



MidAmerican Energy Company
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712 277-7500 Telephone

December 14, 1999

Mr. William Grimley
Emission Measurement Center (MD-19)
U.S. Environmental Protection Agency
Research Triangle Park, North Carolina 27711
Attn: Electric Utility Steam Generating Unit Mercury Test Program

Dear Mr. Grimley:

Enclosed please find two bound copies and 1 unbound copy of the Mercury Emission Test Report for the mercury testing conducted on MidAmerican Energy Company's, George Neal South, Unit 4. Should you have any questions concerning this report, please call me at (712)277-7461.

Sincerely,

A handwritten signature in cursive script that reads "Donald E. Mohning".

Donald E. Mohning
Senior Environmental Coordinator

enclosures

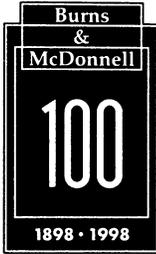
cc T. R. Davis
- S. C. Guyer

MERCURY EMISSIONS TEST REPORT
for
MIDAMERICAN ENERGY
at their
GEORGE NEAL SOUTH STATION
Unit 4

Sioux City, Iowa
September 1999

99-343-3

Prepared by
Burns & McDonnell
Engineers – Architects – Consultants
Kansas City, MO



December 9, 1999

I, Russell D. Fowler, Jr., hereby certify that the mercury emissions testing conducted on Unit 4, at MidAmerican Energy Company's George Neal Station in Sioux City, Iowa was in accordance with procedures established by the United States Environmental Protection Agency. This report accurately and faithfully represents the data obtained from this test and the results determined from analysis of this data.

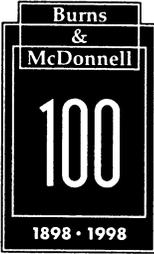
Russell D. Fowler, Jr.

Russell D. Fowler, Jr.
Source Emissions Testing
Senior Test Crew Chief
Energy Division

I, Carl Weilert, hereby attest that I have reviewed the data and the results of the test program and this report accurately presents the results from the testing performed.

Carl V. Weilert

Carl Weilert
Project QA/QC Manager
Energy Division



I, Joel Iserman – Testing Department Manager, hereby attest that all work on this project was done under my supervision and this report accurately presents the results from the testing performed.

A handwritten signature in black ink that reads "Joel R. Iserman". The signature is written in a cursive style and is positioned above a horizontal line.

Joel R. Iserman
Source Emissions Testing
Department Manager
Energy Division

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1.0 INTRODUCTION

1.1 Summary of Test Program

The US Environmental Protection Agency (EPA) requested that specific steam electric generating units conduct speciated mercury emission tests in order to provide additional background information. This background information is being used to determine if it is necessary to regulate mercury emission as a hazardous air pollutant.

The EPA chose MidAmerican Energy Company's George Neal South Station, Unit 4 as being representative of its type within the industry. MidAmerican contracted Burns & McDonnell to conduct the emission testing required.

The project consisted of performing simultaneous inlet and outlet testing of the Electrostatic Precipitator (ESP) serving unit 4 at the George Neal South Station using the Ontario-Hydro Method. Testing was performed September 14, 1999.

MidAmerican Energy's George Neal South Station is located at 2761 Port Neal Circle, Sioux City, Iowa, approximately three (3) miles west of Salix, Iowa.

1.2 Key Personnel

Figure 1-1 presents the test program organization chart. Names and phone numbers of each responsible party are included. The following list identifies personnel and tasks associated throughout their respective period of responsibility for this project.

Facility Coordinator

Mr. Don Mohning

Major Responsibilities

- 1) Coordinate unit readiness between unit operators and project manager.
- 2) Assure the collection of coal samples and unit operating data during the test.
- 3) Provide project manager with any correspondence received from EPA concerning this project.

Project Manager

Mr. Joel Iserman – Burns & McDonnell

Major Responsibilities

- 1) Preparation of the QAPP and Site Specific Test Plan.
- 2) Liaison between all parties involved.
- 3) Assure acceptability of the test locations.
- 4) Oversee and review the test report.

Project QA Manager

Mr. Carl Weilert – Burns & McDonnell

Major Responsibilities

- 1) Review of QAPP for completeness.
- 2) Review of equipment calibration.
- 3) Review of final report.
- 4) Report any discrepancies to the project manager.

