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January 4, 2000

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 Fed Ex**

Dear Mr. Grimley and Ms. Autrey:

**Re: 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), Union Electric Company d/b/a AmerenUE submits two copies of the revised final test report for Meramec unit 4 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. This report replaces the original test report that was submitted to EPA on October 27, 1999. Please dispose of all copies of the original report.

This report contains corrected mercury emission results. After the original report was submitted, Philip Analytical discovered a systematic calculation error in a spreadsheet program that affected all of their calculations of KCl and  $KM_nO_4$  fraction mercury levels. Flue gas mercury levels were recalculated and the corrected results are presented in the revised report.

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 July 30, 1999.

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: Kendall Hale, MDNR  
Christopher Byrne, St. Louis County  
William Spratlin, USEPA Region VII (w/o attachment)

# MERCURY SPECIATION STACK SAMPLING TEST REPORT: MERAMEC UNIT 4

January 2000

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**Principal Investigator**  
Mark D. McDannel, P.E.

**Prepared for**  
Ameren UE  
St. Louis, MO



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# 1

## INTRODUCTION

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### 1.1 Summary of Test Program

Note: This revised report replaces the original Meramec 4 Test Report (Report No. FERCo-R709), which was submitted to the EPA in October, 1999. This version contains corrected mercury emission results, following discovery of a systematic calculation error in the original laboratory report. The author recommends that copies of the original report be disposed of.

#### *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 UE's Meramec 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. Ameren requested, and EPA approved, substitution of Meramec 4 for Meramec 2 for testing purposes.

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 Meramec Unit 4. This unit is operated by Ameren UE, and is located in St. Louis County, Missouri, at the confluence of the Meramec and Mississippi Rivers, approximately 20 miles south of St. Louis. The unit was selected by the EPA as part of the following category:

- Fuel type: bituminous, subbituminous,
- SO<sub>2</sub> control type: none
- Particulate control type: cold side electrostatic precipitator (ESP)

Meramec 4 is rated at 351MW gross, and is a front wall-fired (three coal mills, six exhausters, eighteen burners) Foster Wheeler boiler.

The boiler uses Babcock/Wilcox DRB-XCL® burners and is balanced draft. The burners are low NO<sub>x</sub> burners. The boiler has six rear wall NO<sub>x</sub> ports to control NO<sub>x</sub> and two side wall ports to control flame length of the six lower burners.

Ignition fuel is natural gas torches, main fuel is pulverized coal. Normal coal supply is subbituminous from the Powder River Basin in Wyoming.

The flyash electrostatic precipitator is an American Air Filter Co. weighted rigid tube discharge electrode precipitator divided into four electrical fields with a design SCA of 440ft<sup>2</sup>/10<sup>3</sup> CFM and a guarantee 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 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 UE
- Sampling team: Fossil Energy Research Corp., with Delta Air Quality Services as a major subcontractor
- Sample analysis: Philip Analytical Services

## Dates of Test

The test program was conducted on July 27-30, 1999. Daily activities included:

- July 27: set up and conduct field blanks.
- July 28: started and aborted Run 1 due to equipment problems.
- July 29: conducted Runs 2 and 3.
- July 30: conducted Run 4 and demobilized.

## Document Description

This document is the test report for the Meramec 4 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 Meramec 4 Test Plan (Report No. FERCo R672) and the Meramec 4 Quality Assurance Plan (Report No. FERCo R695). 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 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. Hof, 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
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

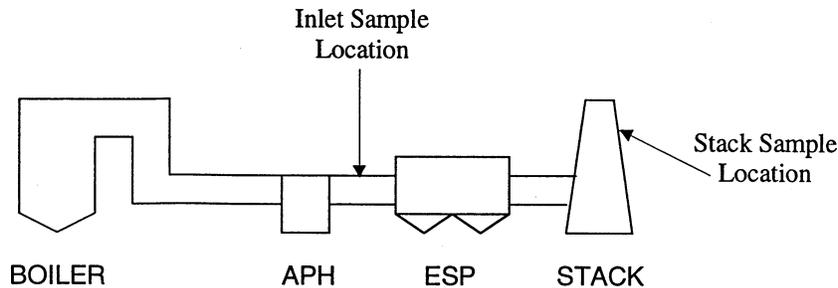
# 2

## PLANT AND SAMPLING LOCATION DESCRIPTIONS

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### 2.1 Process and Control Equipment Description and Operation

Meramec 4 is a front wall fired Foster Wheeler boiler rated at 351 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 supply system, including fuel sampling points.



**Figure 2-1 Meramec 4 Boiler Schematic**

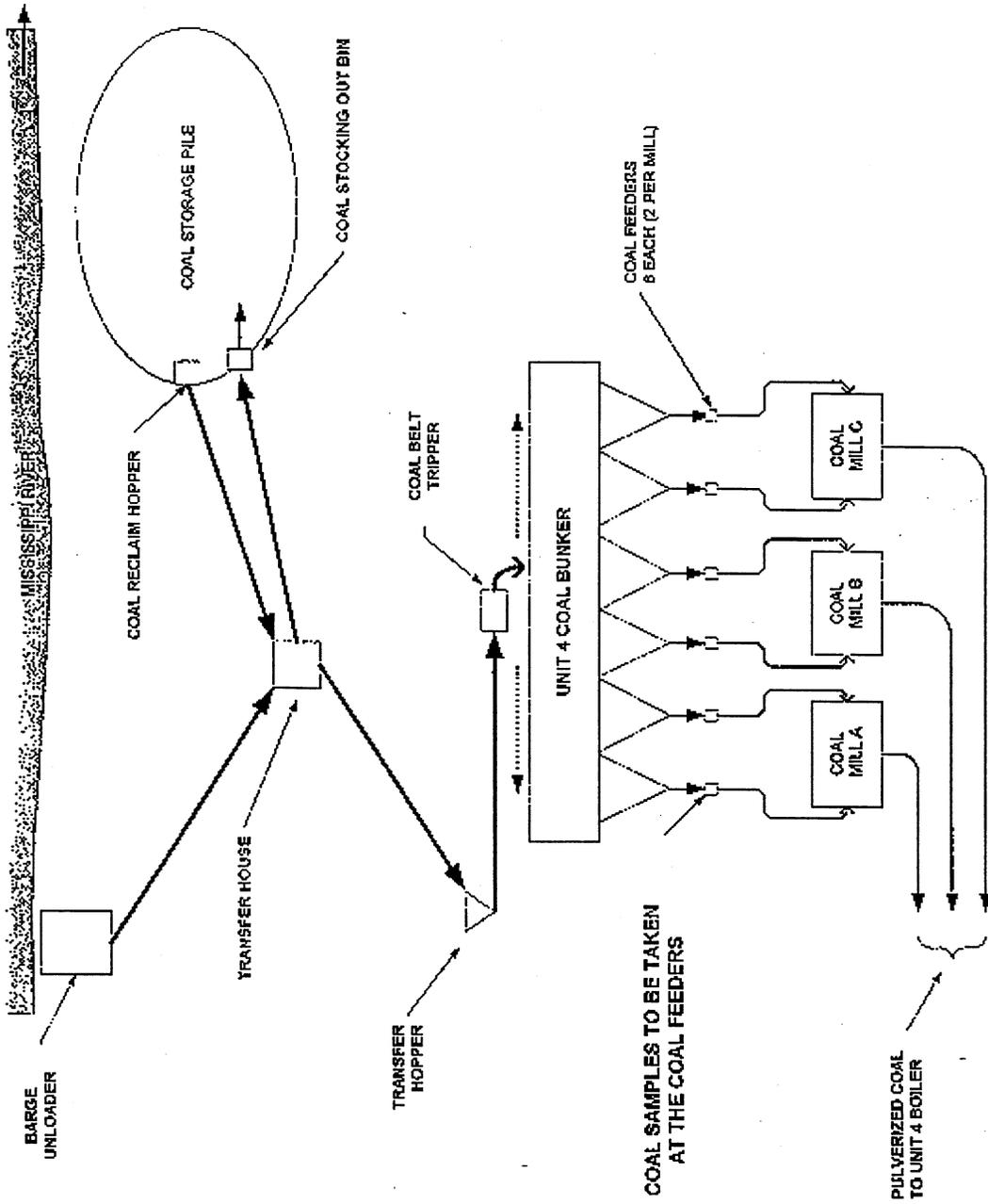
Key unit parameters include:

