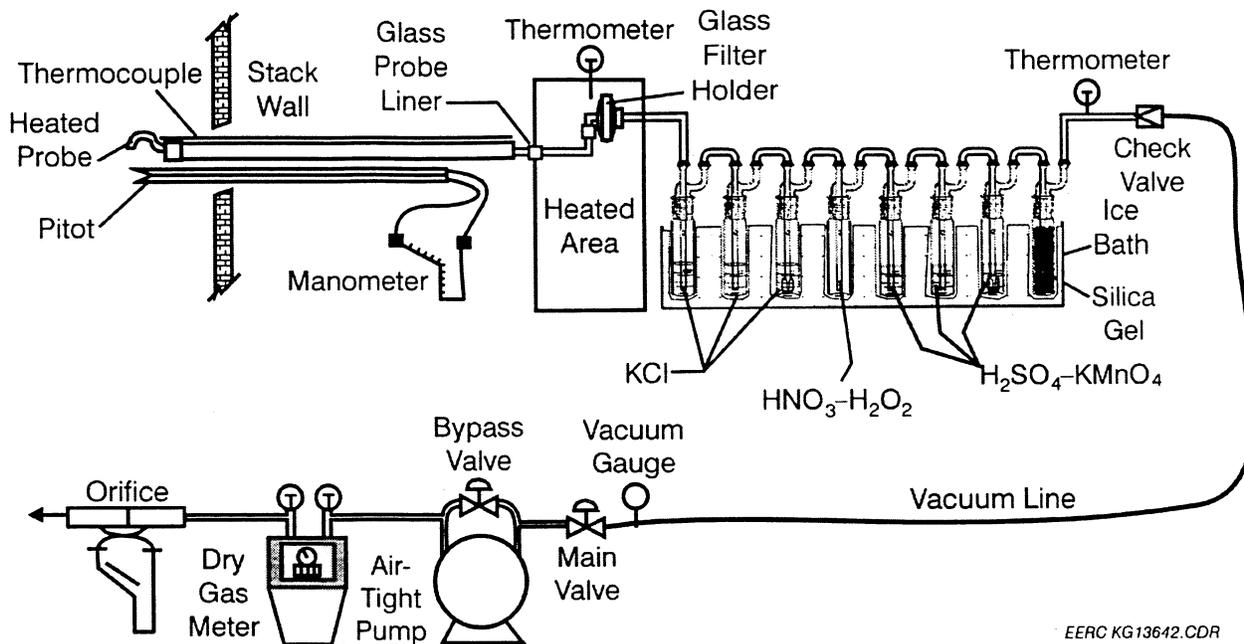
#### 4.1.1 Flue Gas Mercury Speciation

This section contains a summary of the sampling and analytical procedures used to conduct the mercury speciation method required in EPA's ICR entitled *Standard Test Method for Elemental, Oxidized, Particle-Bound and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method)* dated May 1999. For this method, a sample is withdrawn from the flue gas stream isokinetically through the filtration system, which is followed by a series of impingers in an ice bath. Particulate-bound mercury is collected on the front half and filter; oxidized mercury is collected in impingers containing 1 N potassium chloride solution; and elemental mercury is collected in one impinger containing a 5% nitric acid and 10% peroxide solution and in three impingers containing a solution of 10% sulfuric acid and 4% potassium permanganate. An impinger containing silica gel collects any remaining moisture. Quartz fiber filters were used as the filter media for the testing, and the filter holder was glass. A heated Teflon line was used between the probe and impinger train. A target sampling time of 2 hours was used, with a target sample volume of 1 to 2.5 standard cubic meters. A schematic of the sample train is shown in Figure 4-1. Table 4-1 presents a list of sample train components for the EPA Method 17 configuration.

