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December 22, 1999

Mr. William Grimley
Ms. Lara Autry
U.S. Environmental Protection Agency
Emission Measurement Center (MD-19)
Interstate 40 and Page Road
Room No. E-108/E-128
Durham, NC 27703

Via FedEx

Dear Mr. Grimley and Ms Autrey:

Attn: Electric Utility Steam Generating Unit Mercury Test Program



In accordance with the requirements of the EPA's mercury information collection effort (OMB No. 2060-0396), Central Illinois Public Service Company d/b/a AmerenCIPS submits two copies of the final test report for Newton unit 2 for emission testing to determine the particulate, oxidized and elemental mercury in the exhaust gases at the inlet to the cold side electrostatic precipitator and at the stack prior to exhaust to the atmosphere.

Fossil Energy Research Corporation conducted testing in accordance with the "Standard Test Method for Elemental, Oxidized, Particle-Bound, and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method)". On site testing activities were completed on August 2, 1999.

In accordance with the ICR, AmerenCIPS was required to submit the final emission test report to EPA within 90 days of completion of the testing. In this case the report submittal deadline was November 2, 1999. However, on October 21, AmerenCIPS requested an additional 60 days to submit the final test report to EPA. The extension was requested to allow the investigation of discrepancies between the measured gas phase mercury levels and the measured mercury levels in the coal. USEPA granted the extension request on October 22.

Please contact me if you have any questions concerning this submittal.

Sincerely,

A handwritten signature in black ink, appearing to read "Steven C. Whitworth".

Steven C. Whitworth
Supervising Environmental Scientist
Attachment

cc: D. J. Kolaz, IEPA
S. H. Rothblatt, USEPA Region V (w/o attachment)

MERCURY SPECIATION STACK SAMPLING TEST REPORT: NEWTON UNIT 2

December 1999

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Prepared for
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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 are required to collect and analyze flue gas samples for particulate, elemental, and oxidized mercury.

Ameren's Newton 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 stack, speciated mercury concentrations at the inlet of the boiler's last air pollution control device (a cold side electrostatic precipitator), and fuel mercury, chlorine, moisture, sulfur, ash, and heating value.

Test Unit

The test unit is Newton Unit 2. This unit is operated by Ameren CIPS, and is located in Newton, Illinois. The unit was selected by the EPA as part of the following category:

- fuel type: subbituminous
- SO₂ control type: none
- Particulate control type: cold side electrostatic precipitator (ESP)

Newton 2 is rated at 585 MW gross. The unit is a tangentially fired boiler, with low NO_x burners and close-coupled overfire air for NO_x control. Particulate emissions are controlled by an ESP, with a nominal collection efficiency of 99%.

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 stack/exhaust per the Ontario Hydro mercury speciation method.

- Particulate, oxidized, and elemental mercury concentrations at the inlet of one of the two ESPs.
- Mercury and chlorine content of representative coal samples collected from the coal feeders.
- Fuel moisture, sulfur, ash, and heating content.

Responsible Organizations

Responsible organizations for this project are:

- Test site operator: Ameren CIPS
- Sampling team: Fossil Energy Research Corp., with Delta Air Quality Services as a major subcontractor
- Gas sample analysis: Philip Analytical Services
- Fuel analysis: Ameren
- Additional fuel and gas sample analysis for Quality Assurance comparisons: University of North Dakota Energy and Environmental Research Center (EERC)

Dates of Test

The test program was conducted on July 31 and August 2, 1999. Daily activities included:

- July 31: set up, conducted field blanks, and conducted Run 1.
- August 2: conducted Runs 2 and 3, demobilized.

Document Description

This document is the test report for the Newton 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 Newton 2 Test Plan (Report No. FERCo R673) and the Newton 2 Quality Assurance Plan (Report No. FERCo R696). These reports are available from Ameren, the EPA or FERCo.

The Test Plan was approved by Mr. William Grimley of the EPA prior to testing, and the QA Plan was approved by Ms. Lara Autry of the EPA prior to testing. Specific comments on the Test Plan from Mr. Grimley were addressed in an addendum e-mailed from Robert Hof of Ameren to Mr. Grimley dated July 12, 1999. 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.

Mr. McDannel and Ms. Bell were all on-site for the testing. The Ameren on-site coordinator was Mr. David Heath of Newton Station. 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
Ameren	Robert Hof	Ameren Project Manager		(314) 554-2123	(314) 554-4188	robert_r_hof@ameren.com
FERCo	Mark McDannel	Program Manager	Robert Hof	(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

2

PLANT AND SAMPLING LOCATION DESCRIPTIONS

2.1 Process and Control Equipment Description and Operation

Newton 2 is a tangentially fired Combustion Engineering boiler rated at 585 MW gross. Figure 2-1 shows a schematic of the boiler and pollution control equipment, including gas sample points. Figure 2-2 shows a schematic of the coal delivery system, including fuel sample points.

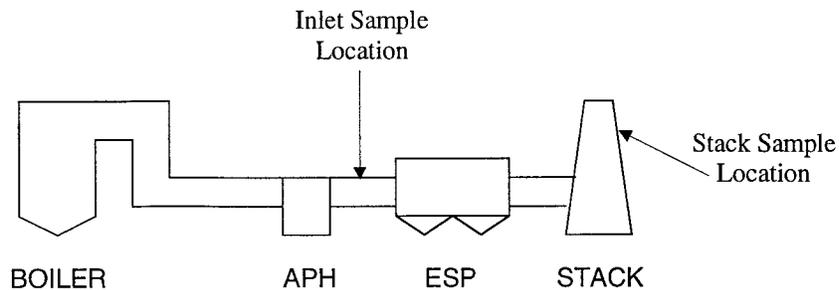


Figure 2-1 Newton 2 Boiler Schematic

Key unit parameters include:

- Unit capacity: 585 MW, gross
- Boiler type: Combustion Engineering tangentially fired
- fuel type: subbituminous, low sulfur
- SO₂ control: none
- Particulate control: ESP, efficiency 99%
- NO_x control: Low NO_x burners and close-coupled overfire air

Fuel samples were collected at the coal feeders ahead of the boiler, inlet samples were collected at one of two inlet ducts on one of the two ESPs and outlet samples were collected at the stack.

The sample gas at the inlet and stack is approximately 325°F.

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.

