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February 18, 2000
FERCo-R744tl

Mr. William Grimley
US EPA
OAQPS TSD EMB, Mail Drop 19
Research Triangle Park, NC 27711

Dear Mr. Grimley:

Enclosed are six bound and one unbound, single-sided copy of the report entitled "Mercury Speciation Stack Sampling Test Report: San Juan Unit 2,", Report No. FERCo R744.

This report is being submitted by FERCo on behalf of Public Service Company of New Mexico to satisfy the requirements of the Information Collection Request.

Please feel free to call me if you have any questions.

Sincerely,
Fossil Energy Research Corp.

Mark D. McDannel

Mark D. McDannel, P.E.

MERCURY SPECIATION STACK SAMPLING TEST REPORT: SAN JUAN UNIT 2

February 2000

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R744 - San Juan 2

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INTRODUCTION

1.1 Summary of Test Program

Purpose of Test

The United States 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 were required to collect and analyze flue gas samples for particulate, elemental, and oxidized mercury.

Public Service Company of New Mexico's (PNM's) San Juan Unit 2 was selected at random by the EPA to provide speciated mercury emissions data, which will then be used to develop emission factors for boilers in its class.

Measurements collected were speciated mercury emissions at the outlet of one of the three operating wet scrubber modules, speciated mercury concentrations at the inlet of the same module, and fuel mercury, chlorine, moisture, sulfur, ash, and heating value.

Test Unit

The test unit is San Juan 2. This unit is operated by Public Service Company of New Mexico, and is located in Waterflow, New Mexico. The unit was selected by the EPA as part of the following category:

- Fuel type: subbituminous
- SO_2 control type: wet scrubber
- Particulate control type: hot side electrostatic precipitator (ESP)

The unit is rated at 350 MW gross. San Juan 2 is a Foster Wheeler front wall-fired boiler, with overfire air for NO_x control. It fires approximately 0.9% sulfur subbituminous coal. SO_2 emissions are controlled by limestone wet scrubbers.

Test Measurements

The program included the following tests, with triplicate sets of measurements performed simultaneously at each test location:

- Particulate, oxidized, and elemental mercury emissions at the outlet of Scrubber Module H per the Ontario Hydro mercury speciation method.
- Particulate, oxidized, and elemental mercury concentrations at the inlet of Scrubber Module H. This location, referred to as the "inlet", is downstream of the hot side electrostatic precipitators and upstream of the wet scrubber.
- Mercury and chlorine content of representative coal samples collected from the coal feeders.
- Coal moisture, sulfur, ash, and heating content.

Responsible Organizations

Responsible organizations for this project are:

- Test site operator: Public Service Company of New Mexico
- Program sponsor: Electric Power Research Institute (EPRI)
- Sampling team: Fossil Energy Research Corp. under contract to EPRI, with Delta Air Quality Services as a major subcontractor
- Sample analysis: Philip Analytical Services (flue gas mercury, coal chlorine), Commercial Testing and Engineering (coal HHV, S, ash, moisture), Frontier Geosciences (coal mercury)

Dates of Test

The test program was conducted on October 20-22, 1999. Daily activities included:

- October 20: set up.
- October 21: conducted Run 1, which was voided, and Run 2.
- October 22: conducted Runs 3 and 4; conducted field blanks.

Document Description

This document is the test report for the San Juan Unit 2 mercury ICR testing. It has been prepared in accordance with Emission Measurement Center Guideline Document GD-043, as required in the ICR.

The work described here is based on the San Juan Unit 2 Test Plan (Report No. FERCo R677), the San Juan Unit 2 Quality Assurance Plan (Report No. FERCo R700), and the San Juan Unit 2 Test Plan Addendum (Report No. FERCo R725). These reports are available from PNM, the EPA or FERCo.

The Test Plan Addendum was prepared in response to initial EPA review of the Test Plan. The Test Plan Addendum was approved by Mr. William Grimley of the EPA. The QA Plan was

approved by Ms. Lara Autry of the EPA prior to testing. EPA comments on the draft QA Plan were incorporated into the final version of the QA Plan.

1.2 Key Personnel

Table 1-1 lists the test program organization and key individuals with responsibilities, phone numbers, and e-mail addresses. A program organizational chart is shown in Figure 1-1.

The program was jointly funded by PNM and EPRI. FERCo was under contract to EPRI. The Project Quality Assurance Officer was Greg Quartucy of FERCo, who reported directly to Larry Muzio, FERCo's Vice President. External QA activities were performed by Dennis Laudal of UNDEERC. Mr. Laudal reported directly to Paul Chu of EPRI. Both UNDEERC and FERCo are contractors to EPRI. The reporting function from Mr. Laudal to Mr. Chu is considered to be external to FERCo's project.

Mr. Farley, Mr. McDannel, and Ms. Bell were all on-site for the testing. There were no observers from regulatory agencies.

Table 1-1. Test Program Organization and Responsibilities

Organization	Individual	Responsibility	Reports To	Phone Number	Fax Number	E-mail Address
Project Management and Oversight						
Electric Power Research Institute	Paul Chu	EPRI Project Manager	N/A	(650) 855-2812	(650) 855-2619	pchu@epri.com
FERCo	Lawrence Muzio	Vice President	N/A	(949) 859-4466	(949) 859-7916	lmuzio@ferco.com
FERCo	Greg Quartucy	QA Manager	Lawrence Muzio	(949) 859-4466	(949) 859-7916	gquartucy@ferco.com
Host Utility						
PNM	Michael Farley	Program Coordinator and Site Contact	N/A	(505) 598-7628	(505) 598-6036	mfarley@mail.pnm.com
FERCo/Delta Sampling Team						
FERCo	Mark McDannel	Program Manager	Paul Chu	(949) 859-4466	(949) 859-7916	mmcdannel@ferco.com
Delta	Arlene Bell	Project Chemist	Mark McDannel	(714) 279-6777	(714) 279-6781	deltaaqs@aol.com
Philip Environmental	Ron McLeod	Sample Analyses	Mark McDannel	(905) 332-8788	(905) 332-9169	rmcleod@philipinc.com
External QA/QC						
UNDEERC	Dennis Laudal	External QA/QC	Paul Chu	(701) 777-5138	(701) 777-5181	dlaudal@eerc.und.nodak.edu