- Unit capacity: 351 MW, gross
- Boiler type: Foster Wheeler, front wall-fired, balanced draft
- Fuel type: bituminous, subbituminous
- SO<sub>2</sub> control: none
- Particulate control: ESP, guarantee efficiency 99%, SCA 440 ft<sup>2</sup>/10<sup>3</sup> cfm
- 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 ESP 4C (one of two ESPs on the unit) and outlet samples were collected at the stack.

The sample gas at the inlet and stack is approximately 330°F.

**MERAMEC UNIT 4 COAL DISTRIBUTION SYSTEM**



JDM 7/7/89

**Figure 2-2. Meramec 4 Coal Distribution 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 Meramec 4 Process Data**

Run No.	2	3	4		
Date, 1999	29-Jul	29-Jul	30-Jul		
Start time	1010	1400	0745		
Stop time	1230	1635	0959		
Unit load, MW gross	357	325	303		
Steam flow, klb/hr	2838	2638	2618		
Coal mills in service	All 3	All 3	All 3		
Coal flow, klb/hr	325	277	265		
APH exit gas temp, F	330	319	293		
APH inlet gas temp, F	720	704	717		
CEMS data					
CO <sub>2</sub> , % wet	10.5	10.8	10.9		
SO <sub>2</sub> , lb/mmBtu	1.48	1.83	1.71		
NO <sub>x</sub> , lb/mmBtu	0.44	0.42	0.42		
Opacity, %	4	4	3		
Stack flow, wscfh	1,056,000	917,900	819,600		
ESP data					
Power level, kW	see note	see note	676		
Sections out of service	4C3	4C3	4C3		
Note: Precipitator data was not captured for Tests 2 and 3 due to a computer malfunction.					
Power level was 770 kW just prior to testing, and operation was considered to be normal.					

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. Thomas Hart of Meramec Station, the engineer responsible for ESP operation. Despite the missing ESP power data on July 29 due to a failure of the logger to record data, ESP operation was normal.

During Run 2, there was a drop in load from 345 to 280 MW over a period of 5-10 minutes. This load drop was required by generator seal oil problems. A review of boiler operation indicated that there were no serious upsets in unit operating parameters that would impact test results. Specifically, there were no large swings in excess O<sub>2</sub> or opacity, which serve as indicators of upsets in combustion and ESP performance, respectively.

## 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 shown in Figure 2-3. Individual discussions of the two locations are presented below.

### *Inlet Locations*

The inlet samples were collected at the inlet of Precipitator 4C. A schematic and cross-section of the inlet location is shown in Figure 2-4. This location does not meet the requirements of EPA Method 1.

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 was expected to be primarily in the gaseous phase and was 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 is 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 inlet sample locations was made. Because of the high particulate loading at the ESP inlet, the probe was designed to maintain the particulate thimble upright rather than pointed down, as is often done with Method 17 type sampling in vertical ducts. This design meant that the thimble holder, thimble support bracket, and connecting sample line were all positioned below the nozzle. With the size of this assembly, the lowest traverse point (Point 1 on the drawing, Point 5 as designated on the test data sheets) could not be reached. To accommodate the thimble assembly and to provide a safety margin against damage, the lowest point the probe could reach was Point 2. Therefore, sample was not collected at Point 1, and Point 2 was double-sampled. Thus, sampling in each sample port included 5 minutes each at Points 3, 4, and 5, and 10 minutes at Point 2. Duplicate sets of readings were taken at Point 2. This change adds some uncertainty to the particulate mercury measurement, since large particles will preferentially settle toward the bottom of the duct. However, since little is known about mercury concentration vs. particle size the magnitude and direction of the bias are unknown. There should not be any additional uncertainty on gaseous elemental and oxidized mercury measurements.

### *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 2-5.

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 was checked for cyclonic flow per Method 1. There was no cyclonic flow (<5 deg) at all sample points.

### **2.3 Coal Sampling Location**

Coal samples were collected at the coal feeders to each individual mill by Meramec Station personnel. One scoop sample was collected from each coal 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.

