



**Energy &
Environmental
Research
Center**

**FINAL REPORT
MERCURY SPECIATION SAMPLING FOR THE ICR AT PUBLIC
SERVICE COMPANY OF COLORADO VALMONT STATION**

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EPA

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1.0 INTRODUCTION

1.1 SUMMARY OF TEST PROGRAM

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

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

The test program was performed to meet the requirements of the EPA mercury ICR. EPA selected the test unit based on plant configuration to provide speciated mercury emissions data, which is to be used to develop emission factors for boilers in its class. The intent of the ICR is to provide the EPA with data that can be used to develop speciated mercury emission factors for coal-fired boilers in the United States and to provide information on particulate and SO₂ control device efficiency for collecting mercury. The units to be tested have been selected on the basis of the type of coal burned, type of SO₂ control, and type of particulate control device(s).

As the owner of one of the selected units, Public Service Company of Colorado, through a tailored collaboration with EPRI and the U.S. Department of Energy (DOE), contracted the Energy & Environmental Research Center (EERC) to conduct the required tests at its Valmont Station.

Responsible organizations for this project are:

- Test site operator: Public Service Company of Colorado (Valmont Station)
- Sampling and analytical team: University of North Dakota EERC
- QA/QC oversight: RMB Consulting and Research

The test unit was Valmont Station Unit 1. This unit is operated by Public Service Company of Colorado and is located in Boulder, Colorado, in EPA Region 8. The unit was selected by EPA as part of the following category:

- Fuel type: western bituminous
- SO₂ control type: none
- Particulate control type: baghouse

The Valmont Station consists of one operating electric generating unit. This unit was constructed in the late 1970s and has a generating capacity of 200 MW. This unit has a tangentially fired Combustion Engineering boiler equipped with a 16 compartment reverse-gas baghouse for particulate control. The baghouse operates at a nominal air-to-cloth ratio of 2 ft/min using woven-glass bags. The baghouse provides a particulate collection efficiency of >99.9%. The coal burned at the Valmont Station is a Colorado bituminous coal from the Eagle or 20-Mile mine that is delivered to the station by unit trains. This compliance coal is used to meet the SO₂ emission limits (sulfur content of the coal is 0.45% on a dry basis).

The dates of the testing were November 2–3, 1999. Measurements using the OH speciation method were completed to determine speciated mercury emissions at the inlet of the baghouse (the last air pollution control device) and at the stack. Fuel samples were collected at the coal feeders and analyzed for mercury and chlorine content. In addition, fly ash samples were collected from the baghouse hopper and analyzed for mercury to verify the concentration of particulate-bound mercury.

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

Samples were taken to generate the following data:

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

1.2 KEY PERSONNEL

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

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

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

Organization	Individual	Responsibility	Reports To	Phone Number	Fax Number	E-mail Address
Plant Representative						
PSCo	Mark Fox	Project Manager	NA	(303) 571-7047	(303) 571-7880	markfox@pscoco.com
Project Management						
EPRI	Paul Chu	Project Manager	NA	(650) 855-2812	(650) 855-2002	pchu@epri.com
DOE	Tom Brown	Project Review	NA	(412) 892-4691	(412) 892-5917	Thomas.Brown@fetc.doe.gov
EERC	Dennis Laudal	Principal Investigator	Paul Chu	(701) 777-5138	(701) 777-5181	dlaudal@eerc.und.nodak.edu
EERC Field Team						
EERC	Richard Schulz	Field Manager	Dennis Laudal	(701) 777-5218	(701) 777-5181	rschulz@eerc.und.nodak.edu
EERC	Ray DeWall	Project Chemist	Richard Schulz	(701) 777-5186	(701) 777-5181	rdewall@eerc.und.nodak.edu
EERC	Karen Uhrich	Assistant	Richard Schulz	(701) 777-5191	(701) 777-5181	kuhrich@eerc.und.nodak.edu
EERC	Marlys Heidt	Team Leader	Richard Schulz	(701) 777-5168	(701) 777-5181	mheidt@eerc.und.nodak.edu
EERC	Jeff Thompson	Team Leader	Richard Schulz	(701) 777-5245	(701) 777-5181	jthompson@eerc.und.nodak.edu
EERC	Don Toman	Sample Custodian	Richard Schulz	(701) 777-5227	(701) 777-5181	dtoman@eerc.und.nodak.edu
EERC	Grant Dunham	Assistant Sampler	Richard Schulz	(701) 777-5034	(701) 777-5181	gdunham@eerc.und.nodak.edu
EERC	Steve Evanson	Assistant Sampler	Richard Schulz	(701) 777-5000	(701) 777-5181	---
QA/QC						
RMB	Ralph Roberson	QA/QC Manager	NA	(919) 510-0376	(919) 510-5104	roberson@rmb-consulting.com
EERC	Dave Brekke	EERC QA Manager	EERC Director	(701) 777-5154	(701) 777-5181	dbrekke@eerc.und.nodak.edu

TABLE 1-2
TEST PERSONNEL AND RESPONSIBILITIES

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

The Project QA Manager is Mr. Dave Brekke of the EERC, who is also the EERC's QA Manager. Mr. Brekke has no specific project technical assignments other than QA. As Project QA Manager, he was independent and reported only to the Office of the Director at the EERC. Mr. Ralph Roberson of RMB Consulting and Research provided outside QA services under separate direct contract with EPRI. Activities included review of test plans and procedures as well as provision of blind spike materials for analysis. Mr. Tom Brown from DOE also provided technical review for the project. Figure 1-1 shows the organizational chart.

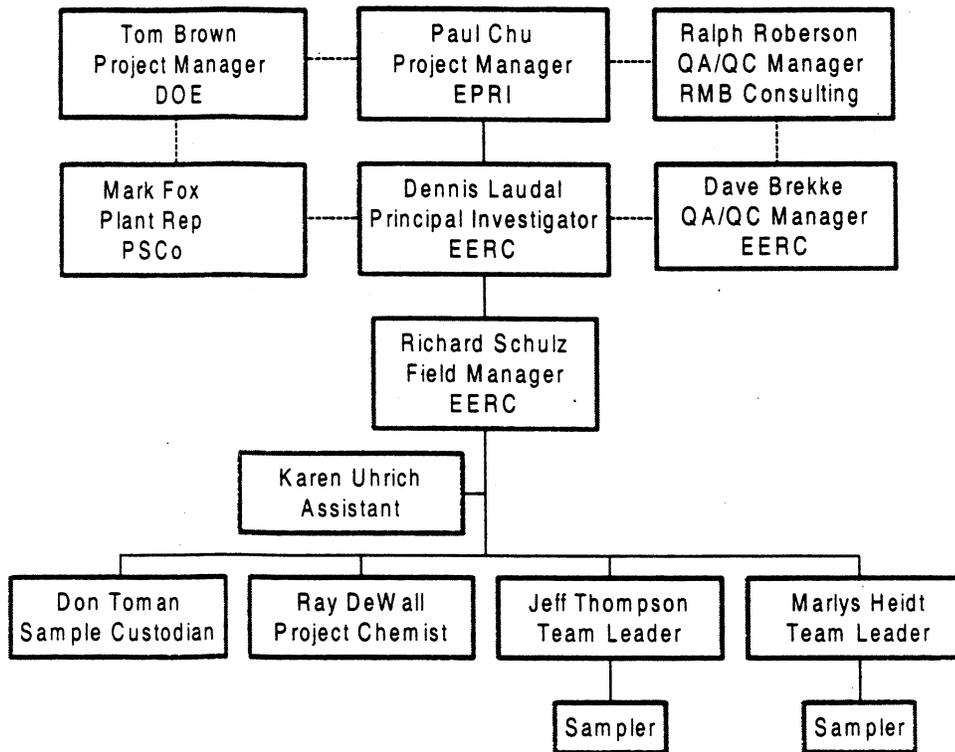


Figure 1-1. Project organizational chart.