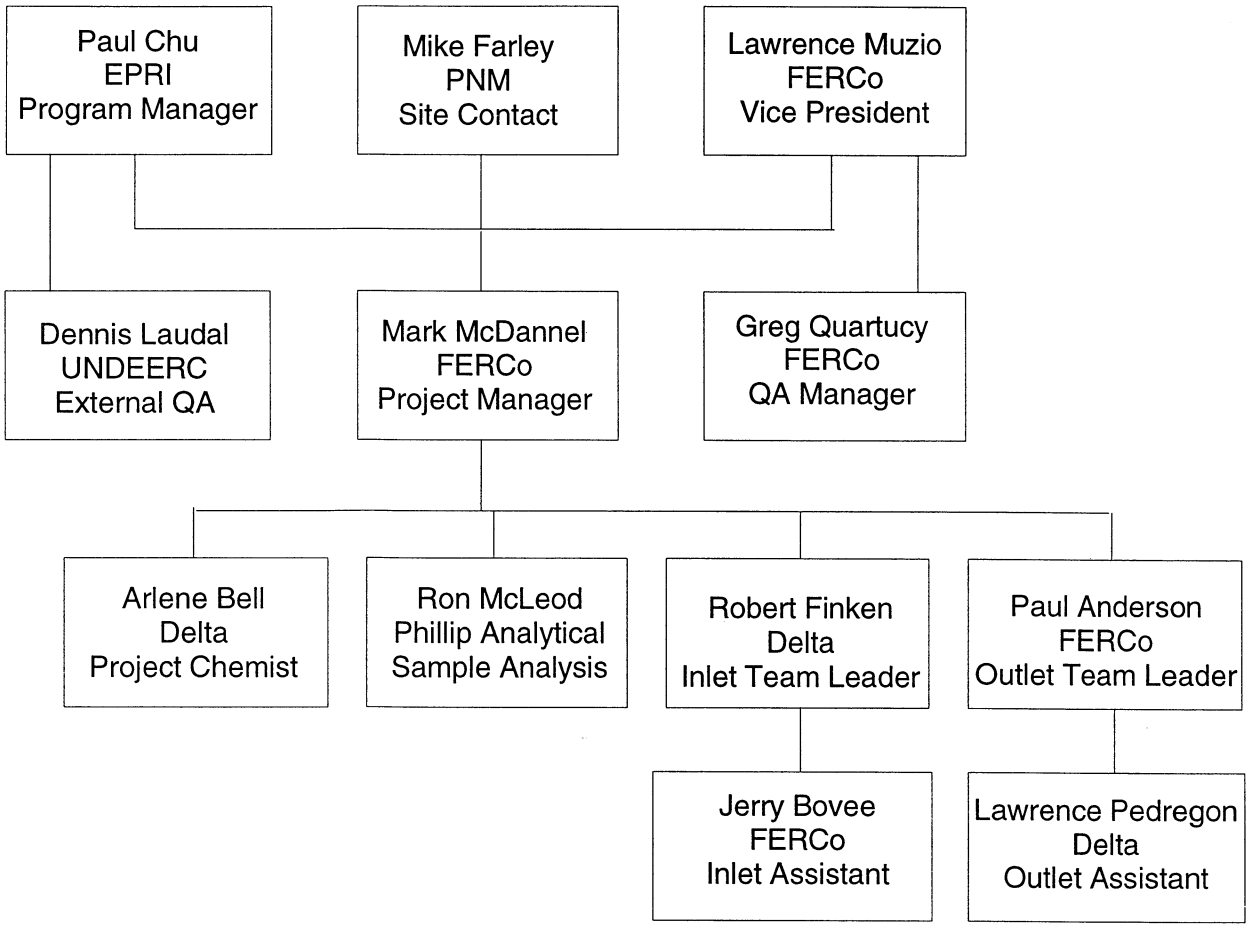

Figure 1-1. Project Organization Chart

2

PLANT AND SAMPLING LOCATION DESCRIPTIONS

2.1 Process and Control Equipment Description and Operation

San Juan 2 is an opposed-fired Babcock & Wilcox boiler rated at 350 MW gross. Figure 2-1 shows a schematic of the boiler and pollution control equipment, including sample points.

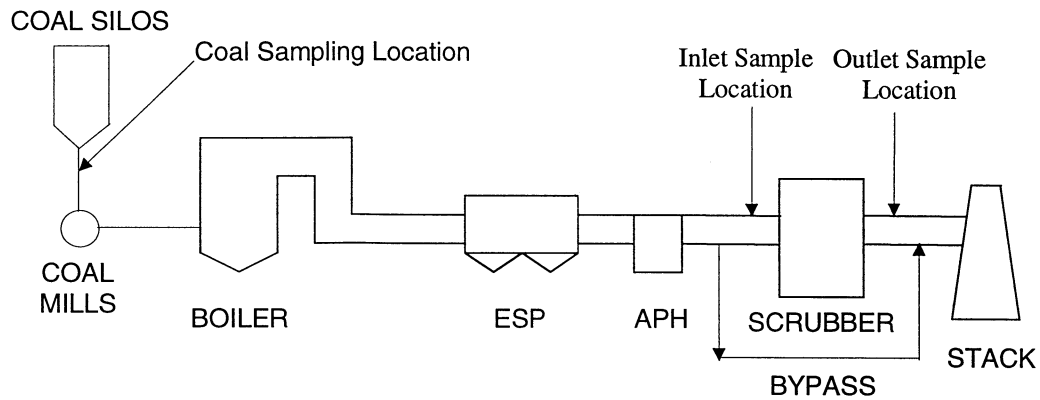

Figure 2-1. San Juan Unit 2 Schematic

Key unit parameters include:

- Unit capacity: 350 MW gross
- Boiler type: Foster Wheeler, front wall-fired, balanced draft
- Fuel type: subbituminous, 0.85 – 0.95% S
- SO_2 control: limestone wet scrubber. There are four scrubber modules, designated E, F, G, and H. Module G is inoperable. Approximately 20% of the flue gas is bypassed around the scrubbers. Module SO_2 removal efficiency is 90%, and net unit removal efficiency with bypass is 75%.
- Particulate control: hot side ESP, 99.7% efficiency
- NO_x control: overfire air and combustion control

Fuel samples were collected at the coal feeders ahead of the boiler, inlet samples were collected at the inlet to scrubber module H, and outlet samples were collected at the outlet of scrubber module H.

The sample gas at the inlet was approximately 275-295°F. At the outlet the gas temperature was approximately 120°F, and the gas was saturated with moisture.

Unit operation during testing was at or near nominal full load, at steady state operation. Coal type, boiler operation, and control device operation were all within normal operating ranges. All three operating scrubber modules were balanced with each other, with the following operating ranges for key parameters:

- pH 5.0-6.0
- blower amps 200-400
- solids 15-22%
- sump level 60-70%

Table 2-1 presents a summary of unit operation during the tests. Additional detailed unit data is included in Appendix G.

2.2 Flue Gas Sampling Locations

Table 2-2 presents a summary of key inlet and stack sample location parameters. Individual discussions of the two locations are presented below. Note that the duct dimensions presented here are different than those presented in the test plan, as the ducts were remeasured on site. The new measurements are discussed further in Section 3.2.

Inlet Locations

The inlet samples were collected at the inlet to the H scrubber module on San Juan 2. Drawings of this location are shown in Figures 2-2a and 2-2b. The flue gas exiting the air preheater splits into inlet ducts to the various modules, travels downward to the scrubber booster fan, and then upward to the absorber module. The sample ports are located in the fan inlet duct, 20 feet downstream and 10 feet upstream of the nearest flow disturbances.

The sample traverse scheme for San Juan 2 Module H inlet was:

6 ports x 5 points/port x 6 minutes/point = 150 minutes.

This location meets the requirements of EPA Method 1. A cyclonic flow check was done before testing. The yaw angle was 0 degrees at all points.

Table 2-1. Summary of San Juan Unit 2 Operation

	Run 2	Run 3	Run 4
Date, 1999	21-Oct	22-Oct	22-Oct
Start time	1305	0805	1214
Stop time	1821	1117	1700
Unit load, MW	350	339	344
Coal mills in service	All 4	All 4	All 4
Coal flow, klb/hr	369	333	345
Boiler O_2, A/B	3.1/2.6	2.5/2.0	2.2/2.1
CEMS data			
O_2, % dry	6.33	6.06	5.63
SO_2, lb/MMBtu	0.43	0.35	0.37
NO_x, lb/MMBtu	0.51	0.48	0.45
Opacity, %	9	10	10
Stack flow, kwscfh	882	840	832
Stack temperature, F	150	147	152
Stack Moisture	12.7	13.0	13.1
ESP data			
Power level, kW	222	No Data	No Data
Sections in service	32		
Sections out of service	0		
Scrubber data			
pH			
E Cell	5.53	5.56	5.59
F Cell	5.10	5.10	5.11
H Cell	5.00	5.00	5.01
Blower amps			
E Cell	303	296	292
F Cell	304	313	306
H Cell	301	296	289
Feed solids, %			
E Cell	21	21	21
F Cell	17	17	17
H Cell	19	16	17
Sump level			
E Cell	68	70	69
F Cell	67	68	68
H Cell	68	69	68

Table 2-2. San Juan Unit 2 Sampling Location Descriptions

	Inlet	Outlet
Description	Module H Inlet Duct	Module H Outlet Duct
Elevation	Approximately 50'	Approximately 90'
Physical access	Stairs, ladder	Elevator, stairs
Side or top access	Side	Side
Round or rectangular	Rectangular	Rectangular
Port length (outside of port to inner stack wall)	18"	18"
Number/type of ports	Six 4-inch w/caps	Four 4-inch w/flanges
Inside dimensions	14' 2" deep x 12' 2" wide Equivalent diameter 13.1 ft	10' 0 deep x 13' 0" wide Equivalent diameter 11.3 ft
Nearest upstream disturbance		
Disturbance	Right angle turn	Isolation dampers
Distance, ft	20'	2' 8"
Distance, diameters	1.5	0.24
Nearest downstream disturbance		
Disturbance	30 deg jog in duct	Right angle turn
Distance, ft	10'	30'
Distance, diameters	0.7	2.7