**Table 2-2. Meramec 4 Sampling Location Descriptions**

	<b>Inlet</b>	<b>Stack</b>
Description	Inlet duct to one of two ESPs	Conventional stack test platform
Elevation	25' above grade	229' above grade
Physical access	Two ladders	Ladder
Side or top access	Top	Side
Round or rectangular	Rectangular	Round
Port length (outside of port to inner stack wall)	20 inches	17 inches
Number/type of ports	Ten 6-inch ports with flanges – five were used	Four 6-inch ports with flanges
Inside dimensions	19' 6" wide by 10' 6" deep Equivalent diameter 13.6 feet	17' 9" ID
Nearest upstream disturbance		
Disturbance	Outlet of retired ESP	ID fan discharge ducts
Distance, ft	14'	173'
Distance, diameters	1.0	9.8
Nearest downstream disturbance		
Disturbance	Duct split to ESP sections	Stack exit
Distance, ft	16'	124'
Distance, diameters	1.2	7.0
Approximate nominal flue gas conditions		
Temperature, F	330	330
Moisture, %	10	10
Flow rate, dscfm	450,000 (one of two ducts)	900,000
O <sub>2</sub> , % dry	7	7
CO <sub>2</sub> , % dry	11	11
Particulate concentration, lb/MMBtu	2-4	<0.10
SO <sub>2</sub> , lb/MMBtu	1.0	1.0
NO <sub>x</sub> , lb/MMBtu	0.5	0.5

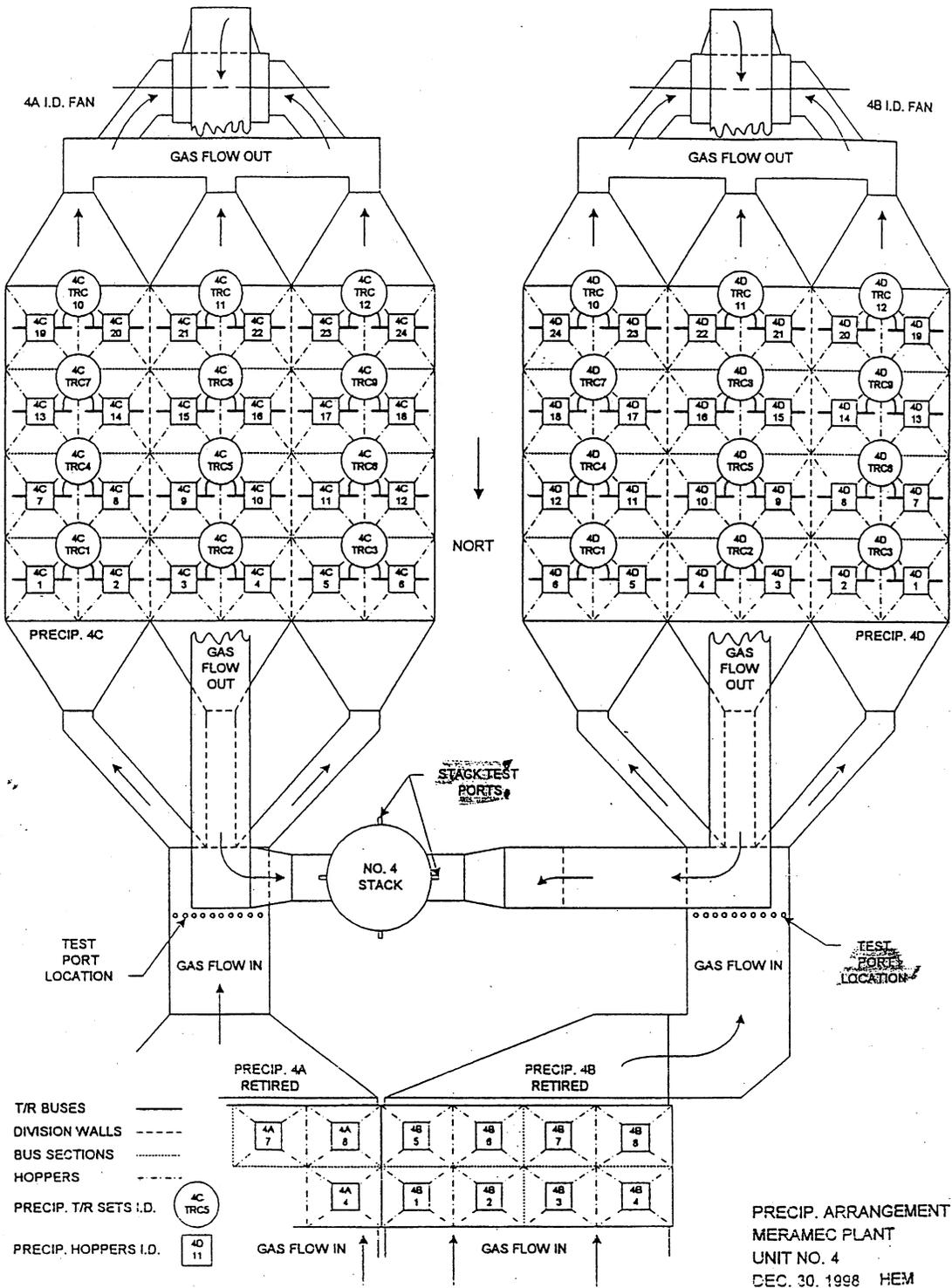


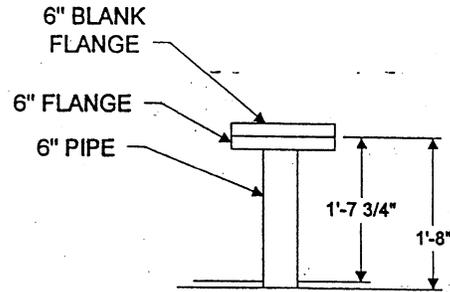
Figure 2-3. Layout of Meramec 4 Sampling Locations