2.0 SOURCE AND SAMPLING LOCATION DESCRIPTIONS

2.1 PROCESS DESCRIPTION

Valmont Station has a tangentially fired Combustion Engineering boiler with a generating capacity of 200 MW. Figure 2-1 shows a schematic of the boiler, baghouse, and stack, including sample points. Key unit parameters include the following:

- Unit capacity: 200 MW gross
- Boiler type: tangentially fired Combustion Engineering boiler
- Fuel type: Colorado bituminous coal from the Eagle or 20-Mile mine
- SO₂ control: none
- Particulate control: Baghouse
- NO_x control: none

Fuel samples were collected from the four coal feeders ahead of the boiler; inlet samples were collected at the inlet to the baghouse; and stack samples were collected at the stack. In addition, ash was collected from the baghouse hopper, and mercury CEMs were operated at the stack location. Unit operation during testing was at or near nominal full load (average 179 MW net) at steady-state operation. Coal type, boiler operation, and baghouse operation were all within normal operating ranges.

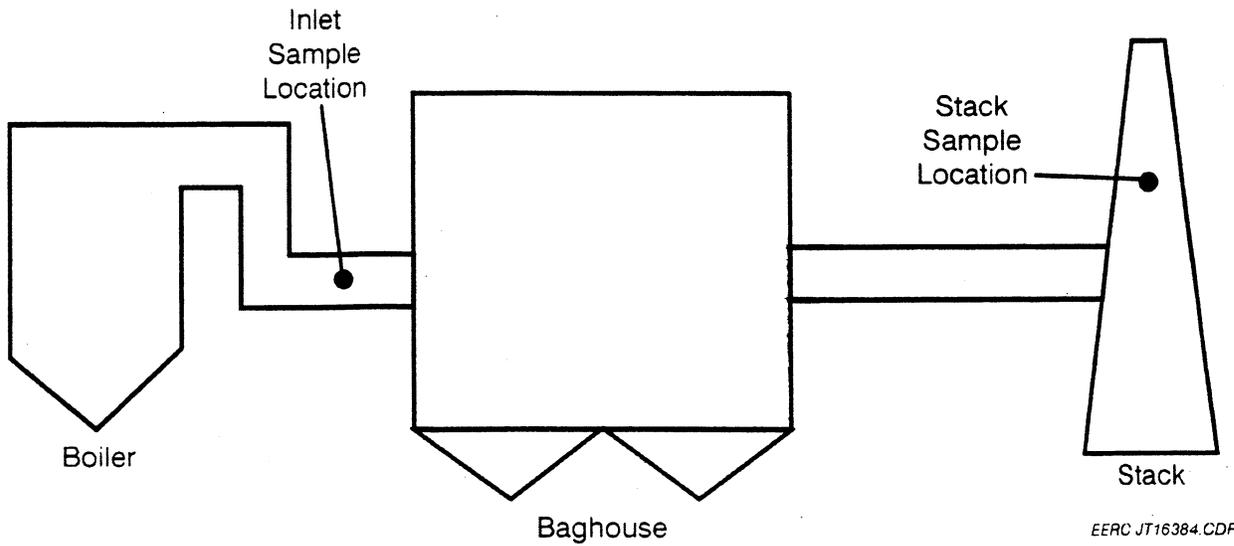


Figure 2-1. Schematic of Valmont Station.

2.2 CONTROL EQUIPMENT DESCRIPTION

Particulate control for the Valmont Station is accomplished using a fabric filter baghouse. The baghouse operates at a nominal air-to-cloth ratio of 2 ft/min using woven-glass bags. The baghouse provides a particulate collection efficiency of >99.9%.

2.3 FLUE GAS SAMPLING LOCATIONS

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

TABLE 2-1
SAMPLING LOCATION DESCRIPTIONS

Description	Baghouse Inlet	Stack
Physical Access	Stairs/ladder	Stairs/ladder
Side or Top Access	Top	Side
Round or Rectangular	Rectangular	Round
Inside Dimensions, ft	26.7 × 15.7	22.2
Equivalent Diameter, ft	19.8	22.2
Number/Type of Ports	7	4
Port Length, ft	1.67	2.25
Port Diameter, ft	0.33	0.33
Nearest Upstream Disturbance	Expansion	Stack Inlet
Distance, ft	3.5	26.7
Distance, equivalent diameters	0.18	1.2
Nearest Downstream Disturbance	Bend	Top of Stack
Distance, ft	3.5	154
Distance, equivalent diameters	0.18	6.9
Typical Flue Gas Conditions		
Temperature, °F	297	284
Moisture, %	9.2	8.1
Flow rate, scfm	NA	443,277
O ₂ , %	NA	3.1
CO ₂ , %	NA	11.7
Particulate Concentration, gr/scf	2.4320	0.0090
SO ₂ , lb/10 ⁶ Btu	NA	0.50
NO _x , lb/10 ⁶ Btu	NA	0.25

2.3.1 Inlet Location

The inlet samples were collected at existing sample ports in the duct at the inlet to the baghouse. A schematic and cross section of the inlet location are shown in Figure 2-2. The sampling location for the inlet to the baghouse does not meet EPA Method 1 criteria because it is located 0.18 equivalent diameters downstream from an expansion in the ductwork and 0.18 equivalent diameters upstream from a bend in the ductwork. The sampling ports are located in a horizontal section of steel ductwork