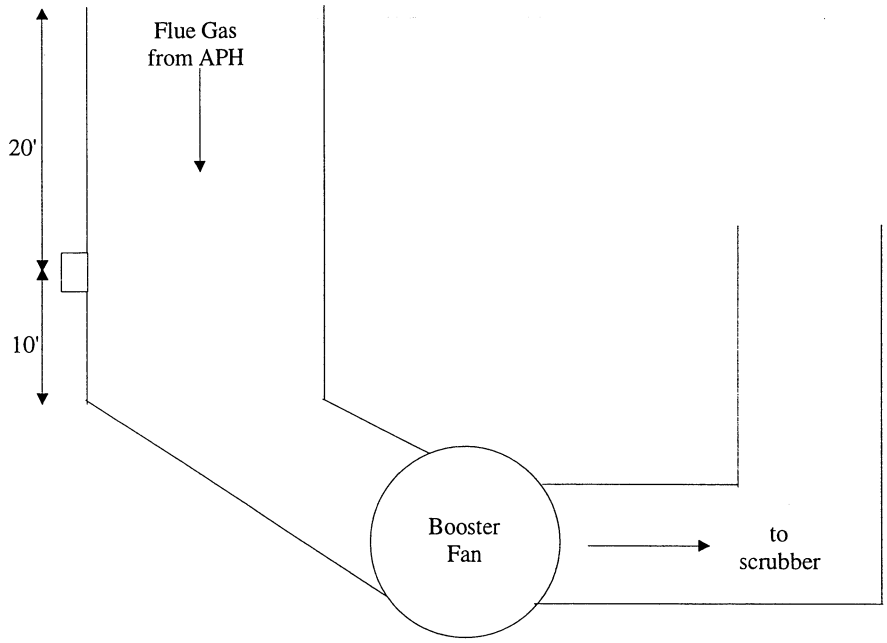

Figure 2-2a. San Juan 2 Inlet Sampling Location

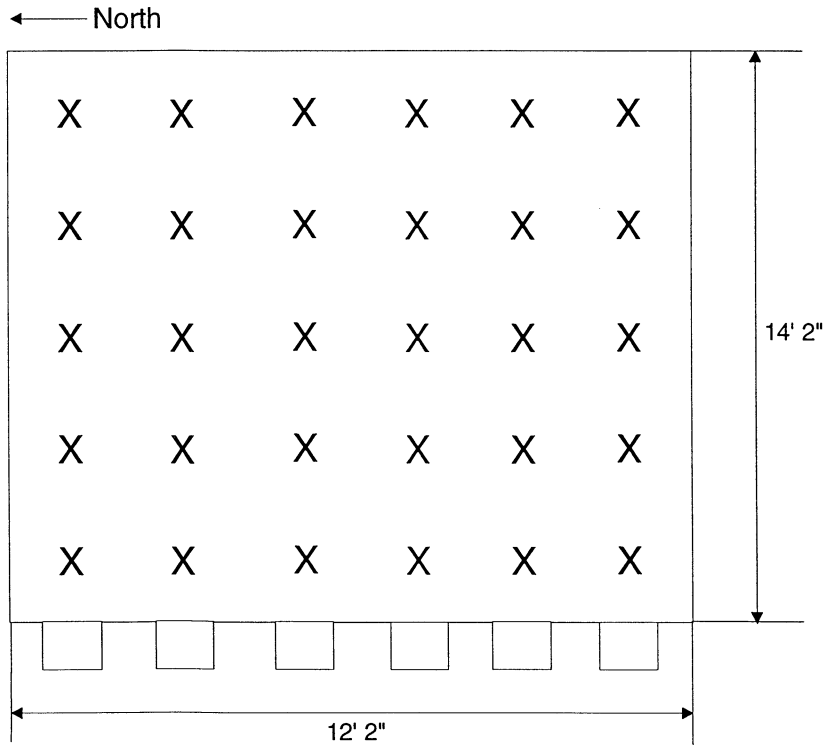

Figure 2-2b. San Juan 2 Inlet Sampling Location

Outlet Location

Because some of the boiler flue gas is bypassed around the FGD system on San Juan 2, it is not possible to directly measure both stack emissions and scrubber removal efficiency. Sampling at the stack provides a direct measure of emissions but removal efficiency must be calculated, while the converse is true for sampling at the scrubber outlet.

For San Juan Unit 2, sampling was performed at the outlet because there are accessible outlet sample ports which allow direct measurement of scrubber removal. Calculation procedures to determine control device efficiency and stack emissions are presented in Section 4.1.

The outlet samples were collected at the ports at the outlet of Module H. A schematic and cross section of the outlet location is shown in Figure 2-3.

The sample traverse scheme for San Juan Unit 2 Module H outlet was:

4 ports x 6 points/port x 6 minutes/point = 144 minutes.

This location does not meet the requirements of EPA Method 1. A cyclonic flow check was performed before testing, and the average yaw angle was 10 degrees with all but one traverse point between 8 and 12 degrees.

2.3 Coal Sampling Location

Coal samples were collected from the silo just above the coal feeders to each individual mill. A one pint scoop was collected from each operating mill during the first and last hour of each test and placed in a bucket. Following testing the bucket of coal was riffled to blend the samples and reduce the amount of coal sent to the laboratories. Samples were collected and riffled by Mark McDannel of FERCo.

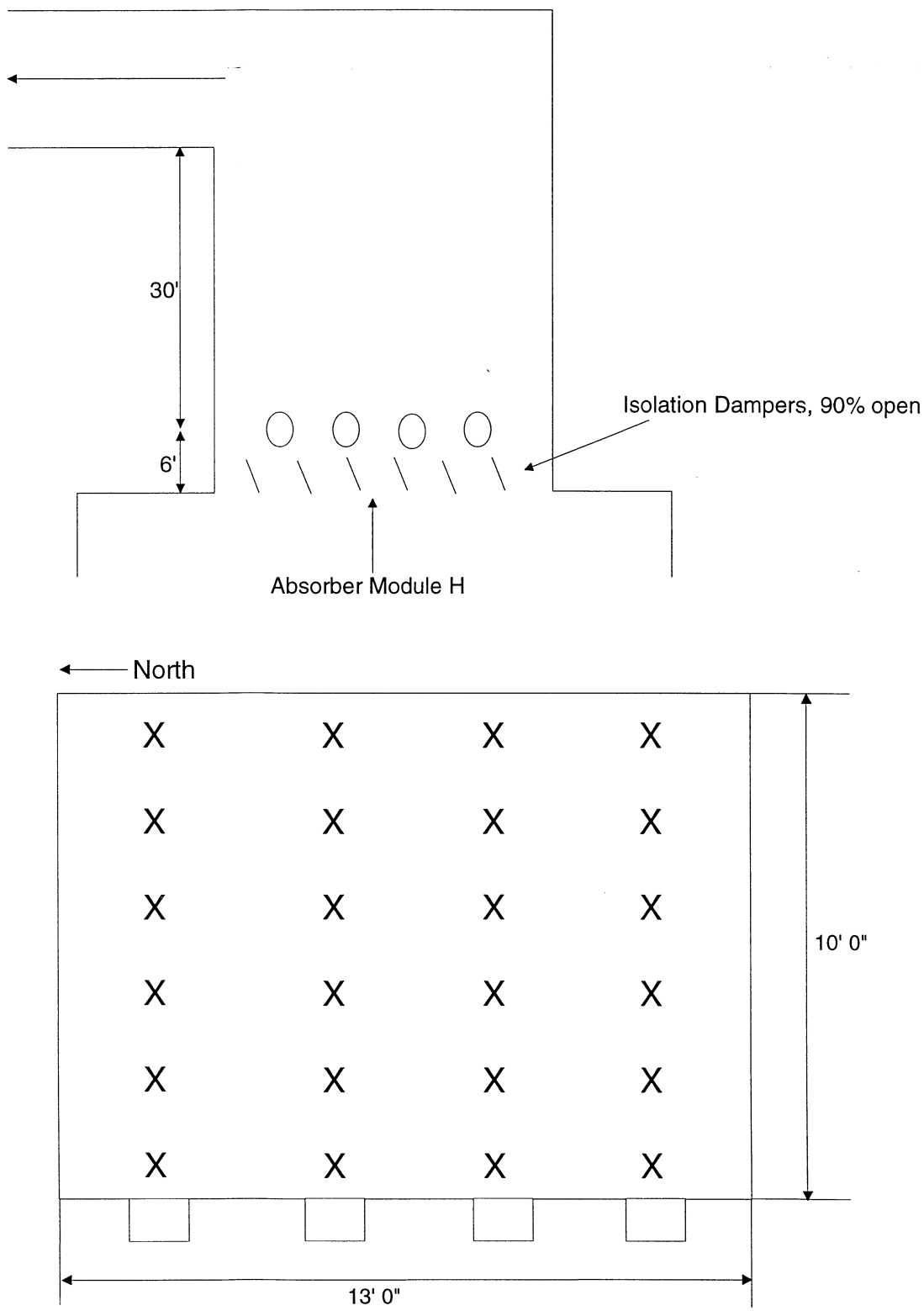

Figure 2-3. San Juan 2 Outlet Sampling Location

3

SUMMARY AND DISCUSSION OF TEST RESULTS

3.1 Objectives and Test Matrix

Objectives

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

- Quantify speciated mercury concentrations at the scrubber outlet, and estimate speciated mercury emissions at the stack.
- Quantify speciated mercury concentrations in the flue gas at the scrubber inlet.
- Quantify fuel mercury and chlorine content during the outlet and inlet tests.
- Provide the above information for use in developing boiler-, fuel-, and control device-specific mercury emission factors.