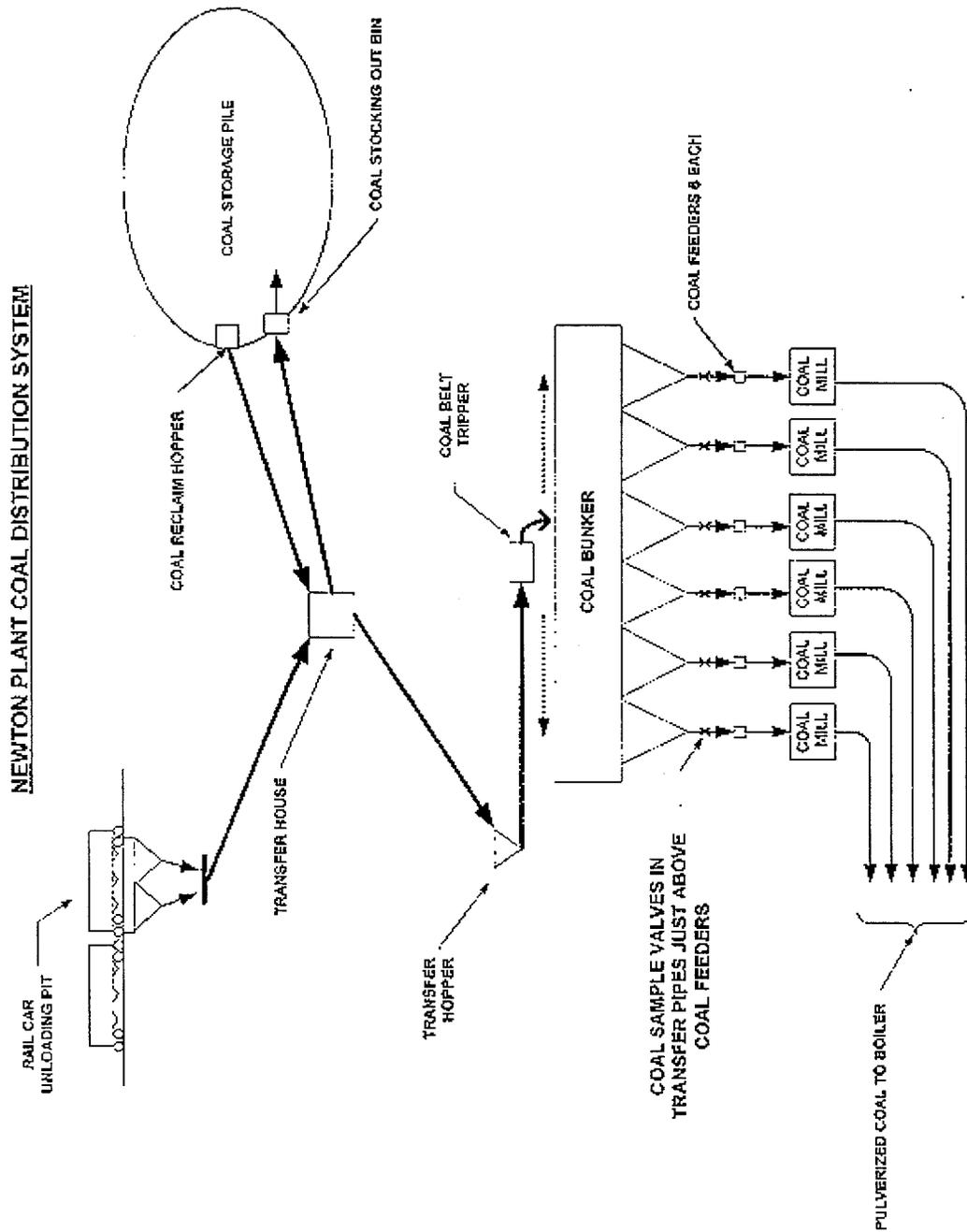


Figure 2-2. Schematic of the Coal Delivery System

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

Table 2-1. Summary of Newton 2 Process Data

Run No.	1	2	3
Date, 1999	31-Jul	2-Aug	2-Aug
Start time	1340	0751	1146
Stop time	1800	1032	1416
Unit load, MW	567	567	567
Coal mills in service	All 6	All 6	All 6
Coal flow, klb/hr	602	622	625
CEMS data			
CO ₂ , % wet	12.05	12.21	12.03
SO ₂ , lb/mmBtu	0.709	0.762	0.766
NO _x , lb/mmBtu	0.327	0.279	0.285
Opacity, %	19	19	19
Stack flow, kwscfh	1,307	1,252	1,217
Stack temperature, F	356	336	345
ESP data			
Power level, kW	1614	1609	1592
Sections out of service	None	None	None

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. ESP operation was monitored by Mr. David Heath of Newton Station.

2.2 Flue Gas Sampling Locations

Table 2-2 presents a summary of key inlet and stack sample location parameters. A layout showing the inlet and stack areas is shown in Figure 2-3. Individual discussions of the two locations are presented below.

Inlet Locations

The inlet samples were collected at one of the two inlet ducts of Precipitator 2A, one of two precipitators on Newton 2. Thus, one fourth of the total inlet flue gas was sampled. A schematic and cross-section of the inlet location are shown in Figures 2-4 and 2-5. This location does not meet the requirements of EPA Method 1.

Table 2-2. Newton 2 Sampling Location Descriptions

	Inlet	Stack
Description	Inlet duct to ESP 2A (one of four ducts)	Conventional stack test platform
Elevation	50' above grade	246' above grade
Physical access	Stairs, ladders	Elevator
Side or top access	Top	Side
Round or rectangular	Rectangular	Round
Port length (outside of port to inner stack wall)	18 inches	18 inches
Number/type of ports	Ten ports – five will be used	Four ports
Inside dimensions	13' 6" wide by 12' 0" deep Equivalent diameter 12.7 feet	26' 4" ID
Nearest upstream disturbance		
Disturbance	Duct elbow	ID fan discharge ducts
Distance, ft	16' 6"	289'
Distance, diameters	1.3	10.6
Nearest downstream disturbance		
Disturbance	ESP inlet	Stack exit
Distance, ft	4' 6"	185'
Distance, diameters	0.4	7.0
Approximate nominal flue gas conditions		
Temperature, F	325	325
Moisture, %	8-12%	8-12%
Flow rate, kscfm	550-650 (one of two ESPs)	1,100-1,300
O ₂ , % dry	3-5	3-5
CO ₂ , % dry	10-13	10-13
Particulate concentration, lb/MMBtu	2-4	0.05
SO ₂ , lb/MMBtu	0.6	0.6
NO _x , lb/MMBtu	0.2-0.3	0.2-0.3