Field Manager

Mr. Russell Fowler – Burns & McDonnell

Major Responsibilities

- 1) Oversee calibration of equipment.
- 2) On-site project coordinator during field sampling.
- 3) Preparation of final test report.

Laboratory Activities

Mr. Russell D. Fowler, Jr.

Major Responsibilities

- 1) Provide coordination between field manager and subcontracting laboratory.
- 2) Preparation of pre/post test of sample media.
- 3) Shipment of samples to subcontracting laboratory.

Data Management

Mr. Russell D. Fowler, Jr.

Major Responsibilities

- 1) Preparation of QAPP site specific test plan.
- 2) Oversee data verification to ensure reporting accuracy.
- 3) Review final test reports and report any discrepancies to the project manager.
- 4) Oversee timely delivery of test reports.

Subcontracting Analytical Laboratory

Philip Analytical Services – Mr. Ron McLeod

Major Responsibilities

- 1) Provide SOP for inclusion in the QAPP.
- 2) Provide calibration data and documentation to project QA/QC manager for review.
- 3) Provide analysis of sample media.

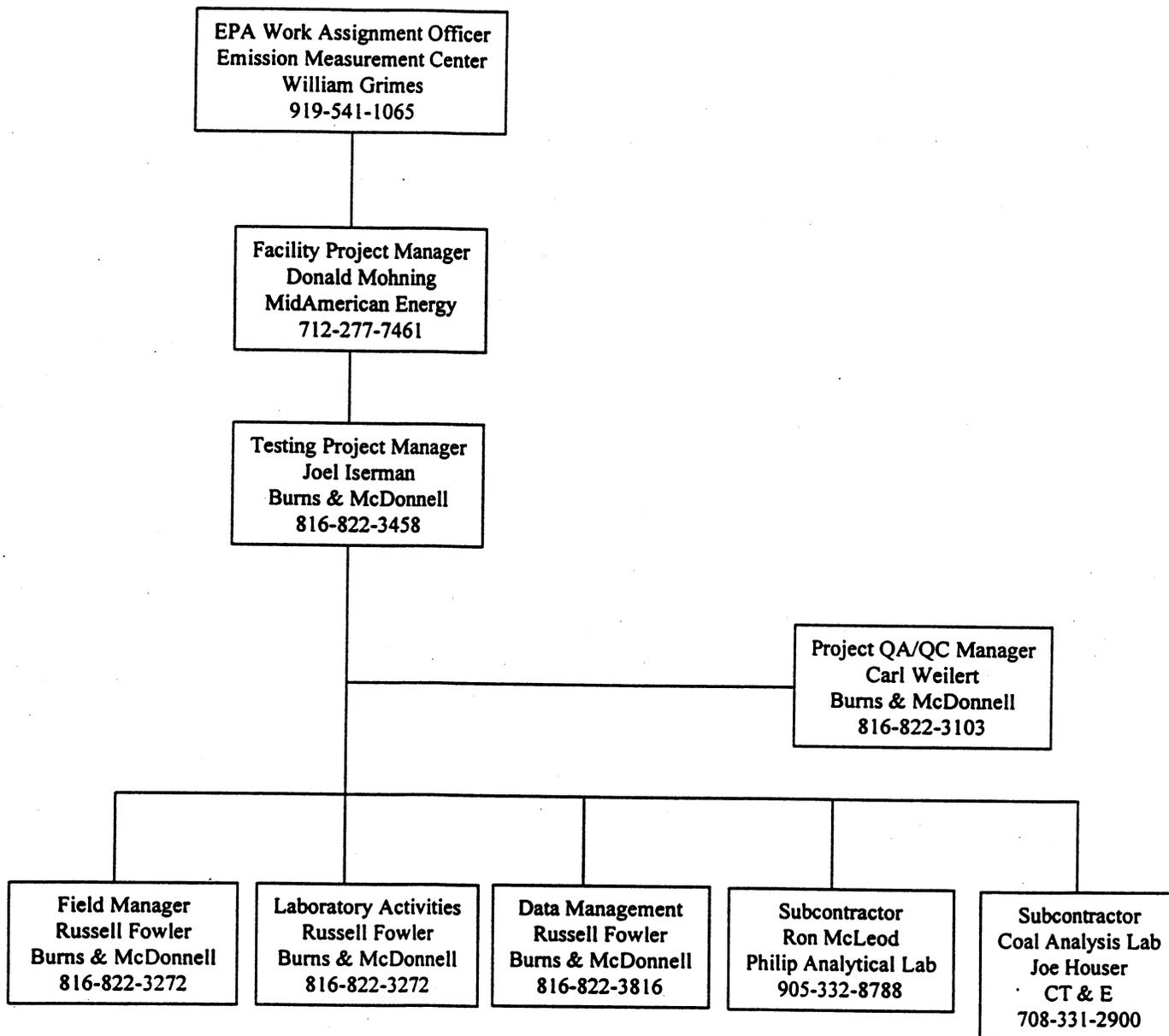
Subcontracting Coal Analytical Laboratory