Test Matrix

The test matrix is presented in Table 3-1, and actual test times are shown in Table 3-2. Table 3-1 includes a list of test methods used. In addition to speciated mercury, the flue gas measurements included moisture, stack gas flow, and O_2/CO_2.

3.2 Field Test Changes and Problems

Outlet Duct Width

During set up the width of the outlet duct was remeasured and found to be 13'0" rather than 12'6" as stated in the test plan. The duct depth was confirmed as 10'0".

Inlet Duct Depth, Void Run 1

After Run 1 was completed and shortly after Run 2 was started, the duct dimensions on the inlet duct were remeasured and found to be significantly different than the dimensions presented in the test plan (14'2" deep by 12'2" wide vs. 10'0" deep by 12'6" wide). This difference was significant enough that Run 1 was voided, and Run 2 was restarted with a fresh sample train and a 16' probe rather than a 10' probe.

This change out took approximately 2 ½ hours. During that time sampling was suspended at the outlet, where one sample port had been traversed. After the inlet test was restarted and one sample port had been sampled, the outlet test was restarted.

Run 3 Outlet Filter Heater Failure

After the start of Run 3-Outlet, it became difficult to maintain the minimum filter temperature of 248°F (temperatures were running 195-210°F). At the end of the first port traverse, sampling was suspended and troubleshooting was performed. A failed heater element was diagnosed. Since the probe heater had been working properly (probe temperature 262-298°F) and there was no visible water or acid condensation at the filter outlet, it was decided that the test was not compromised. The sample train was leak checked, a new filter holder and filter were installed, the sample train was leak checked again, and the test was restarted.

As a check to ensure that there was no unintended collection of mercury on the filter due to the low temperature, the first filter was recovered and analyzed separately. There was no particulate mercury detected on the filter used before correction of the heater problem, indicating that no gaseous mercury was unintentionally collected on the filter. Therefore, it is concluded that this discrepancy had no impact on the results.

Run 4 Delay

At 1214 the inlet and outlet sample trains for Run 4 appeared close to starting, so the outlet test was started. After the outlet test was started, problems were experienced in final leak checking of the inlet train. When it became apparent that the problems would not be resolved immediately, outlet sampling was suspended after three points were sampled. When the inlet problems were resolved, Run 4-Outlet was resumed after a delay of 90 minutes.

Change of Analytical Method and Laboratory for Mercury in Coal

The test plan called for coal mercury analysis to be performed by Philip Analytical, using EPA SW 846. However, the result for one of the three samples was not detected less than 0.04 ppm, and the other two samples were 0.06 and 0.055 ppm.

In an effort to achieve lower detection limits and more precisely quantify mercury in the coal, splits of the samples were analyzed by Frontier Geosciences. The samples were digested by cold aqua regia (modified EPA 7371) and analyzed by cold vapor atomic fluorescence (modified EPA 1631). This approach provides a detection limit of 0.002 ppm. These methods provided detectable levels of mercury in the coal for all three samples, and are used as the reported mercury values.

Table 3-1. Test Matrix for Mercury ICR Tests at San Juan 2

Sampling Location	No. of Runs	Species Measured	Sampling Method	Sample Run Time	Analytical Method	Analytical Laboratory
Outlet	3	Speciated Hg	Ontario Hydro	120 min	Ontario Hydro	Philip Services
Outlet	3	Moisture	EPA 4	Concurrent	Gravimetric, compared with saturation value	FERCo
Outlet	3	Gas Flow	EPA ½	Concurrent	Pitot Traverse	FERCo
Outlet	3	O_2	Batch Sample	Concurrent	Portable O_2	FERCo
Outlet	3	CO_2	N/A	Concurrent	Stoichiometric calculation	FERCo
Inlet	3	Speciated Hg	Ontario Hydro	125 min	Ontario Hydro	Philip Services
Inlet	3	Moisture	EPA 4	Concurrent	Gravimetric	FERCo
Inlet	3	Gas Flow	EPA ½	Concurrent	Pitot Traverse	FERCo
Inlet	3	O_2	Batch Sample	Concurrent	Portable O_2	FERCo
Inlet	3	CO_2	N/A	Concurrent	Stoichiometric calculation	FERCo
Coal Feeders	3	Cl in coal	Modified ASTM D2234	1 grab sample per coal feeder per run	EPA SW 846: 5050/9056 (Cl)	Philip
Coal Feeders	3	HHV, Ash, S, Moisture	Modified ASTM D2234	1 grab sample per mill per run	ASTM D514290	CTE
Coal Feeders	3	Hg in coal	Modified ASTM D2234	1 grab sample per mill per run	Modified EPA 7371/1631	Frontier Geosciences

Table 3-2. San Juan Unit 2 Sampling Times

	Run 2	Run 3	Run 4
Date, 1999	21-Oct	22-Oct	22-Oct
Inlet Tests			
Start time	1454	0815	1403
Stop time	1821	1117	1700
Total sample time, min	150	150	150
Outlet Tests			
Start time	1305	0805	1214
Stop time	1729	1104	1620
Total sample time, min	144	144	144
Notes:			
1. Gas flow, moisture, and O_2 were concurrent with mercury tests.			
2. Coal samples were collected during the first and last hour of each run.			
3. Sampling suspended on Run 2-Outlet from 1341 to 1535 to wait on 2-Inlet restart.			
4. Sampling suspended on Run 4-Outlet from 1232 to 1404 to wait on 4-Inlet start.			

3.3 Presentation of Results

The test results are presented in the following tables and figure:

- Table 3-3. Sample gas conditions.
- Table 3-4. Mercury concentration and speciation results.
- Table 3-5. Mercury removal across scrubber by species and estimated stack concentrations.
- Figure 3-1. Mercury speciation across scrubber.

Results are calculated as µg/scm (at a reference temperature of 68°F), and normalized for dilution by converting to a lb/10^{12} Btu basis. This method allows direct comparison of inlet and outlet results without incorporating uncertainties involved in gas flow measurement.

Major observations that can be made from the results are:

1. Particulate mercury was negligible, and gaseous mercury was roughly equally split between oxidized and elemental at the inlet (0% particulate, 52% oxidized and 48% elemental), while at the outlet it was almost all elemental mercury (1% particulate, 6% oxidized, 93% elemental).

Table 3-3. San Juan Unit 2 Sample Gas Conditions

	Run 2	Run 3	Run 4	Average
Test Date	21-Oct	22-Oct	22-Oct	
Module H Inlet Gas Properties				
Temperature, F	294	278	296	289
Gas flow, dscfm from pitot traverse	198,078	185,927	168,354	184,120
Calculated flow, dscfm*	205,276	188,857	189,227	194,491
O_2, %	5.61	5.32	4.63	5.19
CO_2, %	13.46	13.72	14.33	13.84
H_2O, %	8.96	8.37	9.41	8.91
Module H Outlet Gas Properties				
Temperature, F	118	118	119	118
Gas flow, dscfm from pitot traverse	262,205	237,051	222,400	240,552
Calculated flow, dscfm*	205,276	188,857	189,227	194,491
O_2, %	5.80	5.70	5.16	5.55
CO_2, %	13.30	13.39	13.86	13.51
H_2O, %	14.42	13.73	14.54	14.23
Stack Gas Conditions and Bypass Calculations				
CEMS Stack Gas Flow, dscfm	770,492	730,595	722,776	741,288
Stack Gas Flow, Calculated from Fuel	811,989	791,562	755,755	786,435
Bypass Flow, by Heat Balance	20%	22%	21%	21%

* Note: calculated flow is based on stack CEMS flow and bypass fraction, assuming equal gas flow to all three scrubbers.