# MERAMEC 4 C & D PRECIP.

## LOCATION OF TRAVERSE POINTS

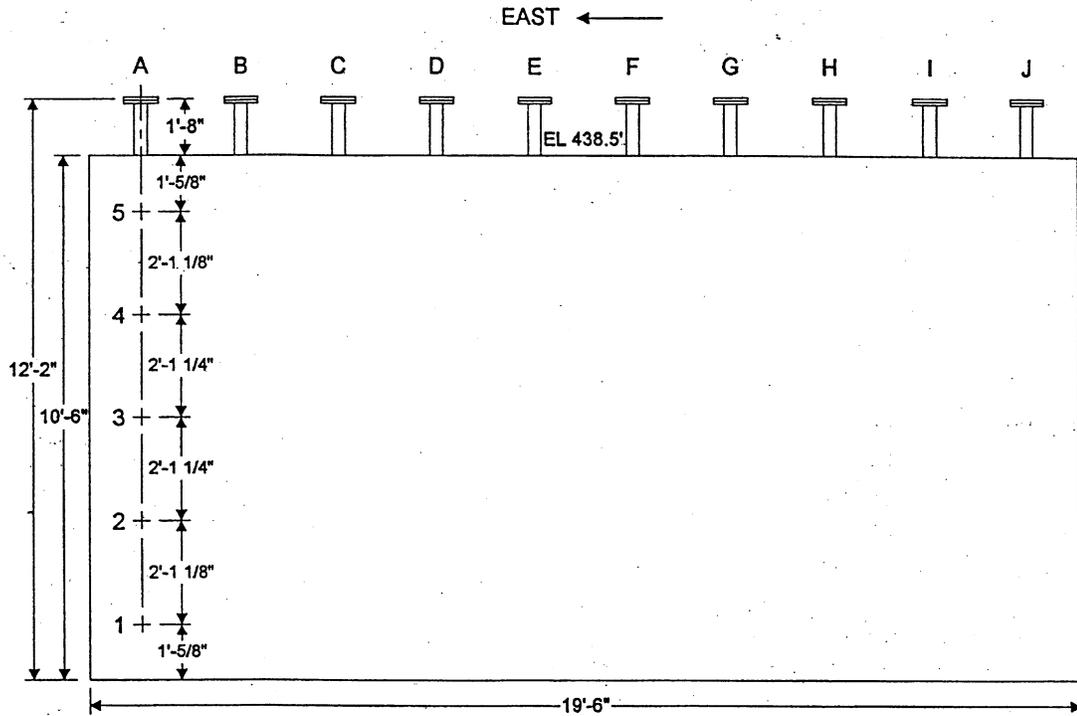
### VERTICAL TRAVERSE

POINT	DEPTH
5	2'-8 5/8"
4	4'-9 3/4"
3	6'-11"
2	9'-1/4"
1	11'-1 3/8"



NO. PTS. 50

DUCT AREA 204.75 SQ. FT.



A DUCT EAST SIDE  
B DUCT WEST SIDE

C & D PRECIPITATORS  
INLET TEST PORTS  
MERAMEC PLANT  
UNIT NO. 4  
APRIL 16, 1998 HEM

Figure 2-4. Meramec 4 Inlet Sampling Location

# MERAMEC 4

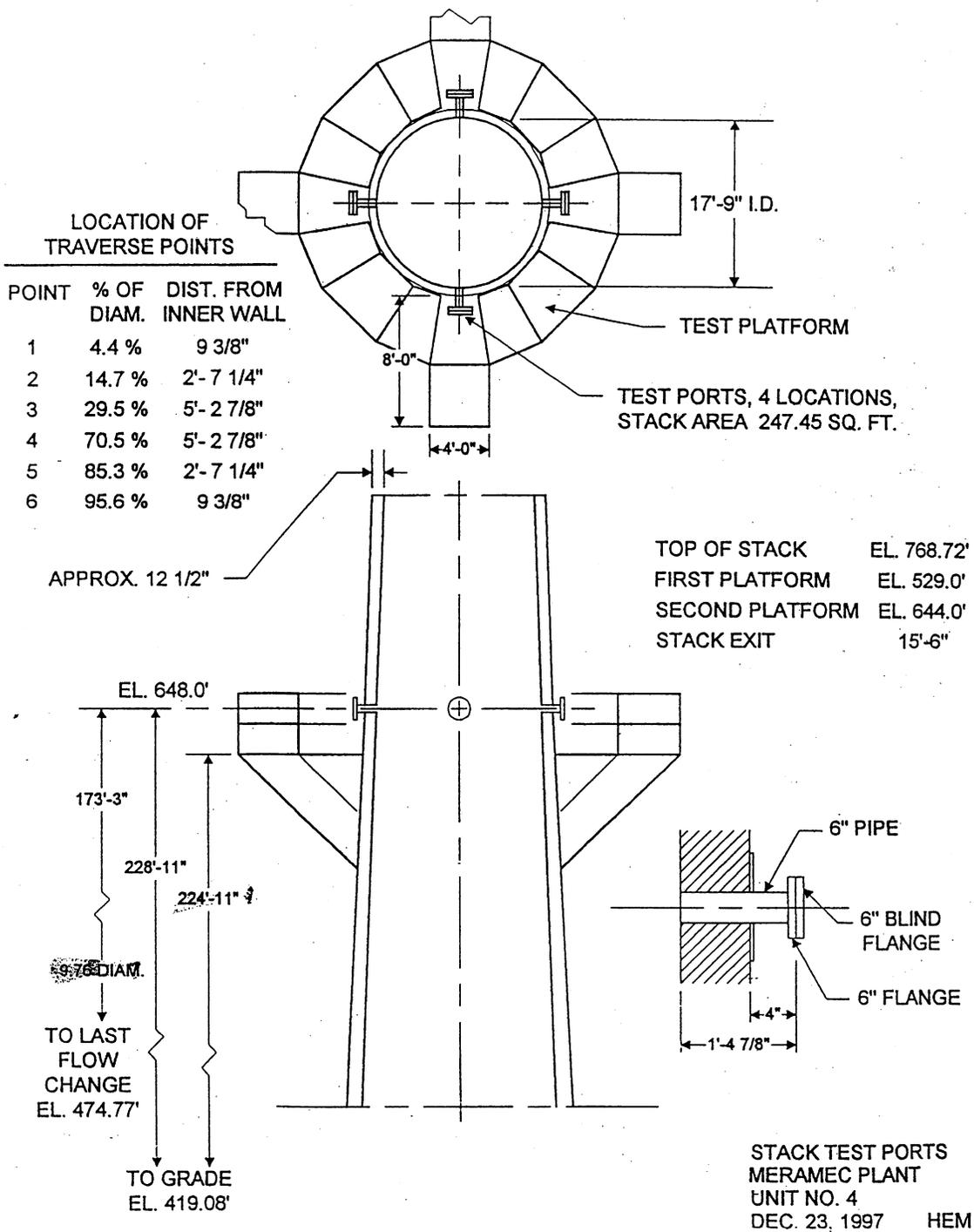


Figure 2-5. Meramec 4 Stack Sampling Location

# 3

## SUMMARY AND DISCUSSION OF TEST RESULTS

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### 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 O<sub>2</sub>/CO<sub>2</sub>.

### 3.2 Field Test Changes and Problems

#### *Sampling Time at Inlet*

In accordance with a Test Plan comment from the EPA, the sampling time at the inlet was changed from 120 minutes to 125 minutes. This was done to provide 5 minutes for each of the 25 sampling points. The Ontario Hydro Method specifies a minimum of 5 minutes per sample point.

#### *Traverse Points at Inlet*

As discussed in Section 2.2, the probe design precluded sampling at Traverse Point 1 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. Particulate mercury was 60% of total mercury for these tests.