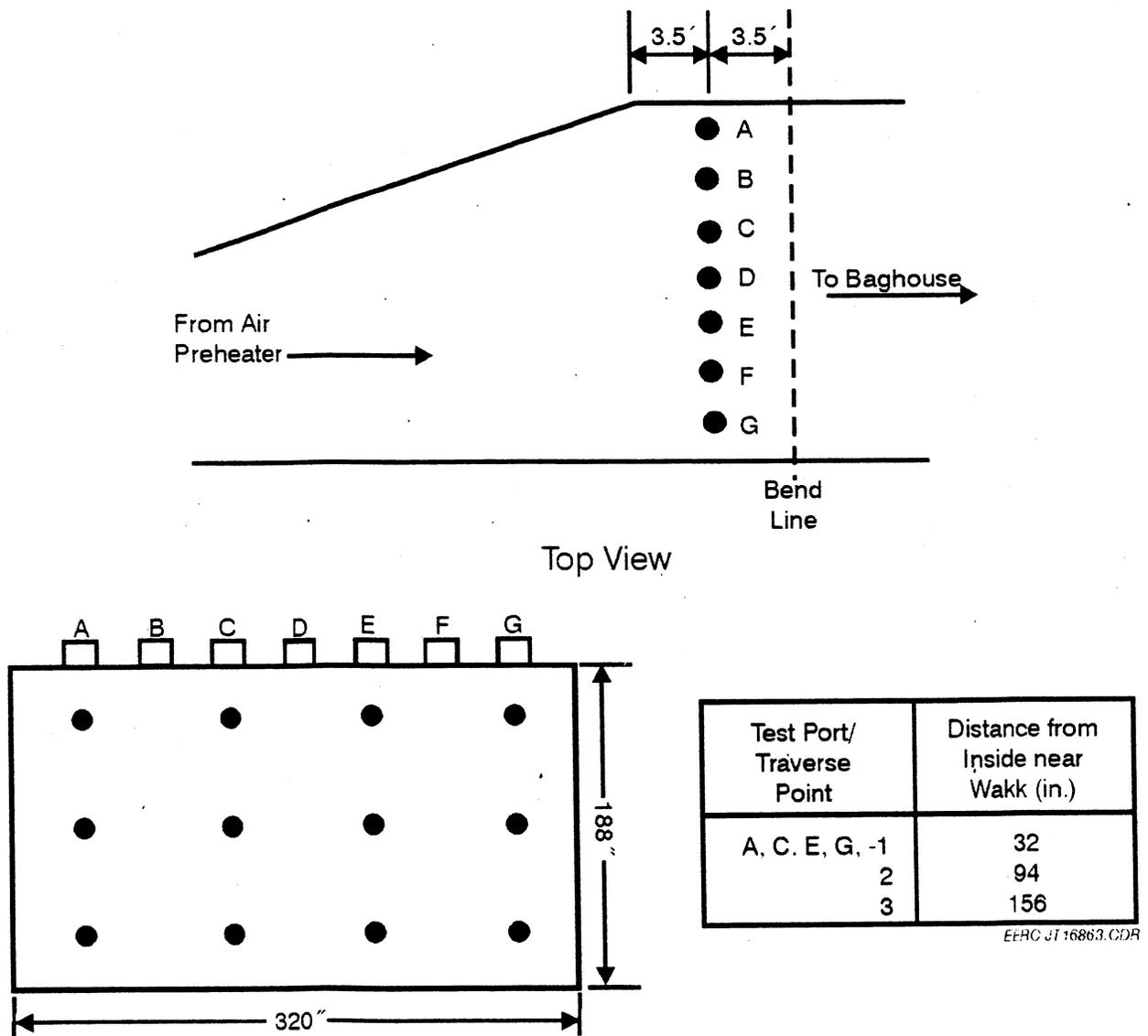


Figure 2-2. Illustration of inlet sampling location.

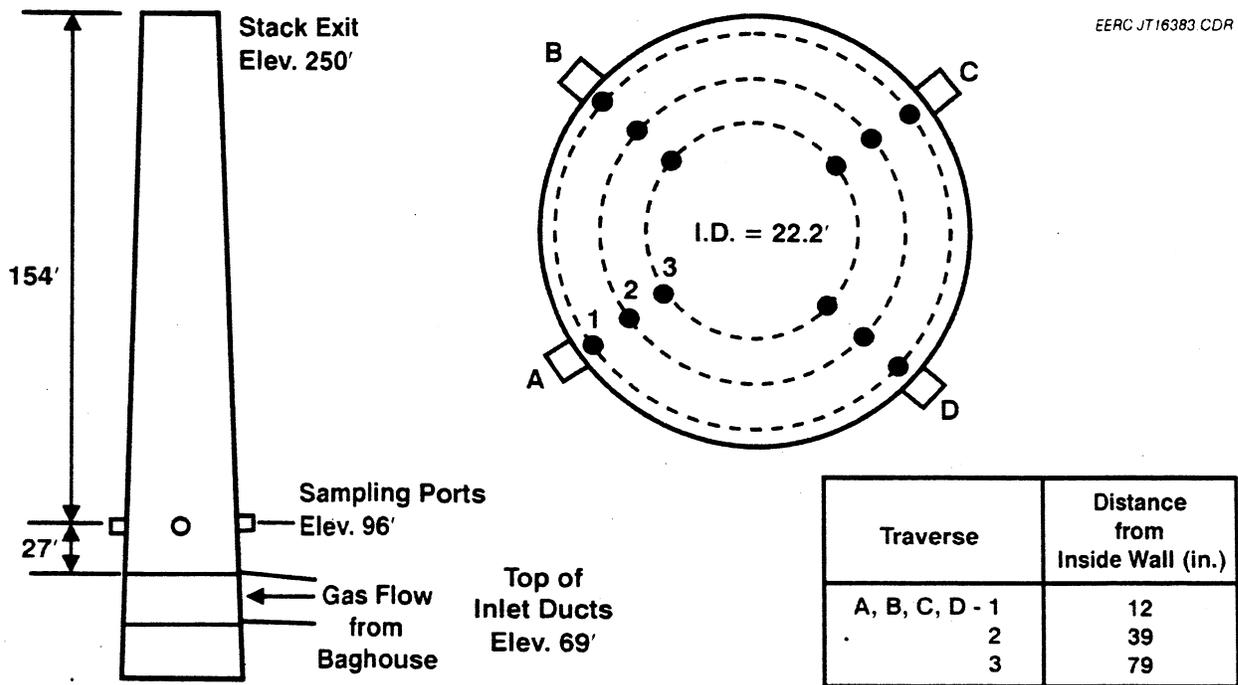


Figure 2-3. Illustration of stack sampling location.

2.4 PROCESS SAMPLING LOCATIONS

Coal samples were collected at the coal feeders to each individual mill. These samples were collected during each of the three flue gas sampling periods. In accordance with ASTM procedures, 16 individual coal samples (four from each mill) were collected during each 2-hour sampling period. These samples were then composited prior to analysis.

Although not required by the ICR, baghouse hopper ash was collected concurrently with the OH method sampling. The samples were mixed to create a composite sample for each test run.

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

A probe was placed in an additional port, not used for the ICR sampling, at the stack location, to sample for the mercury CEMs.

3.0 SUMMARY AND DISCUSSION OF RESULTS

3.1 OBJECTIVES AND TEST MATRIX

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

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

The test matrix is presented in Table 3-1. The table includes a list of test methods used. In addition to speciated mercury, the flue gas measurements included moisture, stack gas flow, and O₂/CO₂. Testing at Public Service Company of Colorado's Valmont Station was carried out over the 2-day period of November 2-3, 1999. Table 3-2 presents the date and time information for each individual run.

3.2 FIELD TEST CHANGES AND PROBLEMS

Field testing was carried out according to the test plan in all aspects except traverse points at the inlet location. The test plan called for a 4 × 5 sample grid at the inlet location. This required the top traverse point to be 19 inches from the inside wall of the duct. Because of using Method 17, in-stack filtration, and the length of the filter housing, the probe could not be placed in the duct at that location. For this reason the sample grid was changed to a 4 × 3 grid to clear the minimum distance

TABLE 3-1
TEST MATRIX FOR MERCURY ICR TESTS AT VALMONT STATION

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

TABLE 3-2
RUN TIMES FOR MERCURY ICR TESTS AT VALMONT STATION

Run No.	1	2	3
Date	11/02/1999	11/02/1999	11/03/1999
Starting Clock Time	1126	1445	1454
Sampling Time, min			
Inlet	120.0	120.0	120.0
Stack	120.0	120.0	120.0

from the inside wall allowed by the filter holder. It was determined that this change would not affect the validity of the sampling or the intent of the ICR.

In addition to the sample grid change, a problem occurred during testing at the inlet location. A faulty thermocouple in the sampling probe used at the inlet location gave incorrect readings. To correct for the problem, the probe heater was continually on, and the probe temperature was manually checked to ensure the minimum temperature of 250°F was maintained. In addition, visual checks of the connection between the probe and the umbilical line were made to ensure that no condensation appeared throughout the testing.

3.3 PRESENTATION OF RESULTS

The results of the testing at Valmont Station are included in the following subsections:

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

3.3.1 Process Data

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

3.3.2 Sampling Data

Sampling data for each of the three runs at the inlet and stack are summarized in Tables 3-4 and 3-5. The tables also include the resulting calculated values according to the appropriate method (EPA Methods 2-5). The target sample volume of 1 to 2.5 m³ was obtained as well as the isokinetic sampling rate range of 90% to 110%. The calculated sample volume used for subsequent mercury calculations is shown in bold. The sampling volume was corrected to standard conditions (68°F, 29.92 in Hg, dry, and 3% O₂).