Figure 4-2 is a schematic of the sample recovery procedure for the impinger train. The samples were recovered into precleaned glass bottles with vented Teflon-lined lids for submission to the laboratory. The following sample fractions were recovered (specific rinse solutions are contained in the method):

Container 1 – The sample filter

Container 2 – The front half rinse (includes all surfaces upstream of the filter)



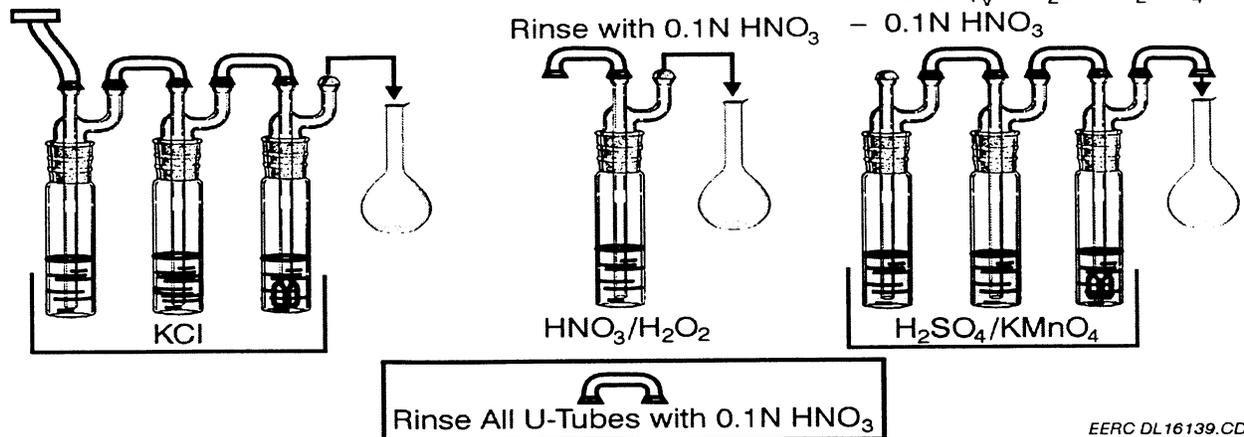
EERC KG13642.CDR

Figure 4-1. Schematic of mercury speciation sample train (Method 5 option is shown; Method 17 in-stack is also allowed).

TABLE 4-1  
SAMPLE TRAIN COMPONENTS – EPA METHOD 17 CONFIGURATION

Component	Details
Nozzle	Quartz
Filter	Quartz, in glass
Probe	Quartz heated to a minimum temperature of 120°C
Connector Line	Teflon line used to connect from probe to impingers. Heated to a minimum of 120°C
Impingers 1 and 2	1 N KCl solution; modified Smith Greenburg (SG) impinger
Impinger 3	1 N KCl solution; standard SG impinger
Impinger 4	5% nitric acid–10% hydrogen peroxide; modified SG impinger
Impingers 5 and 6	10% sulfuric acid–4% potassium permanganate; modified SG impinger
Impinger 7	10% sulfuric acid–4% potassium permanganate; standard SG impinger
Impinger 8	Silica gel; modified SG impinger

1. Rinse filter holder and connector with 0.1N HNO<sub>3</sub>.
2. Add 5% w/v KMnO<sub>4</sub> to each impinger bottle until purple color remains.
3. Rinse with 10% v/v HNO<sub>3</sub>.
4. Rinse with a very small amount of 10% w/v NH<sub>2</sub>OH·H<sub>2</sub>SO<sub>4</sub> if brown residue remains.
5. Final rinse with 10% v/v HNO<sub>3</sub>.



**Figure 4-2. Sample recovery scheme for the mercury speciation sampling train.**

Container 3 – Impingers 1 through 3 (KCl impingers) and rinses

Container 4 – Impinger 4 (HNO<sub>3</sub>-H<sub>2</sub>O<sub>2</sub> impinger) and rinses

Container 5 – Impingers 5 through 7 (H<sub>2</sub>SO<sub>4</sub>-KMnO<sub>4</sub> impingers) and rinse

Silica Gel – Impinger 8 (silica gel impinger). Note that this sample is weighed for moisture determination and is not included in the mercury analysis.