Commercial Testing and Engineering – Mr. Joe Houser

Major Responsibilities

- 1) Provide analysis of coal samples.
- 2) Provide Calibration Data and documentation to project QA/QC Manager for review.

Figure 1-1
Organizational Chart and Lines of Communication



2.0 SOURCE AND SAMPLING LOCATION DESCRIPTION

2.1 Process Description

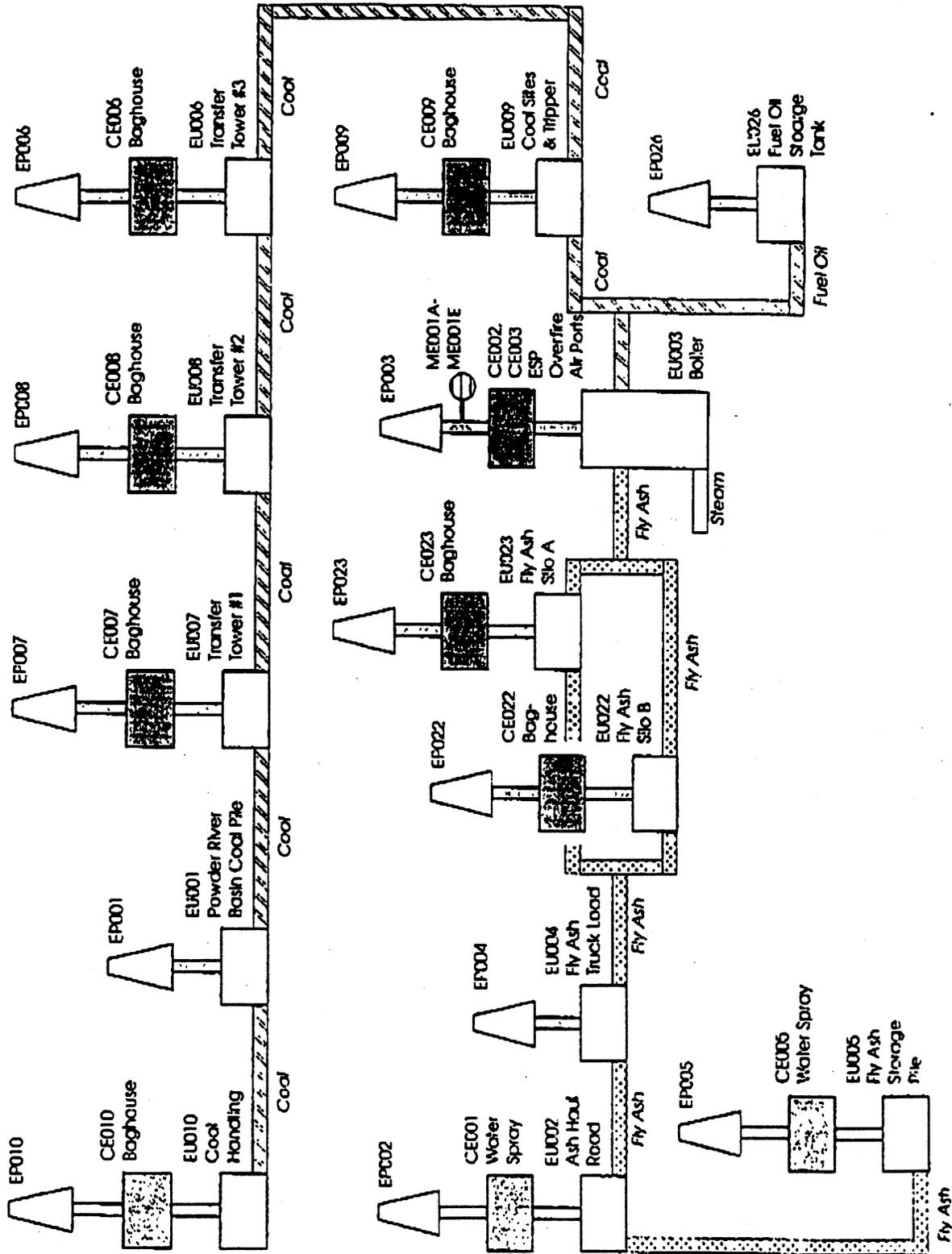
MidAmerican Energy's George Neal South Station, Unit 4, is a Foster-Wheeler balanced draft boiler firing low sulfur Powder River Basin pulverized coal.