3.3.3 Flue Gas Mercury Data

The speciated mercury data for the inlet and stack locations are shown in Tables 3-6 and 3-7. The raw mercury data obtained from the analytical lab are included in the appendices. The raw data were

**TABLE 3-3
AVERAGE UNIT PROCESS DATA**

Boiler			
Unit Load, MW gross	181	181	176
Steam Flow, klb/hr	1246	1247	1205
Coal Mills in Service	4	4	4
Coal Flow, lb/hr	155,968	154,911	139,125
Exit Gas Temperature (air heater inlet), °F	729	731	714
Plant CEMs			
CO ₂ , %	12.2	11.8	11.2
O ₂ , %	3.0	3.0	3.2
SO ₂ , lb/10 ⁶ Btu	0.43	0.43	0.64
NO _x , lb/10 ⁶ Btu	0.23	0.25	0.28
Opacity, %	3.31	3.04	2.88
Stack Gas Flow, scfm	422,604	458,007	449,222
Fabric Filter			
Pressure Drop, psi	2.48	2.87	3.47
Gas Inlet Temperature, °F	292	294	311

TABLE 3-4
VALMONT STATION INLET SAMPLING DATA

Run No.	1	2	3
Time, min	120.0	120.0	120.0
Ts, °F	288	290	312
Vm, dcf	67.040	51.474	66.964
Tm, °F	101	94	102
Pm, in. Hg	25.06	24.93	24.96
Ps, in. Hg	24.44	24.32	24.35
An, in. ²	0.0539	0.0415	0.0539
SQRTΔP	0.537	0.516	0.520
H ₂ O, g	148.6	81.2	88.5
Dust, g	8.65225	5.63336	8.96990
CO ₂ , %	14.2	14.2	14.1
O ₂ , %	5.4	5.3	5.5
N ₂ + CO, %	80.5	80.5	80.5
Cm	1.00	1.00	1.00
Vmc, dcf	67.040	51.474	66.964
Vm(std), dscf	52.822	40.864	52.413
Vw(std), scf	7.006	3.829	4.173
Bws	0.1171	0.0857	0.0737
Md, lb/lb-mole	30.5	30.5	30.5
Ms, lb/lb-mole	29.0	29.4	29.5
Vs, ft/sec	39.6	37.9	38.7
I, %	97	99	98
Vm*(std), Nm³	1.301	1.008	1.282

* Corrected to 3% O₂, dry, 68°F, 29.92 in. Hg.

used to calculate the absolute μg of mercury for each fraction (particulate, oxidized, and elemental) of the sample. The mercury speciation data along with the sample volume data were used to calculate the mercury concentration in $\mu\text{g}/\text{Nm}^3$ for each fraction. The coal feed rate and coal analysis (detailed in Section 3.3.6) were used along with the stack gas flow to calculate the emission rates for mercury in $\text{lb}/10^{12}$ Btu. Sample calculations are included in Section 3.3.8. The average and standard deviation data are included in the tables and show the consistency of the test results.

TABLE 3-5
VALMONT STATION STACK SAMPLING DATA

Run No.	1	2	3
Time, min	120.0	120.0	120.0
Ts, °F	274	281	298
Vm, dcf	53.041	51.932	48.760
Tm, °F	73	78	78
Pm, in. Hg	24.94	24.84	24.81
Ps, in. Hg	24.65	24.56	24.55
An, in. ²	0.0507	0.0507	0.0495
SQRTΔP	0.447	0.435	0.424
H ₂ O, g	83.6	83.4	68.9
Dust, g	0.00875	0.06062	0.00478
CO ₂ , %	13.9	13.9	13.8
O ₂ , %	5.4	5.4	5.5
N ₂ + CO, %	80.7	80.7	80.7
Cm	1.00	1.00	1.00
Vmc, dcf	53.041	51.932	48.760
Vm(std), dscf	43.747	42.263	39.679
Vw(std), scf	3.942	3.932	3.249
Bws	0.0827	0.0851	0.0757
Md, lb/lb-mole	30.4	30.4	30.4
Ms, lb/lb-mole	29.4	29.4	29.5
Vs, ft/sec	32.3	31.6	31.1
I, %	98	98	97
Vm*(std), Nm³	1.076	1.038	0.967

* Corrected to 3% O₂, dry, 68°F, 29.92 in. Hg.

3.3.4 Mercury CEM Data

Concurrently with the ICR sampling, a total of four mercury CEMs were also used to obtain gas-phase mercury concentration data at the stack location. The instruments used included two Semtech

TABLE 3-6
VALMONT STATION INLET MERCURY DATA

Run No.	1	2	3	Average	Std. Dev.
Particulate					
μg	1.19	0.93	1.57		
$\mu\text{g}/\text{Nm}^3$	0.92	0.92	1.22	1.02	0.18
$\text{lb}/10^{12}\text{ Btu}$	0.69	0.79	2.95	1.16	0.25
% Total	75.6	82.2	81.2	79.7	
Oxidized					
μg	0.15	0.07	0.14		
$\mu\text{g}/\text{Nm}^3$	0.11	0.07	0.11	0.10 ^a	0.03
$\text{lb}/10^{12}\text{ Btu}$	0.09	0.06	0.11	0.08	0.03
% Total	9.3	5.9	7.5	7.5	
Elemental					
μg	0.24	0.14	0.22		
$\mu\text{g}/\text{Nm}^3$	0.18	0.13	0.17	0.16	0.03
$\text{lb}/10^{12}\text{ Btu}$	0.14	0.12	0.16	0.14	0.02
% Total	15.1	12.0	11.3	12.8	
Total					
$\mu\text{g}/\text{Nm}^3$	1.21	1.12	1.51	1.28	0.20
$\text{lb}/10^{12}\text{ Btu}$	0.92	0.97	1.43	1.10	0.28

^a The variation of mercury concentration between runs was greater than 25%. Because of the low level of mercury, this is not considered to affect the results of the ICR speciation data.

analyzers, a PS Analytical instrument, and a Tekran instrument. A comparison of the gas-phase mercury concentrations measured in $\mu\text{g}/\text{m}^3$ by the mercury CEMs and gas-phase mercury concentrations determined using the OH method (not corrected to 3% O_2) are provided in Figures 3-1 and 3-2. The data obtained with these instruments verify the low concentrations of mercury obtained with the OH method. The two Semtech analyzers were not very useful for these tests because at the concentrations seen, the signal-to-noise ratio was too great. Both the PS Analytical Sir Galahad and the Tekran instruments are inherently more sensitive (because they are fluorescence-

TABLE 3-7
VALMONT STATION STACK MERCURY DATA

Run No.	1	2	3	Average	Std. Dev.
Particulate					
μg	<0.002	<0.002	<0.002		
$\mu\text{g}/\text{Nm}^3$	<0.002	<0.002	<0.002	<0.002	NA
lb/10 ¹² Btu	<0.002	<0.002	<0.002	<0.002	NA
% Total	<1.2	<1.7	<0.9	<1.3	
Oxidized					
μg	0.13	0.10	0.20		
$\mu\text{g}/\text{Nm}^3$	0.12	0.09	0.20	0.14 ^a	0.06
lb/10 ¹² Btu	0.10	0.08	0.19	0.12	0.06
% Total	78.6	86.0	85.2	83.2	
Elemental					
μg	0.04	0.02	0.03		
$\mu\text{g}/\text{Nm}^3$	0.03	0.02	0.04	0.03 ^a	0.01
lb/10 ¹² Btu	0.03	0.01	0.03	0.02	0.01
% Total	21.4	14.0	14.8	16.8	
Total					
$\mu\text{g}/\text{Nm}^3$	0.15	0.11	0.24	0.17^b	0.07
lb/10 ¹² Btu	0.12	0.09	0.22	0.15	0.07

^a The variation of mercury concentration between runs was greater than 25%. Because of the low level of mercury, this is not considered to affect the results of the ICR speciation data.

^b The variation of mercury concentration between runs was greater than 35%. Because of the low level of mercury, this is not considered to affect the results of the ICR speciation data.

based instruments as opposed to absorbance-based), and the results match very well with the OH results. It should be noted that on November 2, 1999, the PS Analytical instrument was not used as it was being moved from the stack location. However, on November 3, 1999, when both instruments were used, the results for the two were nearly identical.