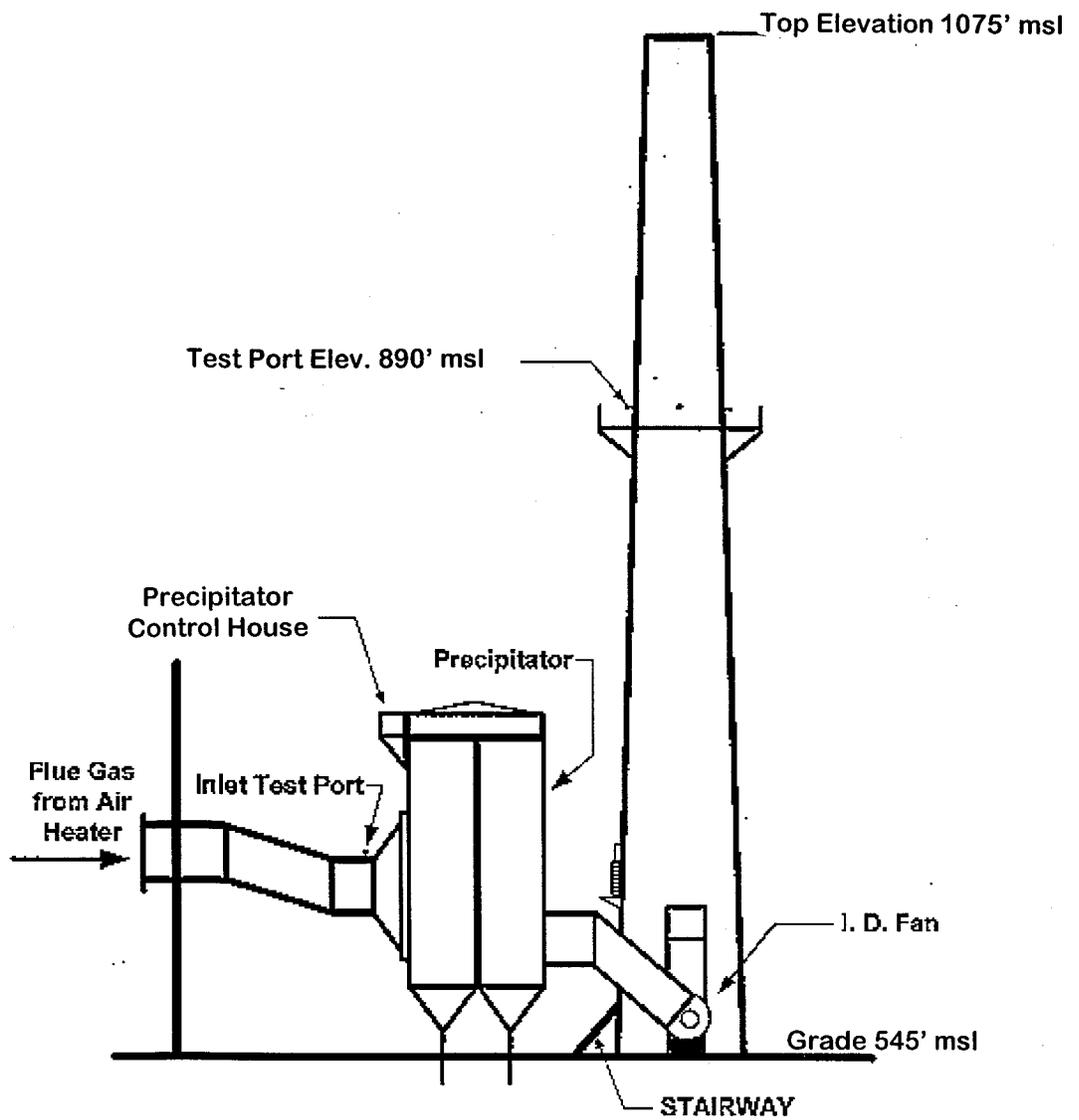
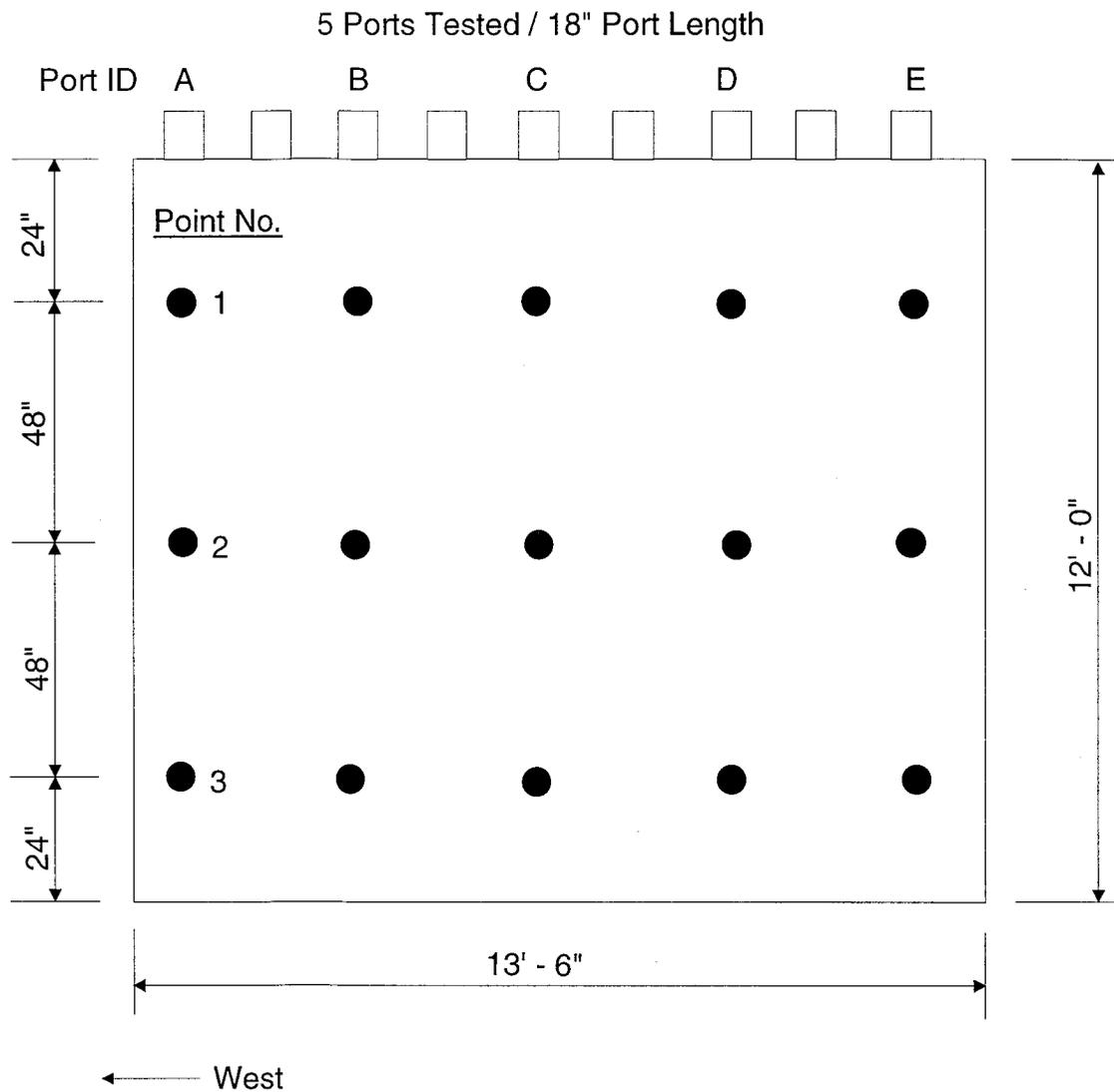


Figure 2-3. Layout of Newton 2 Sampling Locations



15 Points Sampled
 Area = 162.0 ft²
 Typical for 4 Ducts

Figure 2-5. Newton 2 Inlet Sample Location

Although this location does not meet the requirements of Method 1, three-dimensional flow testing as described in Method 1 was not performed because (1) mercury is primarily in the gaseous phase and is not impacted by uncertainties in gas flow and isokinetic sampling rate, (2) stratification of mercury species is not expected, and (3) if an inlet location fails to meet Method 1 criteria for flow angle, there is little that can be reasonably done to correct it. This approach is considered to be consistent with the intent and data quality requirements of the ICR.

Because of the number and location of the inlet ducts, it was not feasible to sample all of the ducts simultaneously with the stack sample without adding an additional sample team. Because mercury speciation is not expected to be stratified, and because the cost of an additional crew is not considered to be consistent with the intent of the ICR, inlet sampling was conducted in one duct. This approach should adequately characterize mercury speciation at the inlet.