Table 3-4. San Juan Unit 2 Mercury Speciation Results

	Run 2	Run 3	Run 4	Average
Test Date	21-Oct	22-Oct	22-Oct	
Inlet Mercury Speciation				
Particulate mercury				
ug/dscm	ND<0.03	0.07	ND<0.03	ND<0.03
lb/10^{12} Btu	ND<0.03	0.06	ND<0.04	ND<0.04
% of total Hg	0%	1%	0%	0%
Oxidized mercury				
ug/dscm	5.34	2.88	4.61	4.28
lb/10^{12} Btu	4.45	2.35	3.61	3.47
% of total Hg	52%	43%	58%	52%
Elemental mercury				
ug/dscm	4.97	3.71	3.29	3.99
lb/10^{12} Btu	4.14	3.03	2.57	3.25
% of total Hg	48%	56%	42%	48%
Total mercury				
ug/dscm	10.32	6.66	7.90	8.29
lb/10^{12} Btu	8.59	5.44	6.18	6.74
Outlet Mercury Speciation				
Particulate mercury				
ug/dscm	0.041	0.071	0.048	0.053
lb/10^{12} Btu	0.035	0.060	0.039	0.044
% of total Hg	0.6%	1.6%	1.1%	1.0%
Oxidized mercury				
ug/dscm	0.38	0.32	0.27	0.32
lb/10^{12} Btu	0.32	0.27	0.22	0.27
% of total Hg	6%	7%	6%	6%
Elemental mercury				
ug/dscm	6.03	4.07	4.10	4.73
lb/10^{12} Btu	5.09	3.41	3.32	3.94
% of total Hg	93%	91%	93%	93%
Total mercury				
ug/dscm	6.45	4.46	4.42	5.11
lb/10^{12} Btu	5.44	3.74	3.58	4.25
Coal Analysis				
Mercury, ppm dry	0.045	0.051	0.065	0.054
Mercury, lb/10^{12} Btu	4.47	4.63	6.31	5.14
Chlorine, ppm dry	200	100	200	167
Moisture, %	5.71	5.35	5.17	5.4
Sulfur, % dry	0.67	0.73	0.82	0.74
Ash, % dry	29.97	23.78	27.55	27.1
HHV, Btu/lb as fired	9,406	10,361	9,819	9,862
Coal flow, lb/hr as fired	369,250	332,800	345,000	349,017
Total Mercury Mass Rates				
lb/hr input in coal	0.016	0.016	0.021	0.018
lb/hr at Module H inlet	0.008	0.005	0.005	0.006
lb/hr at Module H outlet	0.006	0.004	0.004	0.005
estimated total inlet lb/hr	0.030	0.018	0.021	0.023
estimated total stack lb/hr	0.021	0.014	0.014	0.016

Table 3-5. San Juan Unit 2 Mercury Removal Efficiency and Estimated Stack Concentrations

	Run 2	Run 3	Run 4	Average
Date, 1999	21-Oct	22-Oct	22-Oct	
Total mercury				
Inlet, lb/10^{12} Btu	8.59	5.44	6.18	6.74
Outlet, lb/10^{12} Btu	5.44	3.74	3.58	4.25
Removal efficiency, %	37%	31%	42%	37%
Stack, lb/10^{12} Btu	6.07	4.12	4.14	4.78
Particulate mercury				
Inlet, lb/10^{12} Btu	ND<0.03	0.06	ND<0.04	ND<0.04
Outlet, lb/10^{12} Btu	0.03	0.06	0.04	0.04
Removal efficiency, %	N/A	1%	N/A	N/A
Stack, lb/10^{12} Btu	0.03	0.06	0.04	0.04
Oxidized mercury				
Inlet, lb/10^{12} Btu	4.45	2.35	3.61	3.47
Outlet, lb/10^{12} Btu	0.32	0.27	0.22	0.27
Removal efficiency, %	93%	89%	94%	92%
Stack, lb/10^{12} Btu	1.15	0.73	0.95	0.95
Elemental mercury				
Inlet, lb/10^{12} Btu	4.14	3.03	2.57	3.25
Outlet, lb/10^{12} Btu	5.09	3.41	3.32	3.94
Removal efficiency, %	-23%	-12%	-29%	-21%
Stack, lb/10^{12} Btu	4.90	3.33	3.16	3.79
Note: Outlet measurements and removal efficiencies are direct measurements.				
Stack values are calculated from inlet/outlet concentrations and inlet/outlet/stack flows.				

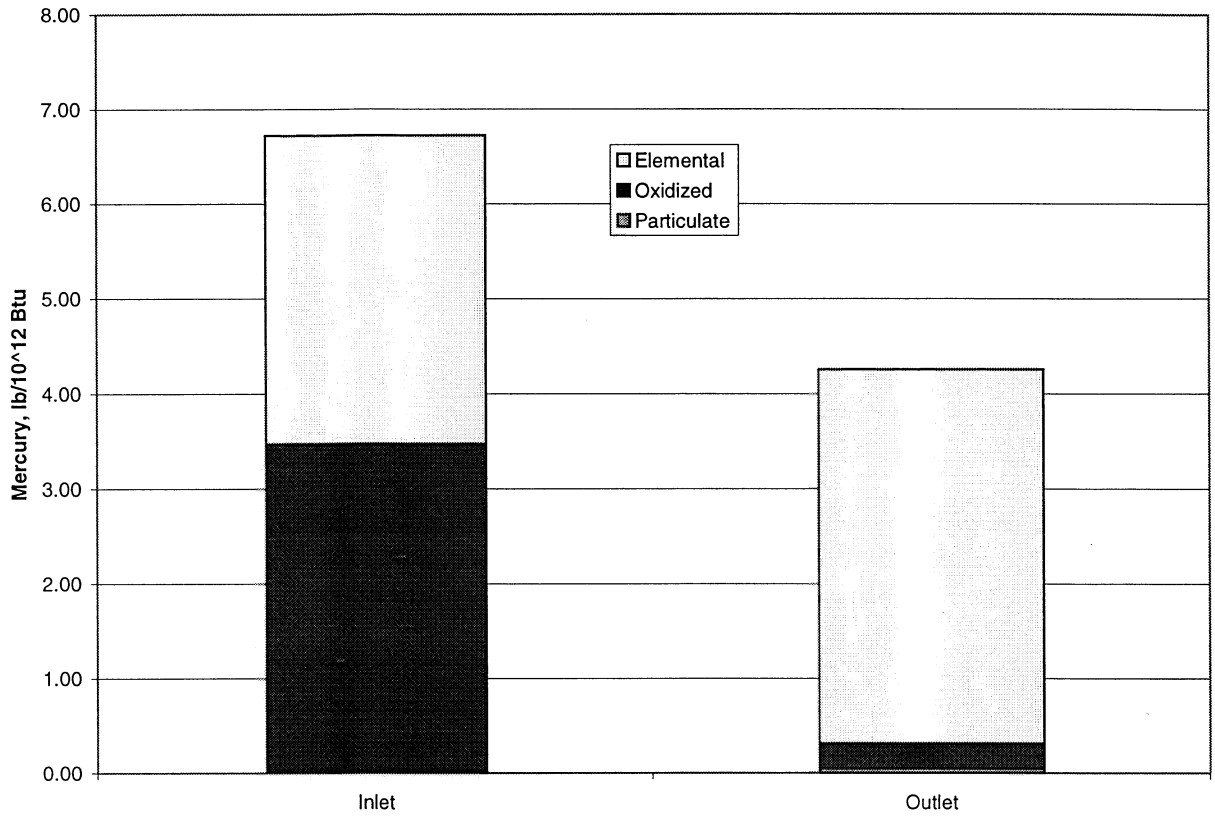

Figure 3-1. Mercury Speciation Across San Juan Unit 2 Scrubber

2. Mercury levels in the coal were 5.1 lb/10^{12} Btu, or 0.054 ppm. This level is 24% less than the mercury level of 6.7 lb/10^{12} Btu measured at the inlet. This difference is considered to be within the uncertainties of the test methods.
3. Oxidized mercury was removed with 92% efficiency across the scrubber.
4. Elemental mercury increased by 21% across the scrubber, and particulate mercury increased from ND<0.03 lb/10^{12} Btu at the inlet to 0.04 lb/10^{12} Btu at the outlet. These differences are on the same order of magnitude as the uncertainties in the measurement methods.