### ***Test Run 1 Voided***

Half way through Test 1, the heated teflon sample line at the stack melted. Repairs could not be made and the test could not be saved. Therefore, Test 1 was voided at the stack and inlet. Replacement parts arrived the next day, and testing proceeded with Test 2. Thus, the 3 valid runs for this program are Tests 2, 3, and 4.

### ***Broken Nozzle***

During Test 3-Inlet, 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, especially considering that 10-12 grams of fly ash were collected in the thimble filter.

### ***Holding Time***

Due to a series of delays in the laboratory, the samples were analyzed 65 to 75 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 done on these samples to provide confirming data.

### ***Lab Calculation Error***

Subsequent to submittal of the original Test Report to the EPA, a systematic error was discovered by Philip Analytical in all of their calculations of KCl and KMnO<sub>4</sub> fraction mercury levels. Flue gas mercury levels were recalculated, and the results are presented in this revised report.

## **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.

Major observations that can be made from the results are:

1. Agreement between total mercury in the coal and at the ESP inlet is excellent, considering the uncertainties in the methods and the low mercury levels, with  $6.8 \text{ lb}/10^{12}$  Btu measured in the coal and  $6.6 \text{ lb}/10^{12}$  Btu measured at the inlet.
2. Mercury removal is 74% across the ESP, with virtually all particulate Hg removed.
3. Oxidized and elemental mercury appear to increase across the ESP. This is most likely an artifact of the test method at the inlet, with some of the gas phase mercury being absorbed as it passes through the large quantity of fly ash collected on the filter.
4. Most of the gas phase mercury is in the oxidized form at both the inlet and stack.

**Table 3-1. Test Matrix for Mercury ICR Tests at Meramec 4**

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 ½	Concurrent	Pitot Traverse	FERCo
Stack	3	o <sub>2</sub>	Batch Sample	Concurrent	Portable o <sub>2</sub>	FERCo
Stack	3	co <sub>2</sub>	N/A	Concurrent	Plant CEMS	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 <sub>2</sub>	Batch Sample	Concurrent	Portable o <sub>2</sub>	FERCo
Inlet	3	co <sub>2</sub>	N/A	Concurrent	Dilution calc	FERCo
Coal Feeders	3	Hg, Cl in coal	Modified ASTM D2234	1 grab sample per coal feeder per run	ASTM D3684	Ameren

**Table 3-2. Meramec 4 Sampling Times**

Run No.	2	3	4
Date, 1999	29-Jul	29-Jul	30-Jul
<b>Inlet Tests</b>			
Start time	1010	1401	0745
Stop time	1224	1635	0959
Total sample time, min	125	125	125
<b>Stack Tests</b>			
Start time	1010	1400	0747
Stop time	1230	1614	0959
Total sample time, min	120	120	120
<b>Notes:</b>			
1. Run 1 was voided after it started due to a melted sample line.			
2. Gas flow, moisture, o <sub>2</sub> were concurrent with mercury tests.			
3. Coal samples were collected during the first and last hour of each run.			

Table 3-3. Meramec 4 Sample Gas Conditions

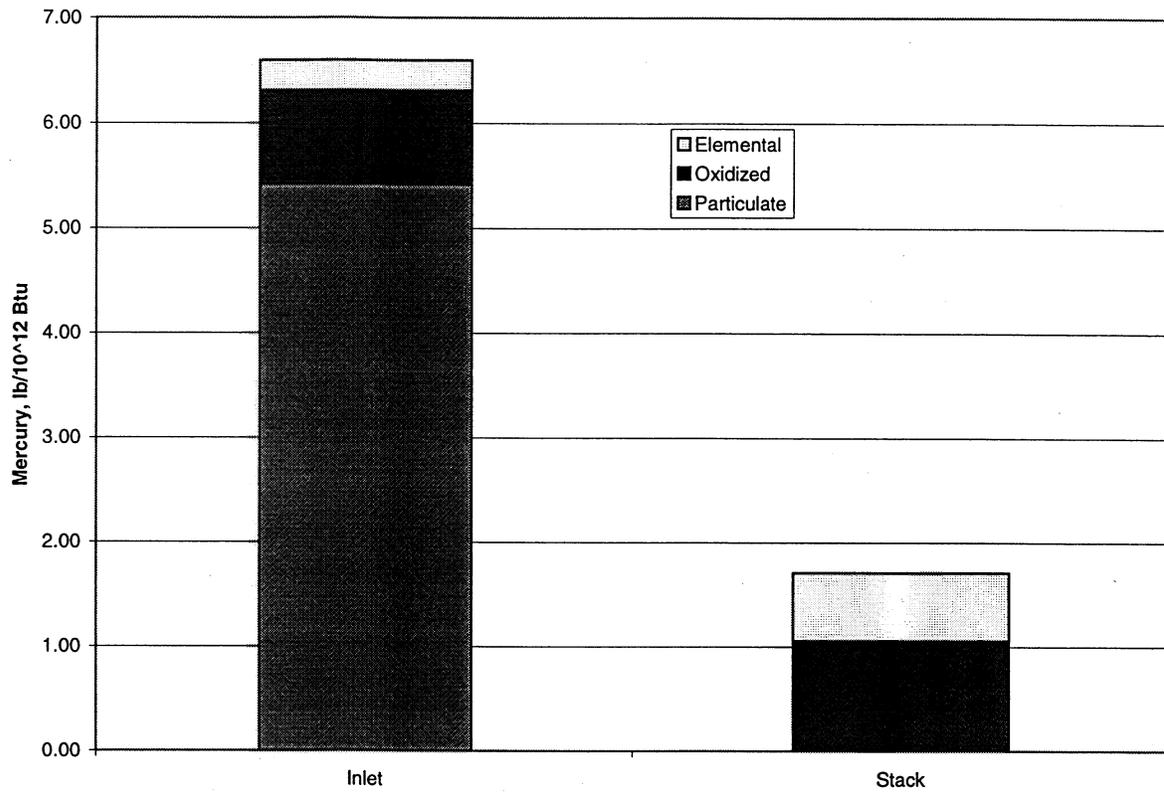
	Run 2	Run 3	Run 4	Average
<b>Test Date</b>	29-Jul	29-Jul	30-Jul	
<b>Inlet Gas Properties</b>				
Temperature, F	337	341	335	338
Gas flow for both ducts, dscfm	937,727	904,359	867,632	904,851
Comparison gas flows, dscfm				
Pitot traverse (x 2)	948,168	826,982	752,499	842,550
Calculated from fuel input and O <sub>2</sub>	883,510	810,535	737,271	810,439
Calculated from fuel input and CO <sub>2</sub>	916,560	823,120	796,295	845,325
O <sub>2</sub> , % dry	6.61	6.82	6.43	6.62
CO <sub>2</sub> , % dry	12.13	12.21	11.80	12.05
H <sub>2</sub> O, %	10.92%	10.29%	10.84%	10.68%
<b>Stack Gas Properties</b>				
Temperature, F	323	326	302	317
Gas flow, dscfm (stack pitot traverse)	983,135	929,443	852,317	921,632
Comparison gas flow, dscfm				
Calculated from fuel input and O <sub>2</sub>	926,291	833,017	724,257	827,855
Calculated from fuel input and CO <sub>2</sub>	960,943	845,951	782,240	863,045
Stack CEMS	960,786	836,095	743,827	846,903
O <sub>2</sub> , % dry	7.27	7.20	6.17	6.88
CO <sub>2</sub> , % dry	11.57	11.88	12.01	11.82
H <sub>2</sub> O, %	9.02%	8.91%	9.25%	9.06%