One field change to the sample grid was made. During preliminary velocity traverses, it was discovered that there was a cross beam or other obstruction across the duct between sample points 2 and 3. While it was possible to maneuver the pitot probe around the obstructions to obtain the velocity traverse, it would not have been possible to maneuver the glass thimble holder and nozzle around the obstruction without breakage. Therefore, it was decided to not sample Point 3, and to double sample Point 2. The impact of this change on the results is considered to be minimal since there was no particulate mercury collected at the inlet. Gaseous oxidized and elemental mercury are not expected to be stratified.

Stack Location

The stack samples were collected at the existing stack sample ports. A schematic and cross section of the stack location is shown in Figure 2-4.

This location meets the requirements of EPA Method 1.

The flue gas at the stack is above the method specification of a minimum filtration temperature of 120°C. Therefore, in stack filtration per Method 17 was used.

Prior to the first test the stack location was checked for cyclonic flow per Method 1. There was some cyclonic flow, but the average angle was below the limit of 20 degrees.

Subsequent to completion of testing it was discovered that there had been some confusion over the stack diameter. A stack diameter of 29.0' was used to set up traverse points, but during post-test review it was determined that the actual stack diameter was 26.36 feet. This change means that the traverse points used for sampling were not at the exact centroids of equal area segments of the stack.

Table 2-3 presents the traverse points that were used and those that should have been used. The table shows that Point 1 was off by 1 ¼", Point 2 was off by 4 ½", and Point 3 was off by 9 ¼". The impact of this discrepancy on project results is expected to be negligible for two reasons:

Table 2-3. Traverse Points for Newton 2 Stack

Traverse Point	1	2	3
% of diameter	4.40%	14.60%	29.60%
Distance used for tests, inches from inside wall	15 1/4	50 3/4	103
Target distance, inches from inside wall	14	46 1/4	93 3/4
Difference, inches	1 1/4	4 1/2	9 1/4

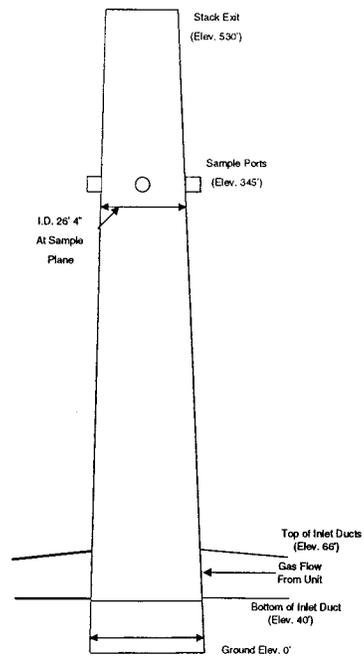
Note: traverses were performed assuming a 29.0 foot diameter stack.

It was subsequently determined that the stack diameter was 26.36 feet.

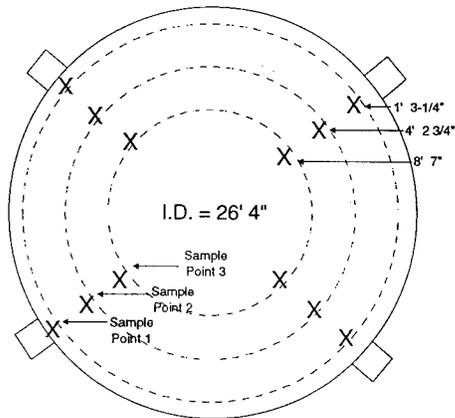
1. There was no particulate mercury measured at the stack. Gaseous elements and oxidized mercury are not generally considered to be stratified.
2. A review of velocities measured during testing indicates that Points 2 and 3 are in a region of uniform velocity. Repositioning Points 2 and 3 would have no impact on velocity measurements. Point 1 is in an area near the wall where velocity is low. Repositioning Point 1 by 1 1/4" might provide lower readings; however, the total impact on stack gas flow rate is expected to be 1-2% at most.

2.3 Coal Sampling Location

Coal samples were collected at the coal feeders to each individual mill by Newton Station personnel. One scoop sample was collected from each feeder during the first and last hour of each test run, and the individual samples were composited and riffled to provide one sample per run for analysis.



a. Diagram of Stack



b. Cross-Section of Sample

Figure 2-6. Newton 2 Stack 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 emissions at the stack.
- Quantify speciated mercury concentrations in the flue gas at the ESP inlet.
- Quantify fuel mercury and chlorine content during the stack 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. The table includes a list of test methods used. In addition to speciated mercury, the flue gas measurements included moisture, stack gas flow, and O₂/CO₂.

3.2 Field Test Changes and Problems

Traverse Points at Inlet

As discussed in Section 2.2, obstructions in the duct precluded sampling at Traverse Point 3 in each of the five inlet sample ports. Point 2 was double-sampled. This may have a slight, unknown impact on particulate phase mercury, but should have no impact on gas phase mercury species. However, the results show that there was no particulate mercury for these tests.

Traverse Points at Stack

As discussed in Section 2.2, an incorrect stack diameter was used to establish test traverse points. The impact of this is considered to be minimal.

Test Run 1 Restarted

Problems were experienced on both the inlet and stack sample trains shortly after the initial start of Test 1. At the inlet electrical power to the sample train was lost during sampling, resulting in

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

Sampling Location	No. of Runs	Species Measured	Sampling Method	Sample Run Time	Analytical Method	Analytical Laboratory
Stack	3	Speciated Hg	Ontario Hydro	120 min	Ontario Hydro	Philip Services
Stack	3	Moisture	EPA 4	Concurrent	Gravimetric	FERCo
Stack	3	Gas Flow	EPA 1/2	Concurrent	Pitot Traverse	FERCo
Stack	3	O ₂	Batch Sample	Concurrent	Portable O ₂	FERCo
Stack	3	CO ₂	N/A	Concurrent	Plant CEMS	FERCo
Inlet	3	Speciated Hg	Ontario Hydro	120 min	Ontario Hydro	Philip Services
Inlet	3	Moisture	EPA 4	Concurrent	Gravimetric	FERCo
Inlet	3	Gas Flow	EPA 1/2	Concurrent	Pitot Traverse	FERCo
Inlet	3	O ₂	Batch Sample	Concurrent	Portable O ₂	FERCo
Inlet	3	CO ₂	N/A	Concurrent	Dilution calc	FERCo
Coal Feeders	3	Hg, Cl in coal	Modified ASTM D2234	1 grab sample per mill per run	EPA 7473 (Leco)	Ameren

Table 3-2. Newton 2 Sampling Times

Run No.	1	2	3
Date, 1999	31-Jul	2-Aug	2-Aug
Inlet Tests			
Start time	1426	0752	1147
Stop time	1743	0957	1351
Total sample time, min	120	120	120
Stack Tests			
Start time	1340	0751	1146
Stop time	1759	1032	1416
Total sample time, min	120	120	120
Notes:			
1. Gas flow, moisture, O ₂ were concurrent with mercury tests.			
2. Coal samples were collected during the first and last hour of each run.			

back flushing of the impingers. A new sample train was loaded, the probe and line were washed, and a new test was started. At the stack, the sample line melted and became plugged after sampling two traverse points in the first port. Additionally, during investigation of the problem the sample nozzle was broken. The line and nozzle were replaced, the sample train was leak checked, and testing was resumed.