4

SAMPLING AND ANALYTICAL PROCEDURES

4.1 Test Methods

This section contains a summary of the sampling and analytical procedures used to conduct the mercury speciation required in EPA's ICR titled, "Standard Test Method for Elemental, Oxidized, Particle-Bound and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method)" dated April 8, 1999. The full text of the method was presented as Appendix A of the Test Plan.

Subsequent to submittal of the Test Plan, additional drafts of the Ontario Hydro Method were published. Wherever possible, the new features of these drafts were incorporated into the program.

Speciated mercury samples were collected in three test runs at the inlet and outlet of the control device. The inlet and outlet sampling were concurrent. A field blank was collected at each test location on October 19. The field blank consisted of assembling a sample train, transporting it to the sample location, conducting a leak check, letting the train sit for two to three hours, and then recovering the train as if it were a sample.

EPA methods to determine flue gas flow rate were used. EPA Reference Method 5 and 17 requirements for isokinetic sampling were followed. Each impinger was weighed before and after sampling to determine flue gas moisture content.

Figure 4-1 presents a schematic of the mercury speciation sample train, Table 4-1 presents a list of sample train components for the Method 17 configuration, and Table 4-2 presents a list of sample train components for the Method 5 configuration. The sampling train was set up with in-stack filtration (EPA Method 17 configuration) for the inlet location and external heated filtration (EPA Method 5 configuration) for the stack location.

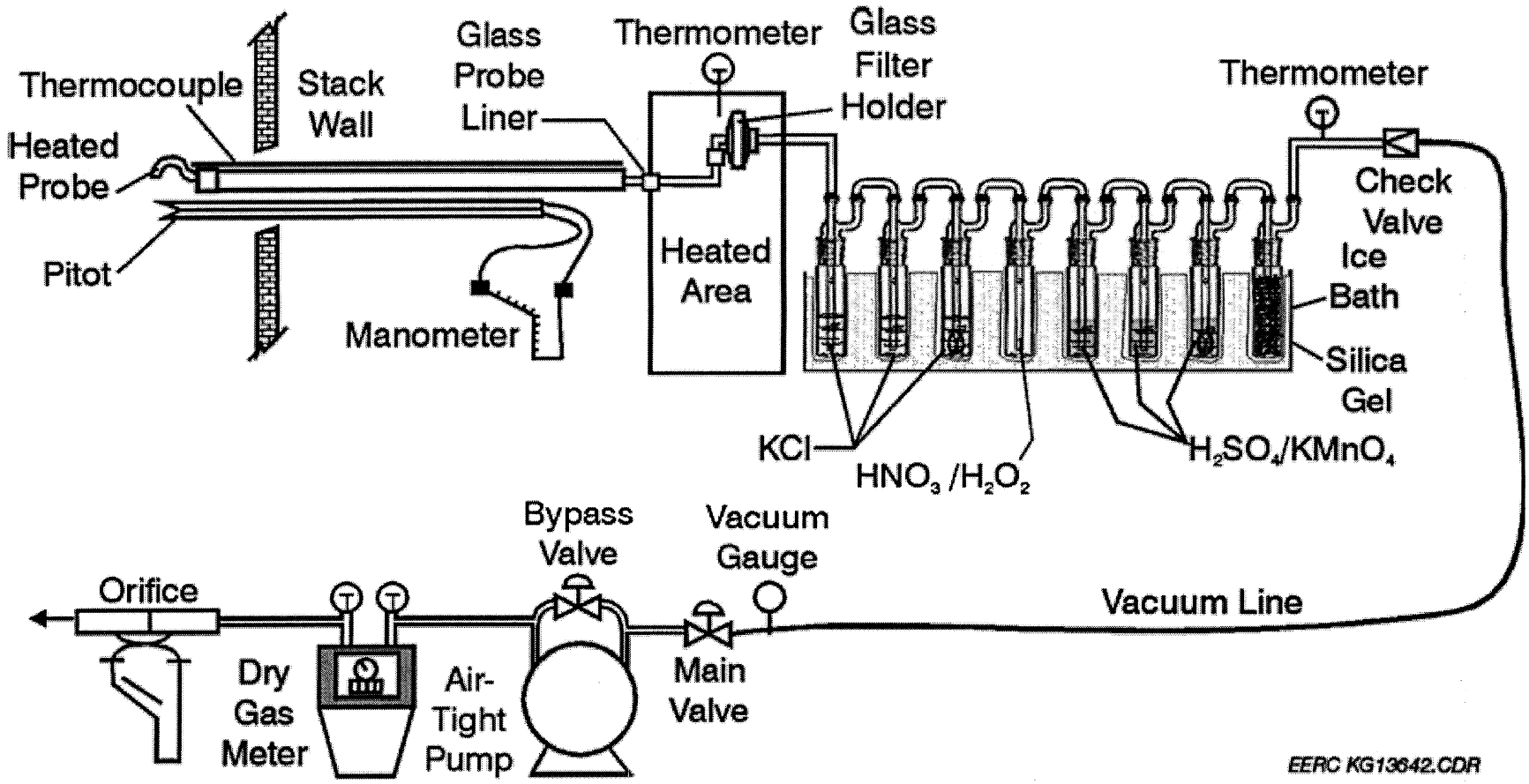

Figure 4-1. Schematic of the Mercury Speciation Sample Train
(Method 5 option as used at the stack is shown; Method 17 in-stack filtration was used for the Inlet on San Juan 2)

Table 4-1. Sample Train Components - Method 17 Configuration

Component	Details
Nozzle	Glass.
Filter	Quartz thimble, in glass thimble holder.
Probe	Teflon, heated to minimum 120 C.
Connector line	Heated teflon line used to connect from probe to impingers. Heated to minimum 120 C.
Impingers 1, 2	1 mol/l KCl solution; modified Smith Greenburg (SG) impinger.
Impinger 3	1 mol/l KCl solution; standard Smith Greenburg impinger.
Impinger 4	5% nitric acid/10% hydrogen peroxide; modified SG impinger.
Impingers 5, 6	4% potassium permanganate/10% sulfuric acid; modified SG impinger.
Impinger 7	4% potassium permanganate/10% sulfuric acid; standard SG impinger.
Impinger 8	Silica gel; modified Smith Greenburg Impinger

Table 4-2. Sample Train Components - Method 5 Configuration

Component	Details
Nozzle	Glass
Probe	Glass, heated to minimum 120 C.
Filter	Quartz, in glass holder, heated to minimum 120 C.
Connector line	Heated teflon line used to connect from probe to impingers. Heated to minimum 120 C.
Filter support	Teflon.
Impingers 1, 2	1 mol/l KCl solution; modified Smith Greenburg (SG) impinger.
Impinger 3	1 mol/l KCl solution; standard Smith Greenburg impinger.
Impinger 4	5% nitric acid/10% hydrogen peroxide; modified SG impinger.
Impingers 5, 6	4% potassium permanganate/10% sulfuric acid; modified SG impinger.
Impinger 7	4% potassium permanganate/10% sulfuric acid; standard SG impinger.
Impinger 8	Silica gel; modified Smith Greenburg Impinger

Sample was withdrawn from the flue gas stream isokinetically through the filtration system, which was followed by a series of impingers in an ice bath. Particulate-bound mercury was collected on the front half and filter; oxidized mercury was collected in impingers containing 1 N potassium chloride solution; and elemental mercury was 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 collected any remaining moisture.

The filter media was quartz fiber filters. At both the inlet and outlet quartz thimbles in a glass holders were used. At the inlet the probe included a heated teflon line; at the stack a heated glass probe was used. An additional heated teflon line was used to transport the flue gas from the end of the probe to the inlet of the first impinger. Both the probe and the line were heated to maintain a minimum gas temperature of 248°F.