**Table 3-4. Meramec 4 Mercury Speciation Results**

	Run 2	Run 3	Run 4	Average
<b>Test Date</b>	29-Jul	29-Jul	30-Jul	
<b>Inlet Mercury Speciation</b>				
Particulate mercury				
ug/dscm	6.08	7.36	4.57	6.07
lb/10 <sup>12</sup> Btu	5.42	6.65	4.02	5.41
% of total Hg	82.3%	75.8%	60.7%	73.2%
Oxidized mercury				
ug/dscm	0.39	1.07	1.56	1.01
lb/10 <sup>12</sup> Btu	0.35	0.97	1.37	0.90
% of total Hg	6.0%	12.2%	23.5%	13.6%
Elemental mercury				
ug/dscm	0.11	0.35	0.50	0.32
lb/10 <sup>12</sup> Btu	0.10	0.31	0.44	0.28
% of total Hg	1.7%	3.9%	7.5%	4.3%
Total mercury				
ug/dscm	6.59	8.78	6.63	7.40
lb/10 <sup>12</sup> Btu	5.87	7.94	5.83	6.59
<b>Stack Mercury Speciation</b>				
Particulate mercury				
ug/dscm	ND<0.004	0.006	ND<0.005	ND<0.005
lb/10 <sup>12</sup> Btu	ND<0.004	0.006	ND<0.004	ND<0.004
% of total Hg	0.0%	0.2%	0.0%	0.0%
Oxidized mercury				
ug/dscm	0.58	1.69	1.24	1.17
lb/10 <sup>12</sup> Btu	0.54	1.57	1.07	1.06
% of total Hg	48.6%	65.7%	65.4%	61.9%
Elemental mercury				
ug/dscm	0.61	0.87	0.65	0.71
lb/10 <sup>12</sup> Btu	0.57	0.81	0.57	0.65
% of total Hg	51.4%	34.1%	34.6%	38.0%
Total mercury				
ug/dscm	1.19	2.57	1.89	1.88
lb/10 <sup>12</sup> Btu	1.11	2.38	1.64	1.71
<b>Coal Analysis</b>				
Mercury, ppm dry	0.085	0.12	0.068	0.091
Mercury, lb/10 <sup>12</sup> Btu	6.41	8.89	5.07	6.79
Chlorine, ppm dry	3,200	3,860	3,800	3,620
Moisture, %	14.09	10.55	11.95	12.20
Sulfur, % dry	0.89	1.29	1.27	1.15
Ash, % dry	7.75	8.18	7.85	7.93
HHV, Btu/lb as fired	11,387	12,075	11,813	11,758
Coal flow, lb/hr as fired	325,462	277,430	265,095	289,329
<b>Total Mercury Mass Rates</b>				
lb/hr input in coal	0.024	0.030	0.016	0.023
lb/hr at ESP inlet	0.023	0.030	0.021	0.025
lb/hr emitted	0.004	0.009	0.006	0.006

**Table 3-5. Meramec 4 Mercury Removal Efficiency**

Run No.	2	3	4	Average
Date, 1999	29-Jul	29-Jul	30-Jul	
<b>Total mercury</b>				
Inlet, lb/10 <sup>12</sup> Btu	5.87	7.94	5.83	6.55
Stack, lb/10 <sup>12</sup> Btu	1.11	2.38	1.64	1.71
Removal efficiency, %	81.1%	70.0%	71.9%	73.9%
<b>Particulate mercury</b>				
Inlet, lb/10 <sup>12</sup> Btu	5.42	6.65	4.02	5.37
Stack, lb/10 <sup>12</sup> Btu	ND<0.004	0.006	ND<0.004	ND<0.004
Removal efficiency, %	100.0%	99.9%	100.0%	100.0%
<b>Oxidized mercury</b>				
Inlet, lb/10 <sup>12</sup> Btu	0.35	0.97	1.37	0.90
Stack, lb/10 <sup>12</sup> Btu	0.54	1.57	1.07	1.06
Removal efficiency, %	-54.5%	-61.3%	21.8%	-18.1%
<b>Elemental mercury</b>				
Inlet, lb/10 <sup>12</sup> Btu	0.10	0.31	0.44	0.28
Stack, lb/10 <sup>12</sup> Btu	0.57	0.81	0.57	0.65
Removal efficiency, %	-461%	-160%	-29%	-128%