The sample fractions were prepared and analyzed as specified in the method and summarized below:

- *Ash Sample (Containers 1 and 2)* – The particulate catch was digested and analyzed using EPA Method 3051 with subsequent analysis using EPA Method 7471A. When the particulate catch was greater than 1 gram (as was the case at the ESP inlet location), an aliquot of the particulate collected on the filter was analyzed. When the particulate catch was less than 1 gram and an aliquot could not be taken for analysis, the entire filter and particulate catch was digested and analyzed.

- *KCl Impingers (Container 3)* – The impingers were prepared using  $H_2SO_4$ ,  $HNO_3$ , and  $KMnO_4$  solutions as specified in the method.
- *$HNO_3$ - $H_2O_2$  (Container 4)* – The impinger solution was prepared using  $HCl$  and  $KMnO_4$  solutions as specified in the method.
- *$H_2SO_4$ - $KMnO_4$  Impingers (Container 5)* – The impinger solution was prepared using hydroxylamine sulfate as specified in the method.

Each prepared fraction was analyzed for total mercury by cold-vapor atomic absorption (CVAAS). CVAAS is a method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrometer. Mercury concentration is proportional to the indicated absorbance. A soda-lime trap and a magnesium perchlorate trap were used to precondition the gas before it entered the absorption cell.

#### 4.1.2 Fuel Mercury and Chlorine

Mercury in coal was determined by digesting the coal with nitric and hydrochloric acid in sealed high-pressure Teflon digestion vessels similar to EPA Method SW846 3051, Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils. The modifications to the method include 1) the use of larger, high-pressure Teflon vessels, designed specifically for coal; 2) the use of nitric and hydrochloric acid for digestion; and 3) the use of multiple cooldown and venting steps to completely digest the coal. The digested samples were analyzed by CVAAS according to EPA Method 7471A. All values were reported as  $\mu g/g$  Hg on a dry coal basis.

Chlorine was determined by igniting a weighed coal sample in a combustion bomb containing oxygen under pressure in the presence of an alkaline solution, according to ASTM Method D2361, Standard Test Method for Chlorine in Coal. The solution was analyzed by ion chromatography for chloride and was reported as  $\mu g/g$  Cl on a dry coal basis.

### 4.1.3 Auxiliary Flue Gas Measurements

EPA methods for auxiliary flue gas measurements included flue gas flow rate using EPA Methods 1 and 2 (pitot traverse), O<sub>2</sub> and CO<sub>2</sub> by EPA Method 3A (portable O<sub>2</sub> analyzer), and moisture by EPA Method 4 (condensation/gravimetric analysis). These measurements were collected as integral parts of all mercury speciation test runs at both the inlet and stack locations. EPA Reference Method 5/17 requirements were followed for isokinetic sampling.

### 4.1.4 Mercury CEM Measurements

#### 4.1.4.1 *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 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. Before the mercury is desorbed from the gold trap, a flushing step is initiated to remove any flue gas that may be present because it has a damping effect on the mercury fluorescence. When this is completed, the analysis step begins. The heating coil is activated, and the gold trap is heated to approximately 500°C. This desorbs the mercury from the trap, and the mercury is carried into the fluorescence detector. The gold trap is rapidly cooled by pumping argon over it, in preparation for the next sample. The total time for the entire process is about 5 minutes.

The system was calibrated using Hg<sup>0</sup> as the primary standard. The Hg<sup>0</sup> 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.

#### **4.1.4.2 Semtech Hg 2000 and Hg 2010**

The commercial Semtech Hg 2000 and Hg 2010 mercury analyzers (Semtech Metallurgy AB, Lund, Sweden) are essentially portable Zeeman-modulated CVAAS instruments that can monitor Hg<sup>0</sup> continuously. The flue gas was converted and conditioned with a separate unit, and the conditioned dry gas was then analyzed using the Semtech Hg 2000 analyzer. The analyzer uses Zeeman effect background correction, by applying a modulated magnetic field to a mercury lamp, to minimize interferences from the presence of SO<sub>2</sub>, hydrocarbons, and fine particulate in the flue gas sample. The operating range of the analyzer is 0.3 μg/Nm<sup>3</sup> to 20 mg/Nm<sup>3</sup> Hg<sup>0</sup>, 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 μg/Nm<sup>3</sup> for total mercury from waste incinerators.