Broken Nozzle

During Test 2-Stack, the glass nozzle was broken upon removal from the third port tested. The nozzle was replaced with a same size nozzle, the sample train was leak checked, and sampling was resumed. The amount of particulate matter lost in the broken nozzle is considered to be negligible and not significant, since no particulate mercury was found in any of the stack samples.

Holding Time

Due to a series of delays in the laboratory, the samples were analyzed 60 to 70 days after sampling. The Ontario Hydro Method specifies 45 days.

This discrepancy is not considered to have any impact on the results. Dennis Laudal of the University of North Dakota (the author of the Ontario Hydro Method) indicates that they have performed stability studies showing that samples are stable for at least 3 months.

Stability studies will be performed on these samples to provide confirming data.

Change in Analysis Method for Mercury in Coal

The method used to measure mercury in coal was changed from the ASTM combustion bomb method cited in the test plan to EPA 7473, using a LECO analyzer. This switch was made because the EPA method provides increased sensitivity at low mercury levels.

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 ESP by species.
- Figure 3-1: Mercury speciation across ESP.

Results are calculated as $\mu\text{g}/\text{sm}^3$ (at a reference temperature of 68°F), and normalized for dilution by converting to a $\text{lb}/10^{12}$ Btu basis. This method allows direct comparison of inlet and stack results without incorporating uncertainties involved in gas flow measurement.

Table 3-3. Newton 2 Sample Gas Conditions

	Run 1	Run 2	Run 3	Average
Test Date	31-Jul	2-Aug	2-Aug	
Inlet Gas Properties				
Temperature, F	336	302	327	322
Gas flow for both ducts, dscfm	1,108,287	1,077,385	1,119,479	1,102,018
Comparison gas flows, dscfm				
Pitot traverse (x 4)	1,272,374	1,197,355	1,200,018	1,223,249
Calculated from fuel input and O ₂	1,076,263	1,094,066	1,077,288	1,082,539
Calculated from fuel input and CO ₂	1,024,479	1,047,916	1,061,205	1,044,534
O ₂ , % dry	4.00	4.05	3.96	4.00
CO ₂ , % dry	15.63	15.49	15.14	15.42
H ₂ O, %	14.98%	14.15%	13.82%	14.32%
Stack Gas Properties				
Temperature, F	336	325	338	333
Gas flow, dscfm (stack pitot traverse)	1,243,695	1,207,044	1,198,735	1,216,491
Comparison gas flow, dscfm				
Calculated from fuel input and O ₂	1,207,758	1,225,732	1,153,557	1,195,683
Calculated from fuel input and CO ₂	1,149,648	1,174,029	1,136,335	1,153,337
Stack CEMS	1,130,408	1,105,523	1,035,213	1,090,381
O ₂ , % dry	5.84	5.86	5.08	5.59
CO ₂ , % dry	13.93	13.83	14.14	13.97
H ₂ O, %	13.51%	11.70%	14.94%	13.38%

The following observations are made regarding the results:

1. There was no particulate mercury measured at either the inlet or stack.
2. The mercury was 91% elemental/9% oxidized at the inlet, and 80% elemental/20% oxidized at the stack.
3. Total mercury removal across the ESP was 8%. 19% of elemental mercury was removed, while oxidized mercury levels doubled. The increase in oxidized mercury could be due to conversation of Hg to HgCl₂ during the several seconds of residence time between the inlet and the stack.
4. Total mercury levels in the coal were within 20% of total mercury levels at the ESP inlet. This agreement is considered to be within the accuracy of the measurement methods.

Table 3-4. Newton 2 Mercury Speciation Results

	Run 1	Run 2	Run 3	Average
Test Date	31-Jul	2-Aug	2-Aug	
Inlet Mercury Speciation				
Particulate mercury				
ug/dscm	ND<0.08	ND<0.07	ND<0.16	ND<0.16
lb/10 ¹² Btu	ND<0.06	ND<0.05	ND<0.12	ND<0.12
% of total Hg	0.0%	0.0%	0.0%	0.0%
Oxidized mercury				
ug/dscm	0.55	0.59	1.56	0.90
lb/10 ¹² Btu	0.41	0.44	1.18	0.68
% of total Hg	5.6%	5.9%	15.1%	9.0%
Elemental mercury				
ug/dscm	9.16	9.28	8.77	9.07
lb/10 ¹² Btu	6.90	7.01	6.60	6.84
% of total Hg	94.4%	94.1%	84.9%	91.0%
Total mercury				
ug/dscm	9.71	9.86	10.34	9.97
lb/10 ¹² Btu	7.31	7.45	7.77	7.51
Stack Mercury Speciation				
Particulate mercury				
ug/dscm	ND<0.007	ND<0.005	ND<0.005	ND<0.007
lb/10 ¹² Btu	ND<0.006	ND<0.004	ND<0.004	ND<0.006
% of total Hg	0.0%	0.0%	0.0%	0.0%
Oxidized mercury				
ug/dscm	1.9	1.4	1.8	1.7
lb/10 ¹² Btu	1.6	1.2	1.5	1.4
% of total Hg	21.5%	19.0%	20.4%	20.4%
Elemental mercury				
ug/dscm	6.8	6.0	7.1	6.6
lb/10 ¹² Btu	5.7	5.1	5.7	5.5
% of total Hg	78.5%	81.0%	79.6%	79.6%
Total mercury				
ug/dscm	8.6	7.4	9.0	8.3
lb/10 ¹² Btu	7.3	6.3	7.2	6.9
Coal Analysis				
Mercury, ppm dry	0.078	0.068	0.083	0.076
Mercury, lb/10 ¹² Btu	6.61	5.61	6.88	6.37
Chlorine, ppm dry	178	ND<50	ND<50	76
Moisture, %	24.31	27.64	28.56	26.8
Sulfur, % dry	0.44	0.40	0.38	0.41
Ash, % dry	9.15	7.36	7.73	8.08
HHV, Btu/lb as fired	8,869	8,700	8,571	8,713
Coal flow, lb/hr as fired	602,000	622,000	625,000	616,333
Total Mercury Mass Rates				
lb/hr input in coal	0.035	0.030	0.037	0.034
lb/hr at ESP inlet	0.040	0.040	0.043	0.041
lb/hr emitted	0.040	0.033	0.040	0.038

Table 3-5. Newton 2 Mercury Removal Efficiency

Run No.	1	2	3	Average
Date, 1999	31-Jul	2-Aug	2-Aug	
Total mercury				
Inlet, lb/10 ¹² Btu	7.3	7.5	7.8	7.5
Stack, lb/10 ¹² Btu	7.3	6.3	7.2	6.9
Removal efficiency, %	0.3%	15.8%	7.3%	7.8%
Particulate mercury				
Inlet, lb/10 ¹² Btu	ND<0.06	ND<0.05	ND<0.12	ND<0.12
Stack, lb/10 ¹² Btu	ND<0.006	ND<0.004	ND<0.004	ND<.006
Removal efficiency, %	N/A	N/A	N/A	N/A
Oxidized mercury				
Inlet, lb/10 ¹² Btu	0.4	0.4	1.2	0.7
Stack, lb/10 ¹² Btu	1.6	1.2	1.5	1.4
Removal efficiency, %	-281.0%	-169.2%	-25.3%	-108.5%
Elemental mercury				
Inlet, lb/10 ¹² Btu	6.9	7.0	6.6	6.8
Stack, lb/10 ¹² Btu	5.7	5.1	5.7	5.5
Removal efficiency, %	17.1%	27.5%	13.1%	19.3%