**Figure 3-1. Mercury Speciation Across ESP**

# 4

## SAMPLING AND ANALYTICAL PROCEDURES

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### 4.1 Test Methods

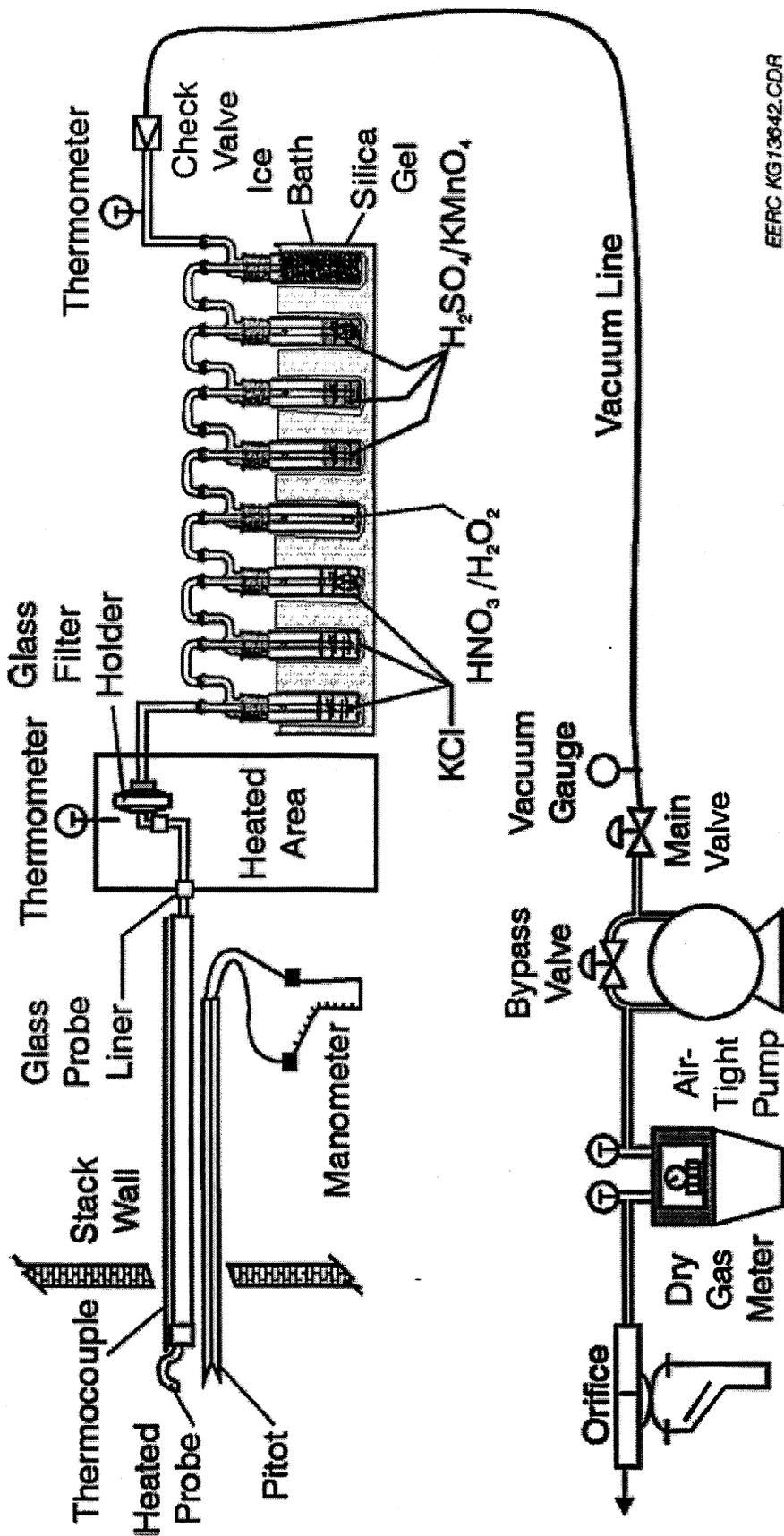
This section contains a summary of the sampling and analytical procedures used to conduct the mercury speciation method required in EPA's ICR titled, "Standard Test Method for Elemental, Oxidized, Particle-Bound and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro 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 27, 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 Meramec 4)

**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 two hour 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 125 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):

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<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> impinger) and rinses;
5. Impingers 5 through 7 (KMnO<sub>4</sub>/H<sub>2</sub>SO<sub>4</sub> 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<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, and KMnO<sub>4</sub> solutions as specified in the method.

#### **KNO<sub>3</sub>-H<sub>2</sub>O<sub>2</sub> Impinger (Container 4)**

The impinger solution is digested using HCl and KMnO<sub>4</sub> solutions as specified in the method.

#### **H<sub>2</sub>SO<sub>4</sub>-KMnO<sub>4</sub> Impingers (Container 5)**

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

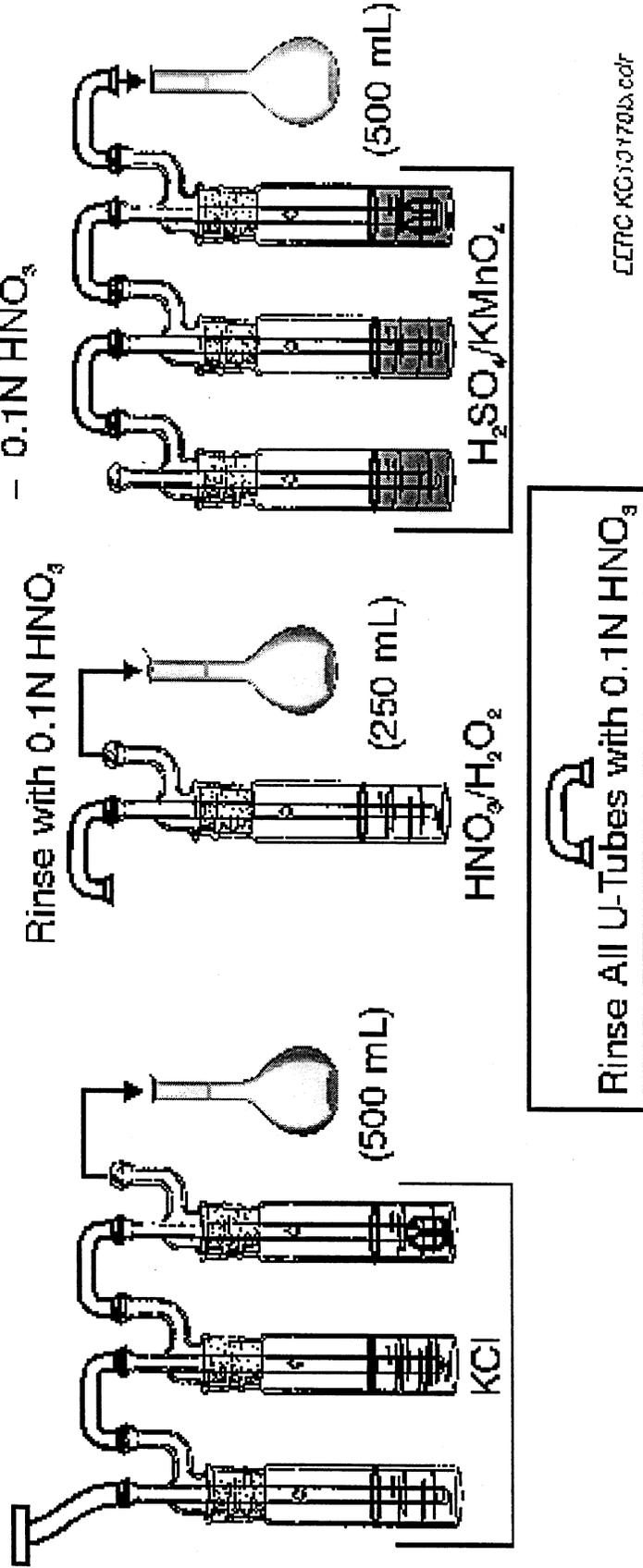
### **Analysis**

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 0.1 N HNO<sub>3</sub>.
2. Add H<sub>2</sub>SO<sub>4</sub>/KMnO<sub>4</sub> to each impinger bottle until purple color remains.
3. Rinse with 0.1 N HNO<sub>3</sub>.
4. Rinse with 8N HCl if brown residue remains.
5. Final rinse with 0.1 N HNO<sub>3</sub>.