## **4.2 PROCESS TEST METHODS**

The operational data collected are listed in Table 4-2. To the fullest extent possible, the data were collected using existing plant instrumentation and computerized log printouts. The objective of the process data collection was to ensure and document normal boiler and air pollution control device operation. Prior to and during each test, unit operation was assessed by the sampling team process monitor in conjunction with station personnel to ensure that operating conditions were within project target ranges.

**TABLE 4-2**  
**PROCESS DATA COLLECTED**

<b>Boiler Data</b> Unit Load, MW net Steam Flow, klb/hr Number of Coal Mills in Service Coal Flow, tons/hr Exit Gas Temperature, °F
<b>CEM Data</b> CO <sub>2</sub> , % wet or dry SO <sub>2</sub> , lb/10 <sup>12</sup> Btu NO <sub>x</sub> , lb/10 <sup>12</sup> Btu (record NO <sub>2</sub> if available) Opacity, % Stack Gas Flow, klb/hr
<b>Spray Dryer/Baghouse</b> Gas Inlet Temperature, °F Gas Outlet Temperature, °F

## 5.0 QA/QC ACTIVITIES

Table 5-1 summarizes the Data Quality Objectives and Results for accuracy, precision, and completeness for flue gas mercury analyses (OH samples). Table 5-2 presents the evaluation and verification checklist.

**TABLE 5-1**  
**DATA QUALITY OBJECTIVES AND RESULTS FOR FLUE GAS MERCURY**  
**ANALYSES**

Measure	Activity	Objective	Result
Accuracy	Reagent blanks – one blank per batch of each reagent	<10% of sample value or <10× instrument detection limit	All reagent blanks were less than the detection limit.
Accuracy	Field blanks – one set per location (inlet, stack) per day	<30% of sample value	All field blanks were less than the detection limit.
Accuracy	Blind reagent spikes and certified reference ash sample	±10% of value	All results from spikes and certified reference materials meet the ±10% criteria.
Precision	Triplicate analyses	±10% of mean	All triplicates were within 10% of means.
Completeness	Any failed or incomplete test will be reviewed and, if necessary, repeated.	100% complete	All tests were completed successfully.

TABLE 5-2

RESULTS EVALUATION AND VERIFICATION CHECKLIST

Measure	Objective	Result
<i>Unit Information</i>		
Unit Operating Conditions	No unusual conditions	No unusual conditions
Air Pollution Control Device Operation	No unusual conditions	No unusual conditions
<i>Sample Train Information</i>		
Trains Leak-Checked Before/After Each Test	<0.02 cfm	<0.02 cfm
Pitot Probe Leak-Checked	Zero leakage	Zero leakage
Filter Temperature Maintained	Minimum 120°C	>250°F
Sample Isokinetics	90%–110%	90%–110%
Sample Volume	1 to 2.5 standard cubic meters	1 to 2.5 standard cubic meters
Posttest Color of Permanganate Impingers	Purple	Purple
<i>Results</i>		
Flow Rate for Triplicate Runs	All runs w/in 10% of mean (adjusted for load if necessary)	All runs w/in 10% of mean
Moisture for Triplicate Runs	All runs w/in 3% of mean	All runs at the inlet w/in 3% of mean. The outlet runs had higher and more variable moisture because of the addition of moisture for the operation of the spray dryer. The runs at the outlet were w/in 5% of mean.
Stack Temperature for Triplicate Runs	All runs w/in 5% of mean	All runs w/in 5% of mean
Mercury for Triplicate Runs	All runs w/in 35% of mean	All runs w/in 35% of mean
% of Mercury in as Particulate-Bound	All runs w/in 25% of mean	The low level of mercury in the particulate (less than 3% of total mercury) resulted in greater variation than 25% of mean at both the inlet and the stack location. The particulate data were flagged.
% of Mercury in Oxidized Form	All runs w/in 25% of mean	The low level of oxidized mercury (4% of total mercury) resulted in greater variation than 25% of mean at both the inlet and the stack location. The data were flagged.
% of Mercury in Elemental Form	All runs w/in 25% of mean	All runs w/in 25% of mean