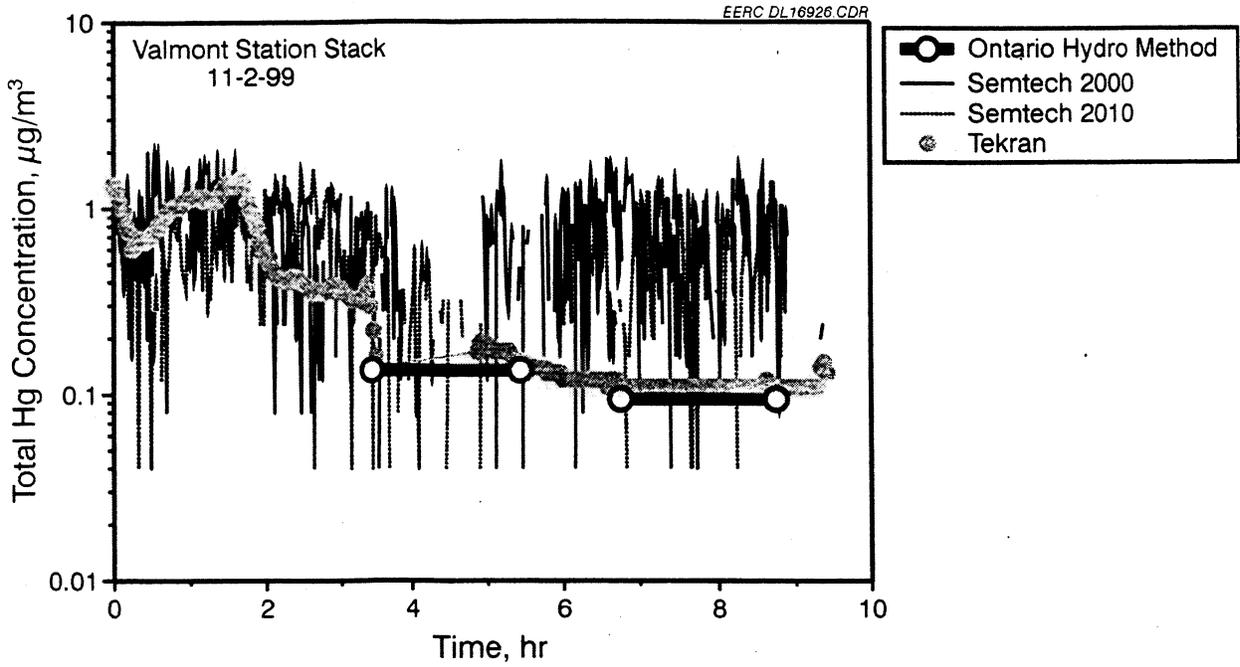


Figure 3-1. Mercury CEM data, Day 1.

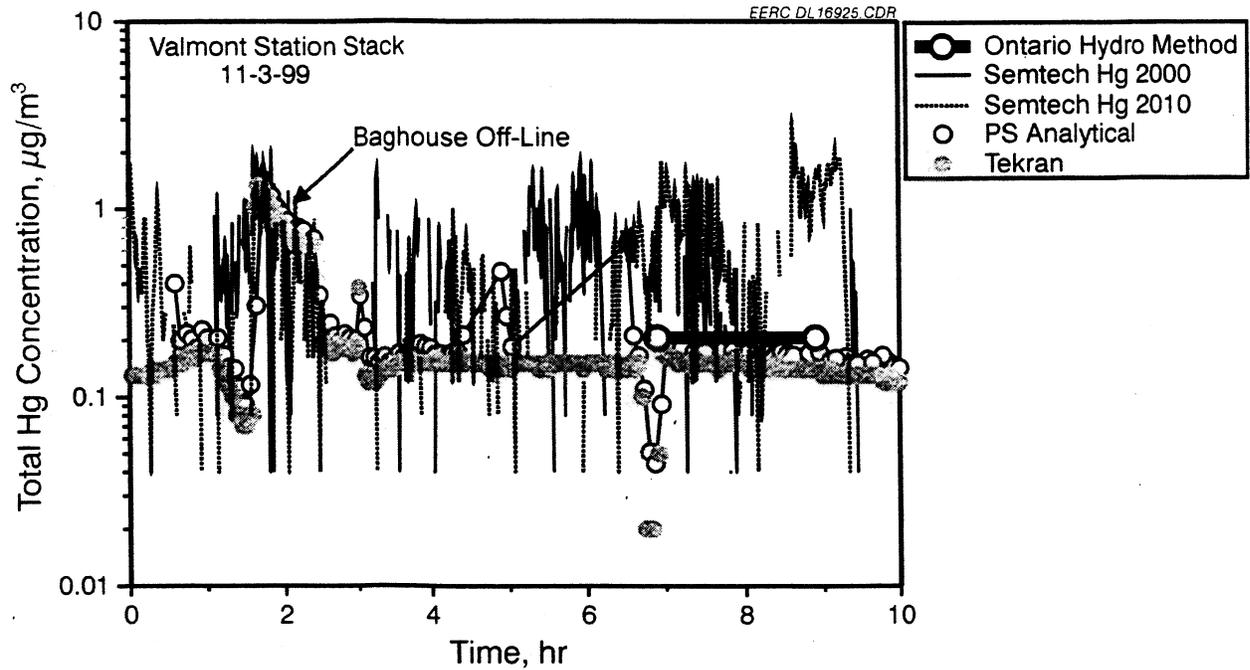


Figure 3-2. Mercury CEM data, Day 2.

The intent was to provide mercury speciation results using the CEMs; however, because of the low concentration of gas-phase mercury emitted at the stack at Valmont Station, the CEMs were only operated in the total mercury analysis mode.

3.3.5 Baghouse Hopper Ash Data

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

3.3.6 Coal Analysis Data

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

3.3.7 Mercury Mass Rates and Removal Efficiencies

Mercury flow rates were calculated for each run, in lb/hr, at each location. The results of these calculations are included in Table 3-10. Although there is a large percentage difference between the rate from the coal and the rate at the inlet, the absolute numbers are low. Therefore, the difference can be attributed to the analytical variability. The removal efficiencies for each species of mercury

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

Run	1	2	3	Average
Baghouse Hopper Ash Hg, $\mu\text{g/g}$	0.132	0.0714	0.141	0.115
OH Particulate Hg, $\mu\text{g/g}$	0.138	0.165	0.175	0.159

**TABLE 3-9
COAL ANALYSIS**

Mercury and Chlorine			
Mercury, ppm (dry)	0.010	0.0055	0.0083
Chlorine (Cl), ppm (dry)	<50	66	<50
Proximate Analysis			
Moisture, %	16.10	16.30	12.10
Volatile Matter, %	35.57	35.01	37.47
Fixed Carbon, %	41.62	41.78	42.72
Ash, %	6.71	6.91	7.70
Ultimate Analysis			
Hydrogen, %	5.66	5.56	5.53
Carbon, %	58.31	57.32	61.54
Nitrogen, %	1.85	1.79	1.97
Sulfur, %	0.25	0.25	0.43
Oxygen, %	27.22	28.18	22.83
Heating Value			
Btu, Btu/lb	10317	10250	10207

were also calculated across the baghouse. The results show good mercury removal (greater than 85%) for the unit.

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

- The mercury level in the coal is low (0.0079 $\mu\text{g/g}$).
- Particulate-bound mercury was removed across the baghouse.
- Greater than 85% of the total mercury was removed across the baghouse.