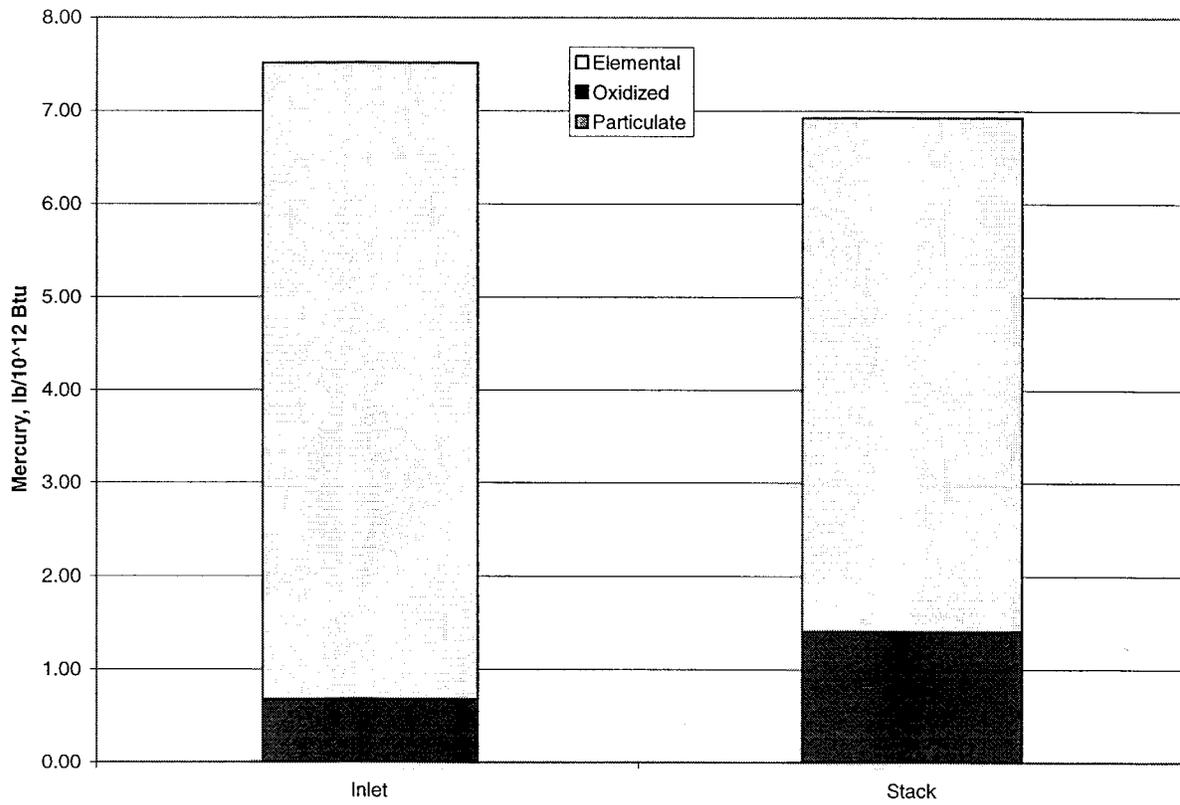


Figure 3-1. Newton 2 Mercury Distribution at Inlet and Stack

4

SAMPLING AND ANALYTICAL PROCEDURES

4.1 Test Methods

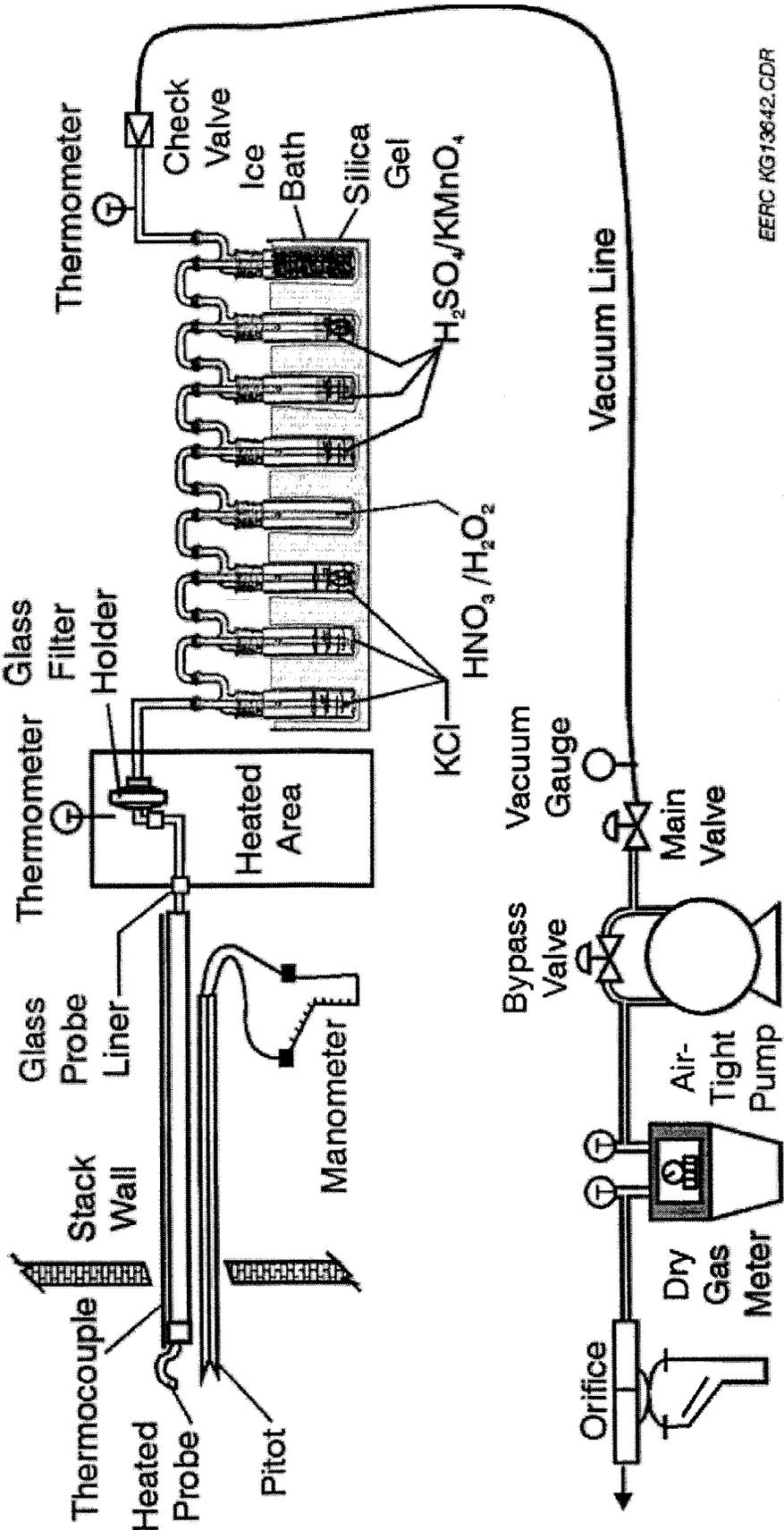
This section contains a summary of the sampling and analytical procedures used to conduct the mercury speciation method required in EPA's ICR titled, "Standard Test Method for Elemental, Oxidized, Particle-Bound and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro 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 July 31, the set up day. 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 17 requirements for isokinetic sampling were followed. The impinger train was weighed before and after sampling to determine flue gas moisture content.