## 5.1 ACCURACY

Three indicators were used for accuracy. A reagent blank was taken from each batch of reagents prepared. The objective was <10% of the sample values or <10 times the instrument detection limit. One field blank was collected at each sample location. The field blank consisted of a sample train that was assembled, taken to the same location as a test sample, and recovered. The quality objective for a field blank was less than 30% of the typical sample values. Two blind reagent spikes and a certified reference ash sample were analyzed as part of the analytical procedure. The blind spikes were provided by RMB Consulting and Research. The objective was  $\pm 10\%$  of the true or certified value. Results for the blind spikes were sent to RMB Consulting and were reported in an audit report. A copy of the report can be obtained from RMB Consulting. Certified reference materials are routinely analyzed by the EERC lab, and results can be obtained upon request. All accuracy criteria were met for this test.

Tables 5-3 and 5-4 show the results for the analysis of reagent blanks and field blanks.

In addition, analytical spikes were completed as an internal check for spike recovery. The results of the spikes are shown in Table 5-5. All recoveries were within  $\pm 15\%$ .

**TABLE 5-3**  
**REAGENT BLANK ANALYSIS RESULTS**

<b>Reagent</b>	<b>Hg, <math>\mu\text{g/L}</math></b>
KCl Reagent Blank	<0.03
H <sub>2</sub> O <sub>2</sub> Reagent Blank	<0.03
KMnO <sub>4</sub> Reagent Blank	<0.03
5% KMnO <sub>4</sub> Blank	<0.03
10% HNO <sub>3</sub> Blank	<0.03
0.1 N HNO <sub>3</sub> Blank	<0.03

**TABLE 5-4**  
**FIELD BLANK ANALYSIS RESULTS**

Sample ID	Sample Type	Volume, mL	Hg, $\mu\text{g/L}$
GRE-U10-D3-FB-IN	KCl	500	<0.03
GRE-U10-D3-FB-IN	H <sub>2</sub> O <sub>2</sub>	250	<0.03
GRE-U10-D3-FB-IN	KMnO <sub>4</sub>	500	<0.03
GRE-U10-D3-FB-OUT	KCl	500	<0.03
GRE-U10-D3-FB-OUT	H <sub>2</sub> O <sub>2</sub>	250	<0.03
GRE-U10-D3-FB-OUT	KMnO <sub>4</sub>	500	<0.03
GRE-U10-D4-FB-IN	KCl	500	<0.03
GRE-U10-D4-FB-IN	H <sub>2</sub> O <sub>2</sub>	250	<0.03
GRE-U10-D4-FB-IN	KMnO <sub>4</sub>	500	<0.03
GRE-U10-D4-FB-OUT	KCl	500	<0.03
GRE-U10-D4-FB-OUT	H <sub>2</sub> O <sub>2</sub>	250	<0.03
GRE-U10-D4-FB-OUT	KMnO <sub>4</sub>	500	<0.03

## 5.2 PRECISION

The precision target for the program was  $\pm 10\%$ . Every tenth sample was analyzed in triplicate. The site-specific test plan called for duplicate analyses of the liquid samples from the OH method for mercury. Because of time limitations in the field and the desire to analyze all of the samples at the site, all of the samples were not analyzed in duplicate in the field. A request for a deviation from the test plan was accepted to eliminate the need for duplicate analyses of all the samples. The precision of mercury analysis using CVAA has been demonstrated and documented using duplicate sample analyses for over 2 years at the EERC. Since the precision of the method has been demonstrated, the samples are no longer routinely analyzed in duplicate. The triplicate analysis of every tenth sample continues to be done to ensure instrument precision. The results of the triplicate analyses for