2.2 Control Equipment Description

The emissions from MidAmerican Energy's George Neal South Station, Unit 4, are controlled by a UOP Cold Side Electrostatic Precipitator. The unit has an SO₃ flue gas-conditioning system to improve the ESP's collection efficiency. Figure 2-1 illustrates the process flow diagram of the facility.

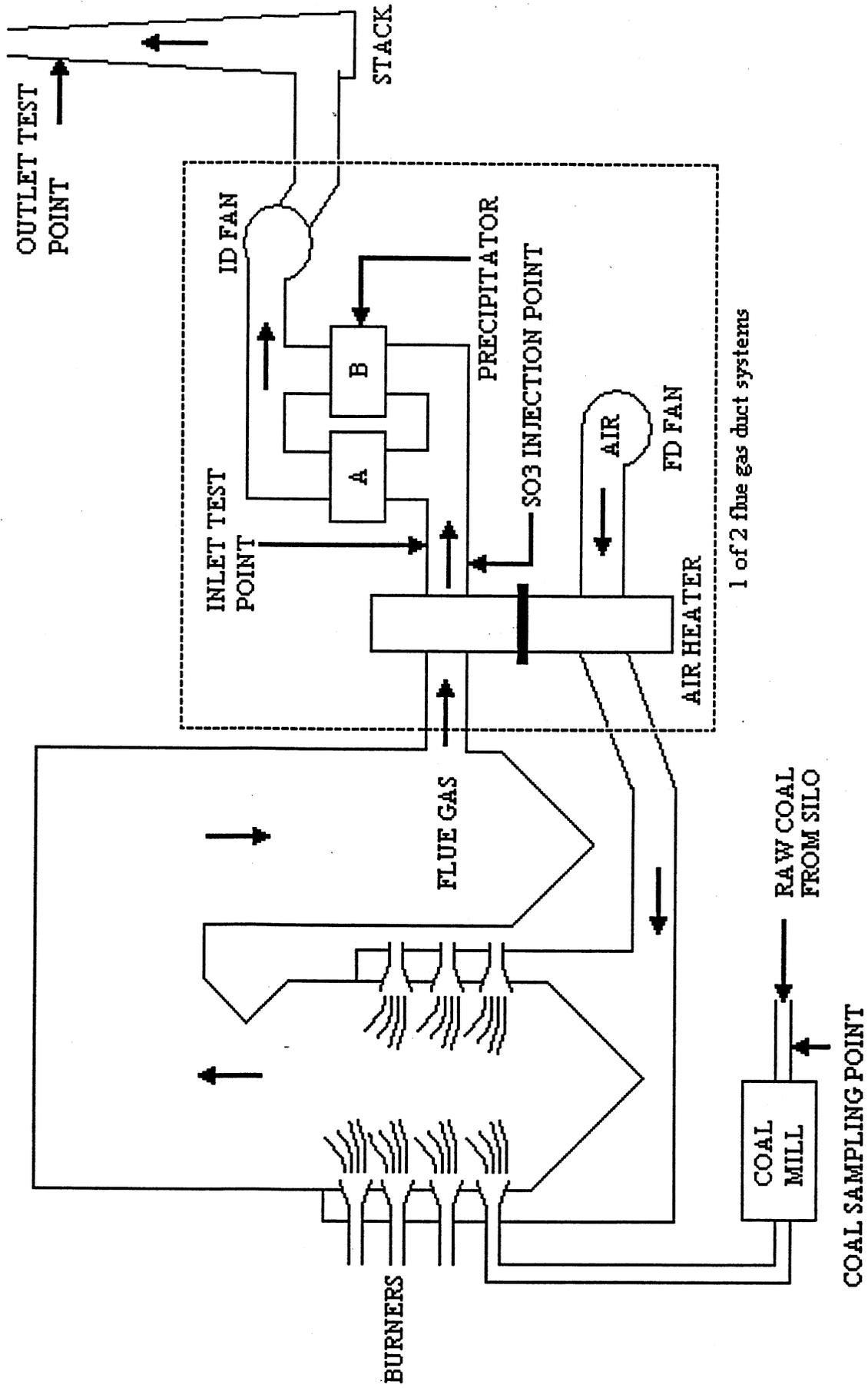
2.3 Flue Gas and Process Sampling Locations