A 150 minute sampling time was used at the inlet, with a target sample volume of 1 to 2.5 standard cubic meters. Sample time at the outlet was 144 minutes.

Sample Recovery

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 shipment to the laboratory. The following sample fractions were recovered (specific rinse solutions are contained in the method):

1. The sample filter;
2. The front half rinse (includes all surfaces upstream of the filter)
3. Impinger 1 through 3 (KCl impingers) and rinses;
4. Impinger 4 (HNO_3/H_2O_2 impinger) and rinses;
5. Impingers 5 through 7 ($KMnO_4/H_2SO_4$ impingers) and rinses;
6. Impinger 8 (silica gel impinger). Note this sample is weighed for moisture determination and is not included in the mercury analysis.

Sample Digestion and Analysis

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

Ash Sample (Containers 1 and 2)

If the particulate catch is greater than 1 gram (as would be the case at most particulate control device inlet locations), an aliquot of the particulate collected on the filter is digested by microwave digestion.

KCl Impingers (Container 3)

The impingers are digested using H_2SO_4, HNO_3, and $KMnO_4$ solutions as specified in the method.

KNO_3-H_2O_2 Impinger (Container 4)

The impinger solution is digested using HCl and $KMnO_4$ solutions as specified in the method.

H_2SO_4-$KMnO_4$ Impingers (Container 5)

The impinger solution is digested using hydroxylamine sulfate as specified in the method.

Analysis

Each digested fraction is analyzed in duplicate 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. Absorbency is measured as a function of mercury concentration. A soda-lime trap and a magnesium perchlorate trap must be used to precondition the gas before it enters the absorption cell.

Handling of Non Detects

This section addresses how data was handled in cases where no mercury was detected in an analytical fraction.

A single analytical fraction representing a subset of a mercury species is not detected. When more than one sample component is analyzed to determine a mercury species and one fraction is not detected, it is counted as zero. This occurred on five of six samples for elemental mercury, which is the sum of the mercury collected in the HNO_3/H_2O_2 impinger and the $H_2SO_4/KMnO_4$ impingers. For example, on Test 3-Outlet the H_2O_2 fraction was ND<0.25 μg and the $KMnO_4$ fraction was 13 μg. Elemental mercury was reported as 13 μg.

Mercury is detected on one or two of three runs. If mercury is detected on one or two of three runs, average mercury is calculated as the average of the detected value(s) and half of the detection limits for the non detect(s). However, if this average is below the detection limit, the average is reported as not detected less than the higher detection limit.

For example, the particulate mercury results for the three inlet tests (in units of lb/10^{12} Btu) are ND<0.03, 0.06, and ND<0.04. The average using half the detection limit is (0.03/2 + 0.06 + 0.04/2)/3, or 0.03. Since this value is below the higher detection limit of 0.04, the results are reported as ND<0.04.

1. Rinse filter holder and connector with 0.1N HNO_3.
2. Add $H_2SO_4/KMnO_4$ to each impinger bottle until purple color remains.
3. Rinse with 0.1N HNO_3.
4. Rinse with 8N HCl if brown residue remains.
5. Final rinse with 0.1N HNO_3.

Rinse Bottles Sparingly with
- 0.1N HNO_3
  8N HCl
- 0.1N HNO_3

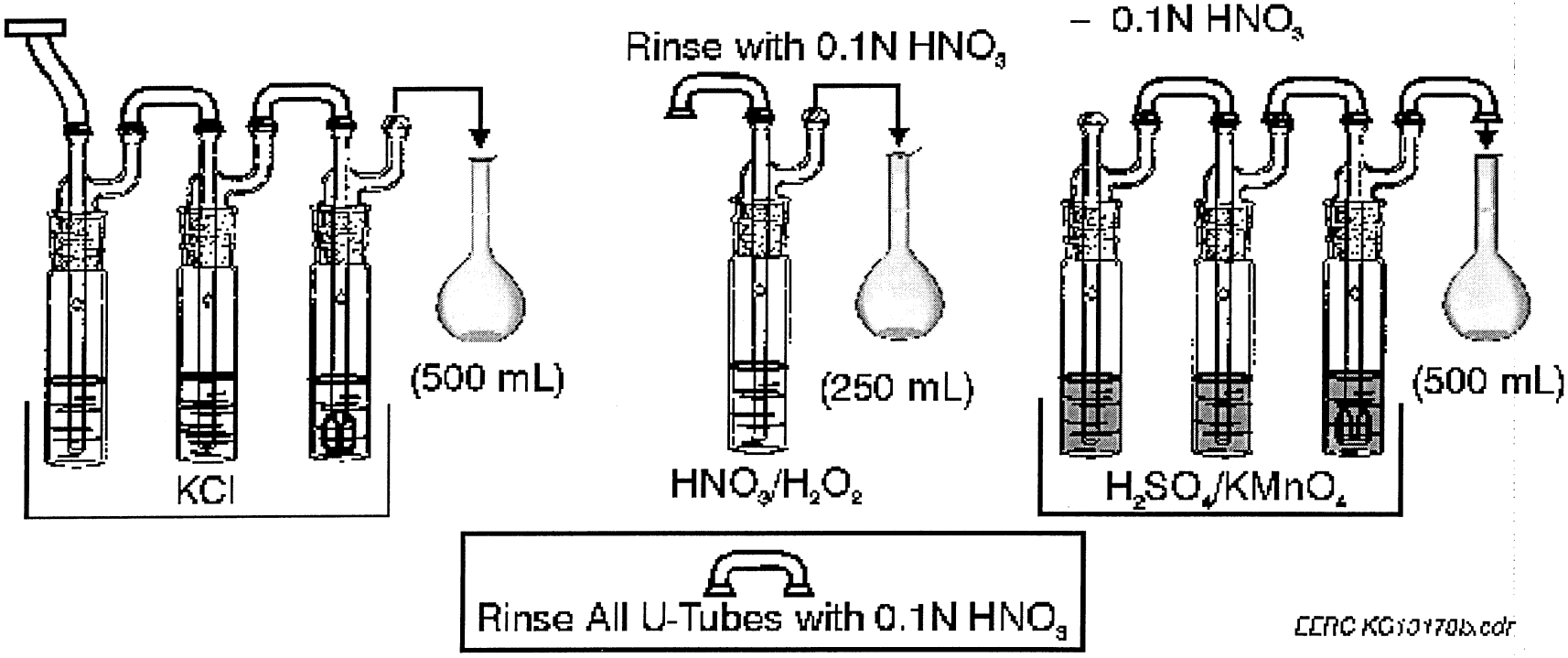

Figure 4-2. Sample Recovery Scheme for the Mercury Sampling Train

Mercury is not detected in one or more species. In summing up individual species to determine total mercury, a value of zero is used for non-detected species. For example, the average inlet mercury values (in lb/10^{12} Btu) were ND<0.04 for particulate mercury, 3.47 for oxidized mercury, and 3.25 for elemental mercury. Total mercury is reported as 3.47 + 3.25, or 6.72.

In calculating the percentage of mercury in each two species, a value of zero is used for the non-detected species. For the example listed in the preceding paragraph, the results are reported as 0% particulate mercury, 52% oxidized mercury, and 48% elemental mercury.

Auxiliary Flue Gas Measurements

Auxiliary flue gas measurements performed were flue gas flow rate per EPA Methods 1 and 2 (pitot traverse), O_2 by portable O_2 analyzer (as described below), and H_2O 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.

Outlet Moisture

Measured moisture values at the outlet were compared with saturation moistures for each test, and found to be about 1% higher than saturation. This excess may be due to collection of liquid water droplets. In accordance with EPA guidelines, saturation moisture was used for determination of gas density, calculation of isokinetic sample rates, and standard duct gas flow rates.

Inlet Flow Determination

Inlet gas flow rate was measured by the pitot traverse conducted as part of the mercury test.

Outlet Flow Determination

Outlet flow was measured by the pitot traverse conducted as part of the mercury test. This flow represents all of the scrubbed gas from Module H.

Comparative Flow Rate Calculations

As a QA indicator, additional flow rate determinations were done. At the inlet, total boiler exhaust gas flow was calculated based on boiler fuel input and oxygen (F_d) F factors. The plant CEMS stack flow rate is also presented.

Additionally, inlet and outlet flow rates for Module H were back calculated from stack CEMS flow rates. These flows were calculated by subtracting the bypass fraction from the stack flow and then assuming that the scrubbed flue gas was equally distributed among the three scrubbers.