Rinse Bottles Sparingly with

- 0.1N HNO<sub>3</sub>
- 8N HCl
- 0.1N HNO<sub>3</sub>



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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 is expected to be well below flue gas levels for most cases if the laboratory uses normal care and state of the art analytical equipment. However, there may be cases where certain fractions of a test do not show detectable mercury levels. This section addresses how non detects were handled in calculating and reporting mercury levels. Note that when example calculations are shown, they are performed using values that are within the expected ranges of measured mercury for coal-fired boilers.

*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 occurred for elemental mercury, which is the sum of the mercury collected in the HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> impinger and the H<sub>2</sub>SO<sub>4</sub>/KMnO<sub>4</sub> impingers. For example, on Test 3-Stack the H<sub>2</sub>O<sub>2</sub> fraction was ND<0.25 µg and the KmaO<sub>4</sub> fraction was 4.7µg. Elemental mercury was reported as 4.7 mg.

*Mercury is detected on one or two of three runs.* If mercury is detected on one or two of three runs, average mercury will be calculated as the average of the detected value(s) and half of the detection limits for the non detect(s).

Example 1. The particulate mercury results for the three stack tests are ND<0.010, 0.015, and ND < 0.010. The reported value was calculated as the average of 0.005, 0.015, and 0.005, which is 0.008 µg.

## ***Auxiliary Flue Gas Measurements***

Auxiliary flue gas measurements performed were flue gas flow rate per EPA Methods 1 and 2 (pitot traverse), O<sub>2</sub> by portable O<sub>2</sub> analyzer (as described below), and H<sub>2</sub>O 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.

### **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 O<sub>2</sub> 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<sub>d</sub>) and carbon (F<sub>c</sub>) F factors.

At the stack, the plant CEMS stack flow rate is presented. At the inlet the pitot traverse results, multiplied by two since only one of two ducts was tested, are presented in Table 3-3.

#### Alternate Methodology for O<sub>2</sub>/CO<sub>2</sub> Determination

As an alternate to conventional Orsat analysis, the following procedure was used for determination of O<sub>2</sub> and CO<sub>2</sub> content.

O<sub>2</sub> determination. O<sub>2</sub> was measured by a portable O<sub>2</sub> 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 O<sub>2</sub> 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 O<sub>2</sub> 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% O<sub>2</sub> 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.

CO<sub>2</sub> determination. CO<sub>2</sub> is used for molecular weight determination. At the stack, CO<sub>2</sub> readings are taken from the plant CEMS. The CEMS values are on a wet basis; dry CO<sub>2</sub> values are calculated using the measured moisture content at the stack.

At the inlet, the CO<sub>2</sub> is calculated via dilution calculations from the inlet O<sub>2</sub>, the stack O<sub>2</sub>, and the stack CO<sub>2</sub>.

#### 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 station personnel to assure that operating conditions were within project target ranges.

# 5

## INTERNAL QA/QC ACTIVITIES

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### 5.1 QA/QC Problems

There were no sampling related QA/QC problems. Sampling operational problems were discussed in Section 3.2. All  $\text{KMnO}_4$  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, except:

1. The target of the results, all runs being within 35% of the mean, was not met for two of the three runs on oxidized mercury at both the inlet and the stack, for one of three runs on elemental mercury at the inlet, and for two of the three runs for total mercury at the stack.

This does not necessarily indicate a problem, just that there was more data scatter than hoped for. The cause could be either process, sampling, or analytical related. Additionally, relative deviations from the mean are higher when emissions are low, which was the case for many of the results at Meramec.

**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**

<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 10 <sup>th</sup> 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. See Section 2.1.
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%	97-102% at inlet 95-102% at stack
Sample volume	1-2.5 std cubic meters	1.2-1.5 m <sup>3</sup> at inlet 2.1-2.4 m <sup>3</sup> 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 1% at inlet W/in 3% at stack
Total mercury for triplicate runs	All runs w/in 35% of mean	Met objective at inlet. One run 37% above mean and one run 37% below mean at stack.
Particulate mercury	All runs w/in 35% of mean	Met objective at inlet and stack.
Oxidized mercury	All runs w/in 35% of mean	One run 55% above mean and one run 61% below mean at inlet. One run 48% above mean and one run 49% below mean at stack.
Elemental mercury	All runs w/in 35% of mean	One run 36% above mean and one run 64% below mean at inlet. Met objective at stack.
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. Meramec 4 Sample Fraction Mercury Measurements**

	Run 2	Run 3	Run 4	Average	Field blank	Field blank/ sample, %
<b>Inlet, µg/sample</b>						
Filter/probe wash (particulate Hg)	9.3	10	5.4	8.2	ND<0.08	ND
KCl fraction (oxidized Hg)	0.6	1.46	1.84	1.3	0.07	5%
H <sub>2</sub> O <sub>2</sub> fraction (elemental Hg)	<0.25	<0.25	<0.25	<0.25	<0.25	ND
KMnO <sub>4</sub> fraction (elemental Hg)	0.175	0.47	0.59	0.4	ND<0.030	ND
<b>Stack, µg/sample</b>						
Filter/probe wash (particulate Hg)	<0.010	0.015	<0.010	0.008	0.011	see note
KCl fraction (oxidized Hg)	1.37	4.05	2.7	2.7	0.071	3%
H <sub>2</sub> O <sub>2</sub> fraction (elemental Hg)	<0.25	<0.25	<0.25	<0.25	<0.25	ND
KMnO <sub>4</sub> fraction (elemental Hg)	1.5	2.1	1.4	1.7	ND<0.030	ND
Note: filter field blank was just above detection limit. Value is not considered significant.						