TABLE 3-10
MERCURY MASS RATES AND REMOVAL EFFICIENCY

Run	1	2	3	Average	Std. Dev.
Mercury Balance					
Coal Feeder, lb/hr	0.0013	0.0007	0.0010	0.0010	0.0003
Inlet, lb/hr	0.0015	0.0015	0.0020	0.0017	0.0003
Stack, lb/hr	0.0002	0.0001	0.0003	0.0002	0.0001
Removal Efficiency					
Particulate-Bound Mercury					
Inlet, lb/10 ¹² Btu	0.69	0.79	1.16	.88	0.25
Stack, lb/10 ¹² Btu	<0.002	<0.002	<0.002	<0.002	NA
Removal, % of total	>75.4	>82.0	>81.1	>79.5	3.6
Oxidized Mercury					
Inlet, lb/10 ¹² Btu	0.09	0.06	0.11	0.08	0.03
Stack, lb/10 ¹² Btu	0.10	0.08	0.19	0.12	0.06
Removal, % of total	-1.1	-2.4	-6.0	-3.2	2.5
Elemental Mercury					
Inlet, lb/10 ¹² Btu	0.14	0.12	0.16	0.14	0.02
Stack, lb/10 ¹² Btu	0.03	0.01	0.03	0.02	0.01
Removal, % of total	12.2	10.6	9.0	10.6	1.6
Total Mercury					
Inlet, lb/10 ¹² Btu	0.92	0.97	1.43	1.10	0.28
Stack, lb/10 ¹² Btu	0.12	0.09	0.22	0.15	0.07
Removal, %	86.8	90.4	84.3	87.1	3.1

3.3.8 Sample Calculations

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

3.3.8.1 Volume of Gas Sample

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

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

$V_m(\text{std}) = 17.64 * 67.040 * 1 * 25.058 / (101 + 460) = 52.822 \text{ dscf}$

Where:

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

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

$P_m =$ Meter pressure (in. Hg)

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

3.3.8.2 Volume of Water Vapor

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

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

$V_w(\text{std}) = 0.04715 * 148.6 = 7.006 \text{ scf}$

Where:

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

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

3.3.8.3 Water Vapor in the Gas Stream

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

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

$B_{ws} = 7.006 / (52.822 + 7.006) = 0.1171$

3.3.8.4 Dry Molecular Weight

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

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

$$\text{Md} = 0.440 * 14.2 + 0.320 * 5.4 + 0.280 * 80.5 = 30.5 \text{ lb/lb-mole}$$

Where:

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

3.3.8.5 Molecular Weight

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

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

$$\text{Ms} = 30.5 * (1 - 0.1171) + 18.0 * 0.1171 = 29.0 \text{ lb/lb-mole}$$

3.3.8.6 Average Stack Gas Velocity

Vs = Average stack gas velocity, ft/sec

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

$$\text{Vs} = 85.49 * 0.84 * 0.537 * [(288 + 460) / (24.44 * 29.0)]^{1/2} = 39.6 \text{ ft/sec}$$

Where:

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

Cp = Pitot tube coefficient, dimensionless

Δp = Velocity head of stack gas (in. Hg)

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

Ps = Stack pressure (in. Hg)

3.3.8.7 Isokinetic Sampling Rate

I = Percent of isokinetic sampling, %

$$I(\%) = K_4 * (Ts + 460) * Vm(std) / (Ps * Vs * An / 144 * \theta * (1 - Bws))$$

$$I = 0.09450 * (288 + 460) * 52.822 / (24.44 * 39.6 * 0.0539 / 144 * 120 * (1 - 0.1171)) = 97\%$$

Where:

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

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

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

3.3.8.8 Volume of Gas Sample Corrected to 3% O₂

Vm*(std) = Volume of gas sample measured by the dry gas meter (Vm(std)), * corrected to 3% oxygen, Nm³

$$Vm*(std) = Vm(std) * (21 - \%O_2) / 18 * K_5$$

$$Vm*(std) = 52.822 * (21 - 5.35) / 18 * 0.02832 = 1.301 \text{ Nm}^3$$

Where:

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

3.3.8.9 Mercury

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

$$Hg = 0.138 * 8.65225 = 1.19 \mu\text{g} \text{ (using the particulate mercury data from Run 1)}$$

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

$$Hg = 1.194 / 1.301 = 0.92 \mu\text{g/Nm}^3$$

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

Oxidized Hg = Sum of mercury from KCl impingers

Elemental Hg = Sum of mercury from H₂O₂ and KMnO₄ impingers (note: all H₂O₂ impinger values were nondetects). Since typically less than 5% of the elemental mercury is trapped in the H₂O₂ impinger, the less than values were not added to the total elemental mercury. Thus the elemental mercury was calculated from the values obtained from the KMnO₄ impingers only.

3.3.8.10 Mass Rates

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

$$\text{Hg (lb/hr) from coal} = 155,968 * (1 - 0.1610) * 0.010 / 10^6 = 0.0013 \text{ lb/hr}$$

Where:

Cf = Coal feed rate (lb/hr)

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

Bc = Coal moisture (fraction)

$$\text{Hg (lb/hr) in flue gas} = \text{Hg}(\mu\text{g}/\text{Nm}^3) * \text{Sf} * (21 - \% \text{O}_2) / 18 * (1 - \text{Bws}) * \text{K}_6$$

$$\text{Hg (lb/hr) in flue gas} = 1.214 * 422,604 * (21 - 5.35) / 18 * (1 - 0.1171) * 3.745 \times 10^{-9} = 0.0015 \text{ lb/hr}$$

Where:

K₆ = 3.745 x 10⁻⁹ (lb/μg)(m/hr)(m³/ft³)

Sf = Stack gas flow (scfm)

3.3.8.11 Emission Rate

$$\text{Hg (lb}/10^{12} \text{ Btu)} = \text{Hg}(\mu\text{g}/\text{Nm}^3) * \text{Sf} * (1 - \text{Bws}) * (21 - \% \text{O}_2) / 18 / \text{Cf} / \text{Hv} * \text{K}_7$$

$$\text{Hg (lb}/10^{12} \text{ Btu)} = 1.214 * 422,604 * (1 - 0.1171) * (21 - 5.35) / 18 / 155,968 / 10,317 * 3745 = 0.92 \text{ lb}/10^{12} \text{ Btu}$$

Where:

$$K_7 = 3745 (m^3/ft^3)(m/hr)(lb/\mu g)(Btu/10^{12} Btu)$$

$$H_v = \text{Heating value (Btu/lb)}$$

4.0 SAMPLING AND ANALYTICAL PROCEDURES

4.1 TEST METHODS

4.1.1 Flue Gas Mercury Speciation

This section contains a summary of the sampling and analytical procedures used to conduct the mercury speciation method required in EPA's ICR entitled *Standard Test Method for Elemental, Oxidized, Particle-Bound and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method)* dated May 1999. For this method, a sample is withdrawn from the flue gas stream isokinetically through the filtration system, which is followed by a series of impingers in an ice bath. Particulate-bound mercury is collected on the front half and filter; oxidized mercury is collected in impingers containing 1 N potassium chloride solution; and elemental mercury is collected in one impinger containing a 5% nitric acid and 10% peroxide solution and in three impingers containing a solution of 10% sulfuric acid and 4% potassium permanganate. An impinger containing silica gel collects any remaining moisture. Quartz fiber filters were used as the filter media for the testing, and the filter holder was glass. A heated Teflon line was used between the probe and impinger train. A target sampling time of 2 hours was used, with a target sample volume of 1 to 2.5 standard cubic meters. A schematic of the sample train is shown in Figure 4-1. Table 4-1 presents a list of sample train components for the EPA Method 17 configuration. Because the flue gas temperature was greater than 120°C (248°F) at both the inlet and stack sampling locations, in-stack filtration (EPA Method 17 configuration) was used.