Alternate Methodology for O_2/CO_2 Determination

As an alternate to conventional Orsat analysis, the following procedure was used for determination of O_2 and CO_2 content.

O_2 determination. O_2 was measured by a portable O_2 analyzer using an electrochemical cell. The gas sample for the portable analyzer was drawn through a tube inserted in the exit gas of the sample gas meter. This provides direct analysis of the gas sampled for the mercury test. Care was taken that the O_2 sample tube was not inserted so far that it interfered with the meter orifice pressure differential reading. Calibration procedures for the portable analyzer included:

1. At the beginning of the test day, the instrument was calibrated on ambient air. As-found readings were then taken using zero gas and an EPA Protocol 1 mid scale O_2 calibration gas (40 to 60% of the span used to collect readings). If these as found readings were within 2% of span, the data was acceptable. If the readings were outside of these ranges, the O_2 cell was replaced, the instrument was repaired, or an alternate instrument was used.
2. During testing, the calibration of the instrument was checked on ambient air every three or four sample points. If the as-read value on air had drifted more than 0.2% O_2 (0.8% of scale), the instrument was recalibrated.
3. At the end of the test day, the calibration error step described in Step 1 above was repeated.

CO_2 determination. CO_2 is used only for molecular weight determination.

At the outlet and inlet the CO_2 was calculated by stoichiometric calculations using standard F factors. It was not possible to calculate CO_2 at these locations by dilution calculations because there was no CO_2 measurement available from the stack. The stack CEMS at San Juan 2 uses O_2 rather than CO_2 as a diluent gas.

Determination of Scrubber Efficiency and Stack Emissions

A fraction of the flue gas at San Juan is scrubbed in order to meet SO_2 emission limits, and the balance bypasses the scrubber. This section presents the calculation procedures used to determine scrubber efficiency and to estimate stack emissions.

Scrubber Efficiency Determination

Scrubber removal efficiency was calculated according to Equation 1 below:

(1) $E = 1 - C_{out}/C_{in}$

Where,

E = Scrubber removal efficiency

C_{out} = Measured concentration at scrubber outlet

C_{in} = Measured concentration at scrubber inlet

It is important that the inlet and outlet values be corrected for air inleakage to provide results on a consistent basis. For this program, the correction was achieved by calculating mercury concentration in units of lb/10^{12} Btu.

Stack Emission Estimates

The stack gas concentration was calculated as shown in Equation 2:

(2) $C_{stack} = (BF \times C_{in}) + ((1-BF) \times C_{out})$

Where,

C_{stack} = Estimated concentration at stack

BF = fraction of gas bypassed, unitless. For San Juan 2, the bypass fraction was calculated by a heat and mass balance approach using moisture and temperature values at the inlet, outlet, and stack. As checks, calculations were also done by a heat balance method and a moisture balance method. Calculation summaries are shown in Appendix A. All three methods showed similar results, with bypass fractions in the range of 21-24%.

The stack mass emissions were calculated as shown in Equation 3:

(3) $M_{stack} = (BF \times M_{in}) + 3 \times M_{\text{Module H out}}$

Where M = mass flow, lb/hr, and subscripts denote location

4.2 Process Data

Process data was collected on computer logs set up by station personnel. Data collected included key boiler, scrubber, and ESP operating parameters, and all CEMS data.

Prior to and during each test, unit operation was assessed by station personnel to assure that operating conditions were within project target ranges.

5

INTERNAL QA/QC ACTIVITIES

5.1 QA/QC Problems

There were no sampling related QA/QC problems. All $KMnO_4$ impingers were purple at the conclusion of each test.

5.2 QA Audits and Data Quality Objectives

QA audit samples were analyzed as specified in the Ontario Hydro Method and listed in Table 5-1. Data quality objectives are listed in Table 5-2. Table 5-3 presents audit results and compares data quality results with data quality objectives. Table 5-4 presents individual mercury fraction mass measurements, along with field blank results.

All data quality objectives were met, with the following exceptions:

1. The target of all runs being within 35% of the mean was not met for the outlet particulate mercury measurements. The high relative scatter seen is more a function of the low absolute values of the measurements than to any potential problems with the tests. Measurements were near or below detection limits, and particulate mercury represented only 2% of total mercury at the outlet.
2. The target of all runs being within 35% of the mean was not met for the outlet oxidized mercury measurements. The high relative scatter seen is more a function of the low absolute values of the measurements than to any potential problems with the tests. Measurements were near or below detection limits, and oxidized mercury represented only 2% of total mercury at the outlet.

Table 5-1. Audit Samples for Ontario Hydro Mercury Speciation

Audit Sample	Acceptance Criteria and Frequency	Reference
Known reagent spike	Every 10 samples.	Ontario Hydro Section 13.4.1
Certified reference ash	One per program.	Ontario Hydro Section 13.4.1

Table 5-2. Data Quality Objectives for Flue Gas Mercury Analyses

Measure	*Objective*	*Approach*
Accuracy	≤10% of sample value or ≤10x instrument detection limit	Reagent blanks-analyze one blank per batch of each reagent
Accuracy	Field blank ≤30% of sample value, or no greater than reagent blank; whichever is higher	Collect and analyze one field blank at inlet and one at outlet; criteria evaluated for each mercury species
Accuracy	±10% of nominal value	One known reagent spike every ten samples
Precision, lab analysis	≤10% RPD	All laboratory samples analyzed in duplicate, every 10th sample analyzed in triplicate
Completeness	≥95%	Failed or incomplete tests to be repeated, if possible and practical

Table 5-3. Results Evaluation and Verification Checklist

Measure	***Objective***	***Result***
Unit Operation		
Unit operating conditions	No unusual conditions	Steady, normal operation
Air pollution control device operation	No unusual conditions	Steady, normal operation
Sample Train Information		
Trains leak checked before/after each test	<0.02 cfm	All tests passed
Pitot probes leak checked	Zero leakage	All tests passed
Probe, line, and filter temperature maintained	Minimum 120 C	All tests passed
Sample rate isokinetics	90-110%	101-102 % at inlet 101-103 % at outlet
Sample volume	1-2.5 std cubic meters	1.7-2.1 m^3 at inlet 2.0-2.3 m^3 at outlet
Post-test color of permanganate impingers	Purple	All tests passed
Results/lab QA		
Flow rate for triplicate runs	All runs w/in 10% of mean	W/in 9% at inlet W/in 9% at outlet
Stack temperature for triplicate runs	All runs w/in 5% of mean	W/in 2% at inlet W/in 1% at outlet
Total mercury for triplicate runs	All runs w/in 35% of mean	W/in 27% at inlet W/in 28% at outlet
Particulate mercury	All runs w/in 35% of mean	Not detected at inlet One run 36% high at outlet
Oxidized mercury	All runs w/in 35% of mean	W/in 32% at inlet W/in 19% at outlet
Elemental mercury	All runs w/in 35% of mean	W/in 27% at inlet W/in 29% at outlet
Sample and blank spikes	W/in 10% of value	All tests passed
Field blanks	<30% of measured values	Passed

Table 5-4. San Juan 2 Sample Fraction Mercury Measurements

	Run 2	Run 3	Run 4	Average	Field blank	Field blank/ sample, %
Inlet, μg/sample						
Filter/probe wash (particulate Hg)	ND<0.070	0.14	ND<0.080	ND<0.080	ND<0.060	ND
KCl fraction (oxidized Hg)	11	5.5	8	8.2	0.3	4%
$KMnO_4$ fraction (elemental Hg)	9.7	7.1	5.7	7.5	0.049	0.7%
H_2O_2 fraction (elemental Hg)	0.53	ND<0.25	ND<0.25	0.26	ND<0.05	ND
Stack, μg/sample						
Filter/probe wash (particulate Hg)	0.095	0.15	0.098	0.114	ND<0.060	ND
Replaced filter (see Section 3.2)		ND<0.080				
KCl fraction (oxidized Hg)	0.89	0.67	0.55	0.70	ND<0.10	ND
$KMnO_4$ fraction (elemental Hg)	14	8.6	8.3	10.3	ND<0.25	ND
H_2O_2 fraction (elemental Hg)	ND<0.25	ND<0.25	ND<0.25	ND<0.25	ND<0.05	ND