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



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

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

Component	Details
Nozzle	Glass, quartz, or teflon-coated stainless steel
Filter	Quartz, in glass or teflon-coated stainless steel holder.
Probe	Glass or teflon, heated to gas temperature.
Connector line	Heated teflon line used to connect from probe to impingers. Heat 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

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.

The filter media was quartz fiber filters. At the inlet, a quartz thimble in a glass holder was used. At the stack, a 47 mm quartz filter in a teflon coated stainless steel holder was used. At both locations, the probe included a heated teflon line. 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 250°F.

A 120 minute sampling time was used at the stack, with a target sample volume of 1 to 2.5 standard cubic meters. At the inlet, a sample time of 120 minutes was used.

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):

5. The sample filter;
6. The front half rinse (includes all surfaces upstream of the filter)
7. Impinger 1 through 3 (KCl impingers) and rinses;
8. Impinger 4 (HNO₃/H₂O₂ impinger) and rinses;
9. Impingers 5 through 7 (KMnO₄/H₂SO₄ impingers) and rinses;
10. 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 H₂SO₄, HNO₃, and KMnO₄ solutions as specified in the method.

KNO₃-H₂O₂ Impinger (Container 4)

The impinger solution is digested using HCl and KMnO₄ solutions as specified in the method.

H₂SO₄-KMnO₄ Impingers (Container 5)

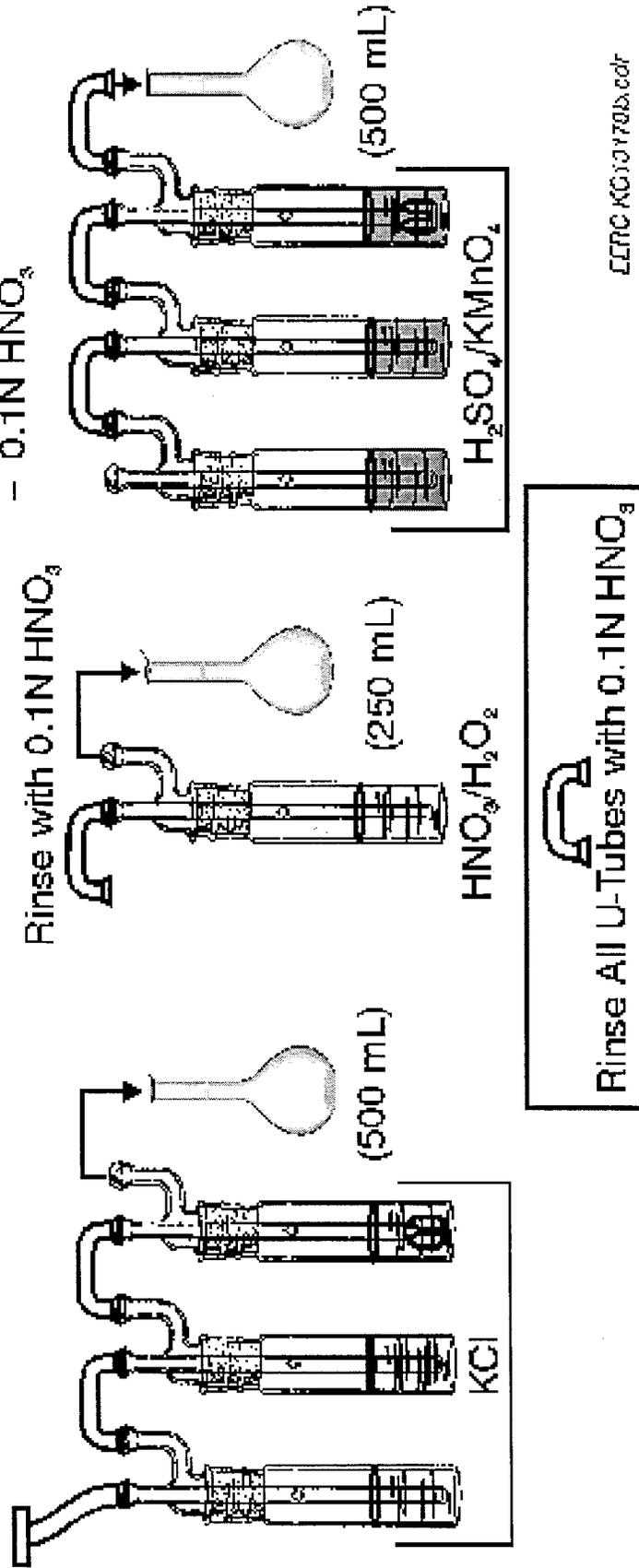
The impinger solution is digested using hydroxylamine sulfate as specified in the method.

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.

1. Rinse filter holder and connector with 0.1N HNO₃.
2. Add H₂SO₄/KMnO₄ to each impinger bottle until purple color remains.
3. Rinse with 0.1N HNO₃.
4. Rinse with 8N HCl if brown residue remains.
5. Final rinse with 0.1N HNO₃.

Rinse Bottles Sparingly with
 - 0.1N HNO₃
 8N HCl
 - 0.1N HNO₃



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

Handling of Non Detects

This section addresses how data was handled in cases where no mercury was detected in an analytical fraction. It should be noted that the analytical method specified in the Ontario Hydro Method has a very low detection limit, which was well below flue gas levels for most cases.

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 will be counted as zero. This can occur for elemental mercury, which is the sum of the mercury collected in the HNO₃/H₂O₂ impinger and the H₂SO₄/KMnO₄ impingers. For example, on Run 2-Inlet the HNO₃/H₂O₂ fraction was ND<0.25 µg and the KMnO₄ fraction was 11.4 µg. Elemental mercury is calculated as 11.4 µg.

No mercury is detected for a species on all three test runs. When all three test runs show no detectable levels of mercury for a mercury species, that mercury species is reported as not detected at less than highest detection limit. For example, the three results for particulate mercury at the inlet are ND < 0.10, ND < 0.08, and ND < 0.20, the results are reported as ND < 0.20.

In summing up individual species to determine total mercury, a value of zero is used for non-detected species. For example, on Test 1-Inlet particulate mercury is ND < 0.10 ug, oxidized mercury is 0.7 ug, and elemental mercury is 11.9 ug, total mercury is reported as 12.6 ug.

In calculating the percentage distribution of mercury species, a value of zero is used for non-detected species. For the example listed in the preceding paragraph, the results are reported as 0% particulate mercury, 6% oxidized mercury, and 94% elemental mercury.