**TABLE 5-5**  
**ANALYTICAL SPIKES**

Sample ID	Type	Spike Amount, $\mu\text{g/L}$	Sample Value, $\mu\text{g/L}$	Spike Reading, $\mu\text{g/L}$	Recovery, %
GRE-U10-D3-IN-OH-1	KCl	5	0.35	4.97	92.40
GRE-U10-D3-IN-OH-1	KCl	10	0.35	9.04	86.90
GRE-U10-D3-FB-SPIKE	KCl	10	0	9.91	99.10
GRE-U10-D3-FB-SPIKE	KCl	5	9.91	14.42	90.20
GRE-U10-D3-FB-SPIKE	KCl	10	14.42	23.27	88.50
GRE-U10-D3-FB-SPIKE	H <sub>2</sub> O <sub>2</sub>	2	0	1.73	86.46
GRE-U10-D3-FB-SPIKE	H <sub>2</sub> O <sub>2</sub>	5	1.73	6.6	97.40
GRE-U10-D3-FB-SPIKE	H <sub>2</sub> O <sub>2</sub>	10	1.73	11.62	98.90
GRE-U10-D3-IN-OH-1	KMnO <sub>4</sub>	5	15.4	20	92.00
GRE-U10-D3-FB-SPIKE	KMnO <sub>4</sub>	10	0	8.95	89.50
GRE-U10-D3-FB-SPIKE	KMnO <sub>4</sub>	5	8.95	13.29	86.80
GRE-U10-D4-IN-OH-1	KCl	5	0.72	6.06	106.80
GRE-U10-D4-IN-OH-1	KCl	10	0.72	11.01	102.90
GRE-U10-D4-FB-SPIKE	KCl	10	0	9.97	99.70
GRE-U10-D4-FB-SPIKE	KCl	5	9.97	14.98	100.20
GRE-U10-D4-FB-SPIKE	KCl	10	14.98	24.47	94.90
GRE-U10-D4-IN-OH-1	H <sub>2</sub> O <sub>2</sub>	5	0.39	5.67	105.60
GRE-U10-D4-IN-OH-1	H <sub>2</sub> O <sub>2</sub>	10	0.39	11.13	107.40
GRE-U10-D4-FB-SPIKE	KMnO <sub>4</sub>	10	0	9.95	99.50
GRE-U10-D4-FB-SPIKE	KMnO <sub>4</sub>	5	9.95	14.5	91.00

the Stanton Station Unit 10 testing are shown in Table 5-6. In all cases, the data fall within  $\pm 10\%$  of the mean.

**TABLE 5-6**  
**RESULTS FROM TRIPLICATE ANALYSES**

Sample ID	Sample Type	Hg Concentration, $\mu\text{g/L}$		
GRE-U10-D3-IN-OH-1	KCl Nos. 1 and 2	0.34	0.37	0.35
GRE-U10-D3-IN-OH-1	$\text{KMnO}_4$	14.32	14.65	14.47
GRE-U10-D4-IN-OH-1	KCl Nos. 1 and 2	0.71	0.74	0.72
GRE-U10-D4-FB-SPIKE	KCl	10.18	9.88	9.84
GRE-U10-D4-FB-SPIKE	$\text{H}_2\text{O}_2$	1.56	1.43	1.5
GRE-U10-D5-IN-OH-1	KCl Nos. 1 and 2	0.36	0.35	0.35
GRE-U10-D5-IN-OH-1	$\text{H}_2\text{O}_2$	1.3	1.28	1.32
GRE-U10-D5-STACK-OH-1	$\text{KMnO}_4$	12.6	12.5	12.6

### 5.3 COMPLETENESS

All samples were completed and verified by the sampling manager for this test.