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

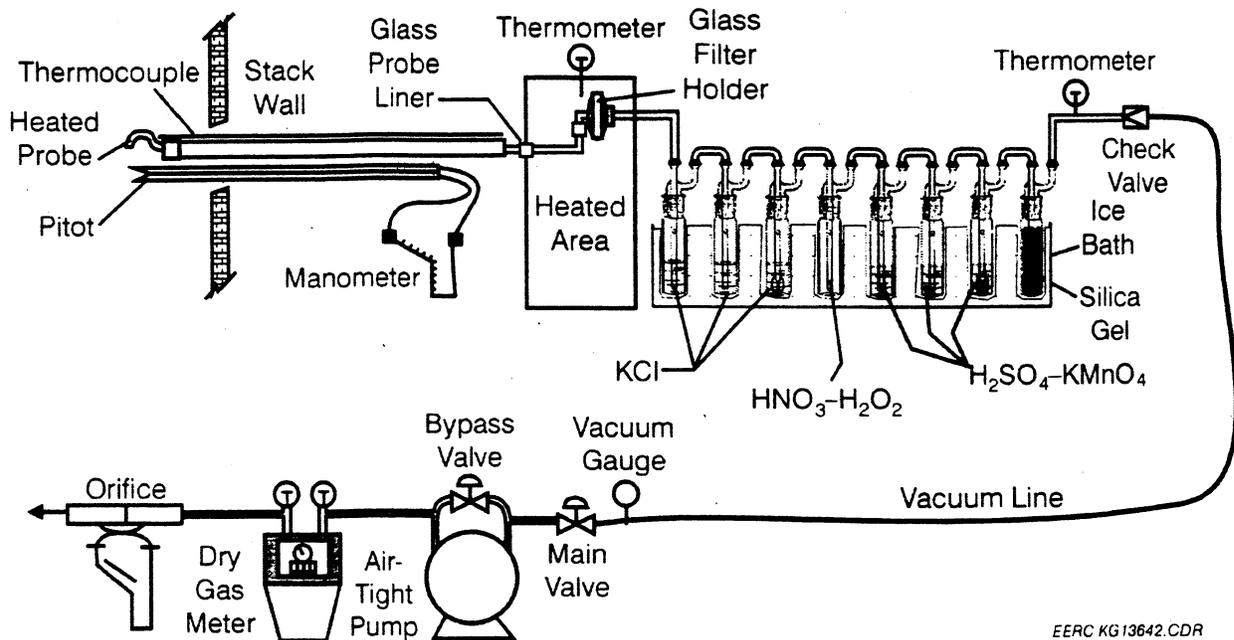


Figure 4-1. Schematic of mercury speciation sample train (Method 5 option is shown; Method 17 in-stack is also allowed).

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

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

1. Rinse filter holder and connector with 0.1N HNO₃.
2. Add 5% w/v KMnO₄ to each impinger bottle until purple color remains.
3. Rinse with 10% v/v HNO₃.
4. Rinse with a very small amount of 10% w/v NH₂OH·H₂SO₄ if brown residue remains.
5. Final rinse with 10% v/v HNO₃.

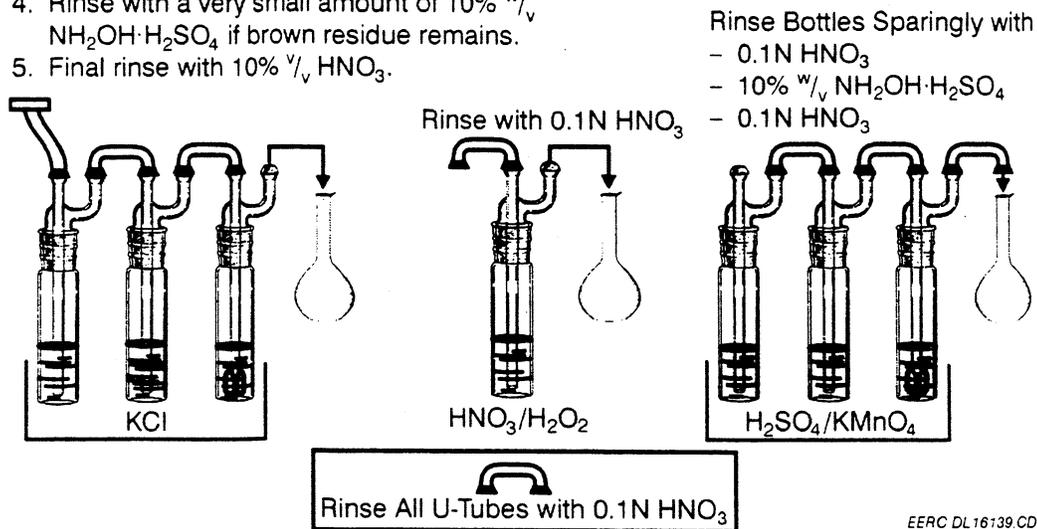


Figure 4-2. Sample recovery scheme for the mercury speciation sampling train.

- Container 1 – the sample filter
- Container 2 – the front half rinse (includes all surfaces upstream of the filter)
- Container 3 – Impingers 1 through 3 (KCl impingers) and rinses
- Container 4 – Impinger 4 (HNO₃-H₂O₂ impinger) and rinses
- Container 5 – Impingers 5 through 7 (H₂SO₄-KMnO₄ impingers) and rinse
- Silica Gel – Impinger 8 (silica gel impinger). Note that this sample is weighed for moisture determination and is not included in the mercury analysis.

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

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

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

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

4.1.2 Fuel Mercury and Chlorine

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

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

4.1.3 Auxiliary Flue Gas Measurements

EPA methods for auxiliary flue gas measurements included flue gas flow rate using EPA Methods 1 and 2 (pitot traverse), O₂ and CO₂ by EPA Method 3A (portable O₂ analyzer), and moisture by EPA Method 4 (condensation/gravimetric analysis). These measurements were collected as integral parts of all mercury speciation test runs at both the inlet and stack locations. EPA Reference Method 5/17 requirements were followed for isokinetic sampling.

4.1.4 Mercury CEM Measurements

4.1.4.1 PS Analytical Sir Galahad

The Sir Galahad analyzer was initially used to monitor total mercury continuously in the urban environment and in natural gas, but it can also be used in a variety of gaseous media including combustion flue gas. The analyzer is based on the principle of atomic fluorescence which provides an inherently more sensitive signal than atomic absorption. The system uses a gold-impregnated silica support for preconcentrating the mercury and separating it from potential interferences that degrade sensitivity.

The Sir Galahad requires a four-step process to obtain a flue gas mercury measurement. In the first step, 2 L of flue gas is pumped through a gold trap which is maintained at a constant temperature. Before the mercury is desorbed from the gold trap, a flushing step is initiated to remove any flue gas that may be present because it has a damping effect on the mercury fluorescence. When this is completed, the analysis step begins. The heating coil is activated, and the gold trap is heated to approximately 500°C. This desorbs the mercury from the trap, and the mercury is carried into the fluorescence detector. The gold trap is rapidly cooled by pumping argon over it, in preparation for the next sample. The total time for the entire process is about 5 minutes.

The system was calibrated using Hg⁰ as the primary standard. The Hg⁰ is contained in a closed vial which is held in a thermostatic bath. The temperature of the mercury is monitored, and the amount

of mercury is calculated using vapor pressure calculations. Typically, the calibration of the unit has proven stable over a 24-hr period.

4.1.4.2 *Semtech Hg 2000 and Hg 2010*

The commercial Semtech Hg 2000 and Hg 2010 mercury analyzers (Semtech Metallurgy AB, Lund, Sweden) are essentially portable Zeeman-modulated CVAAS instruments that can monitor Hg⁰ continuously. The flue gas was converted and conditioned with a separate unit, and the conditioned dry gas was then analyzed using the Semtech Hg 2000 analyzer. The analyzer uses Zeeman effect background correction, by applying a modulated magnetic field to a mercury lamp, to minimize interferences from the presence of SO₂, hydrocarbons, and fine particulate in the flue gas sample. The operating range of the analyzer is 0.3 µg/Nm³ to 20 mg/Nm³ Hg⁰, as specified by Semtech Metallurgy AB. The Semtech Hg 2000 has also been certified by TUEV Rheinland for determining compliance with the German legal limit of 50 µg/Nm³ for total mercury from waste incinerators.

4.1.4.3 *Tekran Model 2537A*

The Tekran mercury CEM has been the standard instrument for measuring ambient mercury concentrations for the past 10 years. With the development of a pretreatment/conversion system by the EERC, the instrument was used to measure mercury flue gas concentrations at the Valmont Station. The analyzer is similar to the PS Analytical Sir Galahad in that it is based on the principle of atomic fluorescence, which provides an inherently more sensitive signal than atomic adsorption. The instrument has a detection limit of 0.1 ng/m³ with a maximum limit of about 100 µg/m³. The instrument collects the mercury on a gold trap which preconcentrates the mercury and separates it from potential interferences.