Auxiliary Flue Gas Measurements

Auxiliary flue gas measurements performed were flue gas flow rate per EPA Methods 1 and 2 (pitot traverse), O₂ by portable O₂ analyzer (as described below), and H₂O by EPA Method 4 (condensation/gravimetric analysis). These measurements will be collected as integral parts of all mercury speciation test runs at both the inlet and stack locations.

Inlet Flow Determination

There will typically be higher uncertainties in gas flow measurements at the inlet location relative to the stack location due to non axial flow. To calculate mercury levels in terms of lb/hr at the inlet, the outlet flow, corrected for dilution using O₂ measurements, is used for inlet values. This allows direct comparison of inlet and outlet mercury measurements without incorporating added uncertainty from the gas flow measurements.

Comparative Flow Rate Calculations

As a QA indicator, additional flow rate determinations were done. At both locations, exhaust gas flow was calculated based on boiler fuel input and both oxygen (F_d) and carbon (F_c) F factors.

At the stack, the plant CEMS stack flow rate is presented. At the inlet the pitot traverse results, multiplied by four since one of four ducts was tested, are presented.

Alternate Methodology for O₂/CO₂ Determination

As an alternate to conventional Orsat analysis, the following procedure will be used for determination of O₂ and CO₂ content.

O₂ determination. O₂ was measured by a portable O₂ analyzer using an electrochemical cell. The gas sample for the portable analyzer is 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 is taken that the O₂ sample tube is not inserted so far that it interferes with the meter orifice pressure differential reading. One reading is taken per traverse point, and the reading is manually recorded on the sample train data sheet.

Calibration procedures for the portable analyzer include:

1. At the beginning of the test day, the instrument is calibrated on ambient air. As-found readings are then taken using zero gas and a mid scale O₂ calibration gas (40 to 60% of the span to be used to collect readings). An EPA Protocol 1 calibration gas is used. If these as found readings are within 2% of span (0.2% O₂ if the 10% scale is used), the data is acceptable.
2. During testing, the calibration of the instrument is checked on ambient air every three sample points. The as-found reading is taken, and the instrument is recalibrated each time.
3. At the end of the test day, the calibration error step described above is repeated.

CO₂ determination. CO₂ is used for molecular weight determination. At the stack, CO₂ readings are taken from the plant CEMS. The CEMS values are on a wet basis; dry CO₂ values are calculated using the measured moisture content at the stack.

At the inlet, the CO₂ is calculated via dilution calculations from the inlet O₂, the stack O₂, and the stack CO₂.

4.2 Process Data

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

Prior to and during each test unit operation was assessed by the sampling team process monitor, in conjunction with 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. Sampling operational problems were discussed in Section 3.2. 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 raw mercury mass measurements and field blank results.

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

All data quality objectives were met, except that the oxidized mercury on Run 3-Inlet was 74% above the mean. This spread could be due to process, sampling, or analytical factors. It does not indicate that the result is invalid.

5.3 Comparison Analyses

As an independent Quality Assurance check of the data, flue gas and coal samples were analyzed by the University of North Dakota Energy and Environmental Research Center (EERC). These results, shown in Table 5-5, indicate excellent agreement between the laboratories for both the gas and coal samples.

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

<i>Measure</i>	<i>Objective</i>	<i>Approach</i>
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

<i>Measure</i>	<i>Objective</i>	<i>Result</i>
<i>Unit Operation</i>		
Unit operating conditions	No unusual conditions	Steady, normal operation
Air pollution control device operation	No unusual conditions	Steady, normal operation
<i>Sample Train Information</i>		
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%	97-105% at inlet 94-104% at stack
Sample volume	1-2.5 std cubic meters	1.2-1.3 m ³ at inlet 1.5-2.2 m ³ at stack
Post-test color of permanganate impingers	Purple	All tests passed
<i>Results/lab QA</i>		
Flow rate for triplicate runs	All runs w/in 10% of mean (adjusted for load)	All load-adjusted flows w/in 4% of mean at inlet, 2% of mean at stack.
Stack temperature for triplicate runs	All runs w/in 5% of mean	W/in 3% at inlet W/in 2% at stack
Total mercury for triplicate runs	All runs w/in 35% of mean	Met target
Particulate mercury	All runs w/in 35% of mean	Met target
Oxidized mercury	All runs w/in 35% of mean	Run 3-inlet 74% above mean
Elemental mercury	All runs w/in 35% of mean	Met target
Sample and blank spikes	w/in 10% of value	All tests passed
Field blanks	< 30% of measured values	See Table 5-4

Table 5-4. Newton 2 Sample Fraction Mercury Measurements

	Run 1	Run 2	Run 3	Average	Field blank	Field blank/ sample, %
Inlet, µg/sample						
Filter/probe wash (particulate Hg)	ND<0.10	ND<0.080	ND<0.20	ND<0.20	ND<0.080	ND
KCl fraction (oxidized Hg)	0.71	0.72	1.96	1.1	0.12	11%
H ₂ O ₂ fraction (elemental Hg)	0.32	ND<.25	ND<.25	ND<.25	ND<.25	ND
KMnO ₄ fraction (elemental Hg)	11.6	11.4	11.0	11.3	ND<0.030	ND
Stack, µg/sample						
Filter/probe wash (particulate Hg)	ND<0.010	ND<0.010	ND<0.010	ND<0.010	ND<0.010	ND
KCl fraction (oxidized Hg)	2.78	2.79	3.7	3.1	0.14	5%
H ₂ O ₂ fraction (elemental Hg)	0.56	ND<.25	0.50	0.40	ND<0.25	ND
KMnO ₄ fraction (elemental Hg)	9.6	11.9	13.9	11.8	ND<0.030	ND

Table 5-5. Results of Independent QA Analyses of Newton 2 Samples

Run No.	1	2	3	Average
Date, 1999	31-Jul	2-Aug	2-Aug	
<i>Inlet laboratory mercury results, µg/sample</i>				
KCl fraction by Philip	0.71	0.72	1.96	1.13
KCl fraction by EERC	0.49	1.16	1.69	1.11
KMnO ₄ fraction by Philip	11.6	11.4	11.0	11.3
KMnO ₄ fraction by EERC	11.2	11.0	11.2	11.1
<i>Stack laboratory mercury results, µg/sample</i>				
KCl fraction by Philip	2.78	2.79	3.70	3.09
KCl fraction by EERC	2.53	2.37	3.38	2.76
KMnO ₄ fraction by Philip	9.6	11.9	13.9	11.8
KMnO ₄ fraction by EERC	9.5	12.0	13.8	11.8
<i>Fuel mercury analyses, ppm dry</i>				
Fuel mercury by Ameren	0.078	0.068	0.083	0.076
Fuel mercury by EERC	0.080	0.065	0.092	0.079
<i>Total mercury mass rates by as-reported methods and by EERC</i>				
Fuel lb/hr by Ameren	0.035	0.030	0.037	0.034
Fuel lb/hr by EERC	0.037	0.029	0.041	0.035
Inlet lb/hr by Philip	0.040	0.040	0.043	0.041
Inlet lb/hr by EERC	0.037	0.041	0.043	0.040
Stack lb/hr by Philip	0.040	0.033	0.040	0.038
Stack lb/hr by EERC	0.039	0.033	0.039	0.037