Although the measurement is a batch process using a dual gold cartridge design, the instrument can provide mercury data in approximately 2.5 minutes. This is done by collecting mercury on one trap while the mercury from the second trap is being desorbed at approximately 500°C into the detector. The gold trap is rapidly cooled by passing argon over it.

An internal elemental mercury permeation source is used to calibrate the instrument. This is done automatically at preset time intervals. It can also be calibrated manually using standard gastight syringes.

4.2 PROCESS TEST METHODS

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

TABLE 4-2
PROCESS DATA COLLECTED

Boiler Data Unit Load, MW net Steam Flow, klb/hr Number of Coal Mills in Service Coal Flow, tons/hr Exit Gas Temperature, °F
CEM Data CO ₂ , % wet or dry SO ₂ , lb/10 ¹² Btu NO _x , lb/10 ¹² Btu (record NO ₂ if available) Opacity, % Stack Gas Flow, klb/hr
Fabric Filter Pressure Drop in Water Column Gas Inlet Temperature, °F

5.0 QA/QC ACTIVITIES

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

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

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

TABLE 5-2
RESULTS EVALUATION AND VERIFICATION CHECKLIST

Measure	Objective	Result
Unit Information		
Unit Operating Conditions	No unusual conditions	No unusual conditions
Air Pollution Control Device Operation	No unusual conditions	No unusual conditions
Sample Train Information		
Trains Leak-Checked Before/After Each Test	<0.02 cfm	<0.02 cfm
Pitot Probe Leak-Checked	Zero leakage	Zero leakage
Filter Temperature Maintained	Minimum 120°C	NA Method 17 used
Sample Isokinetics	90%–110%	90%–110%
Sample Volume	1 to 2.5 standard cubic meters	1 to 2.5 standard cubic meters
Posttest Color of Permanganate Impingers	Purple	Purple
Results		
Flow Rate for Triplicate Runs	All runs w/in 10% of mean (adjusted for load if necessary)	All runs w/in 10% of mean
Moisture for Triplicate Runs	All runs w/in 3% of mean	The high variability of the moisture in the coal ($\pm 20\%$) resulted in greater than $\pm 3\%$ variation of moisture in the flue gas. This is not considered to affect the results of the mercury speciation data, nor the intent of the ICR.
Stack Temperature for Triplicate Runs	All runs w/in 5% of mean	All runs w/in 5% of mean
Mercury for Triplicate Runs	All runs w/in 35% of mean	All runs w/in 35% of mean at the inlet. The low level of mercury at the stack resulted in greater variation than 35% of mean. The data were flagged.
% of Mercury as Particulate-Bound	All runs w/in 25% of mean	All runs w/in 25% of mean at the inlet location. The particulate mercury at the stack was a nondetect.
% of Mercury in Oxidized Form	All runs w/in 25% of mean	The low level of oxidized mercury resulted in greater variation than 25% of mean. The data were flagged.
% of Mercury in Elemental Form	All runs w/in 25% of mean	All runs w/in 25% of mean at inlet location. The low level of elemental mercury at the stack resulted in greater variation than 25% of mean. The data were flagged.

5.1 ACCURACY

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

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

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

TABLE 5-3
REAGENT BLANK ANALYSIS RESULTS

Reagent	Hg, $\mu\text{g/L}$
KCl Reagent Blank	<0.03
H ₂ O ₂ Reagent Blank	<0.03
KMnO ₄ Reagent Blank	<0.03
10% Hydroxylamine HCl Blank	<0.03
0.1 N HNO ₃ Blank	<0.03

TABLE 5-4
FIELD BLANK ANALYSIS RESULTS

Sample ID	Sample Type	Volume, mL	Hg, $\mu\text{g/L}$
VAL-D2-FB-IN	KCl	500	<0.03
VAL-D2-FB-IN	H ₂ O ₂	250	<0.03
VAL-D2-FB-IN	KMnO ₄	500	<0.03
VAL-D2-FB-OUT	KCl	500	<0.03
VAL-D2-FB-OUT	H ₂ O ₂	250	<0.03
VAL-D2-FB-OUT	KMnO ₄	500	<0.03
VAL-D3-FB-IN	KCl	500	<0.03
VAL-D3-FB-IN	H ₂ O ₂	250	<0.03
VAL-D3-FB-IN	KMnO ₄	500	<0.03
VAL-D3-FB-OUT	KCl	500	<0.03
VAL-D3-FB-OUT	H ₂ O ₂	250	<0.03
VAL-D3-FB-OUT	KMnO ₄	500	<0.03

5.2 PRECISION

The precision target for the program was $\pm 10\%$. Every tenth sample was analyzed in triplicate. The results of the triplicate analyses for the Valmont Station testing are shown in Table 5-6. In all cases, the data fall within $\pm 10\%$ of the mean. The duplicate analysis of all samples are included in Appendix B.

5.3 COMPLETENESS

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

TABLE 5-5
ANALYTICAL SPIKES

Sample ID	Type	Spike Amount, $\mu\text{g/L}$	Sample Value, $\mu\text{g/L}$	Spike Reading, $\mu\text{g/L}$	Recovery, %
KCl Reagent Blank	KCl	5	<0.03	5.14	102.8
KCl Reagent Blank	KCl	10	<0.03	9.83	98.3
VAL-D2-IN-OH-1	KCl	5	<0.03	5.53	110.6
VAL-D2-IN-OH-1	KCl	10	<0.03	10.56	105.6
VAL-D2-FB-SPIKE	KCl	5	<0.03	5.3	106.0
VAL-D2-FB-SPIKE	KCl	10	5.3	16.1	108.0
VAL-D2-IN-OH-1	H ₂ O ₂	5	<0.03	4.97	99.4
VAL-D2-IN-OH-1	H ₂ O ₂	10	<0.03	10.61	106.1
VAL-D2-FB-SPIKE	H ₂ O ₂	2	<0.03	1.74	87.0
VAL-D2-FB-SPIKE	H ₂ O ₂	10	1.74	12.95	112.1
VAL-D2-IN-OH-1	KMnO ₄	5	0.256	5.56	106.1
VAL-D2-FB-SPIKE	KMnO ₄	5	<0.03	4.73	94.6
VAL-D2-FB-SPIKE	KMnO ₄	5	4.73	9.98	105.0
VAL-D3-FB-SPIKE	KCl	5	<0.03	5.48	109.6
VAL-D3-FB-SPIKE	KCl	10	5.48	16.1	106.2
VAL-D3-IN-OH-2	KCl	5	<0.03	5.73	114.6
VAL-D3-IN-OH-2	KCl	10	<0.03	10.51	105.1
VAL-D3-FB-SPIKE	H ₂ O ₂	5	<0.03	4.52	90.4
VAL-D3-FB-SPIKE	H ₂ O ₂	10	4.52	14.72	102.0
VAL-D3-FB-SPIKE	KMnO ₄	5	<0.03	5.05	101.0
VAL-D3-FB-SPIKE	KMnO ₄	5	5.05	10.3	105.0

TABLE 5-6
RESULTS FROM TRIPPLICATE ANALYSES

Sample ID	Sample Type	Hg Concentration, $\mu\text{g/L}$		
VAL-D2-IN-OH-1	KCl Nos. 1 and 2	0.292	0.297	0.295
VAL-D2-IN-OH-1	H_2O_2	<0.03	<0.03	<0.03
VAL-D2-IN-OH-1	KMnO_4	0.480	0.479	0.468
VAL-D3-IN-OH-1	KCl	0.107	0.129	0.122
VAL-D3-IN-OH-1	H_2O_2	0.07	0.09	0.10