The emissions testing was conducted and the precipitator inlet and precipitator outlet (stack) test locations. Diagrams of the testing locations showing dimensions and test points sampled can be found in Section 4.1.



PROCESS FLOW DIAGRAM
FIGURE 2-1

UNIT 4 BOILER FLOW DIAGRAM



3.0 SUMMARY OF DECISIONS AND RESULTS

3.1 Objectives and Test Matrix

The purpose of this program is to provide the EPA with background information on the speciated mercury emissions from MidAmerican Energy Company's George Neal South Station, Unit 4. Additionally, the test determines what effect, if any, the ESP has on mercury removal.

The specific objectives are as follows:

- 1) Simultaneously measure the mercury emissions at the inlet of the ESP and the outlet of the ESP or stack.
- 2) Collect and analyze coal samples during the test.
- 3) Obtain normal plant operating parameters during the test.
- 4) Determine the effect that the ESP has upon speciated mercury emissions.

The following table presents the sampling and analytical matrix.

TEST MATRIX

Sampling Location	No. of Runs	Sample Pollutant	Sample Method	Sample Time (min.)	Analytical Method	Analytical Laboratory
ESP Inlet	3	Speciated Mercury	O-H	120-180	O-H	Philips
ESP Inlet	3	O ₂ / CO ₂	BAG/3A	120-180	3A	B&McD
Stack	3	Speciated Mercury	O-H	120-180	O-H	Philips
Stack	3	O ₂ / CO ₂	BAG/3A	120-180	3A	B&McD
Coal Feeders	3	Note A	Composite	120-180	Note A	CT&E

Key:

- O-H** Refers to Ontario-Hydro Test Method
Philips Refers to Philips Analytical Services
B&McD Refers to Burns & McDonnell Engineering
CT&E Refers to Commercial Testing & Engineering
Note A Composite Coal Samples will be analyzed as follows:

<u>Constituent</u>	<u>ASTM Method</u>
Ash	D3174
Sulfur	D4239C
Mercury	D3684
Btu	D3286
Chlorine	D4208
Moisture	D3302

3.2 Field Test Changes and Problems

As for compiling portion of the mercury tests:

Prior to the commencement of the first test at the ESP inlet sampling location there was a failure with the sampling train involving a u-tube on the probe. The u-tube was replaced and the tests continued without any additional changes or problems.

As for the operating parameters of the mercury tests; the unit was set and operated at steady state conditions at full load (approx. 650 MW). No problems with the unit operations occurred other than the sulfur trioxide (SO₃) injection system tripped off twice during the first test run. The documentation reporting the times and injection rates of the SO₃ injection system can be found in Appendix C of this report.

3.3 Summary of Results

The following tables and charts presents the final results of the mercury testing performed by the Ontario-Hydro test method for MidAmerican Energy Company at their George Neal South Station – Unit 4.

MidAmerican Energy Company
 George Neal South Station - Unit 4
 Gas Flow Data DSCFH - 650 MW

Run No.	Sq Root Delta P in. H2O	Flue Temp Deg. F	Barometric Pressure in. Hg	Pitot Coefficient	Static Pressure in. H2O	Carbon Dioxide %	Oxygen %	Molecular Weight lb/lb mole	Velocity fps	Gas Volume	
										acfm	dscfh
Inlet											
Run 1	0.954	300.5	29.15	0.83	-18.9	13.50	5.50	28.78	66.05	1381414	46492461
Run 2	0.987	307.6	29.14	0.83	-19.2	13.05	6.20	28.73	68.75	1437958	47836084
Run 3	0.976	309.2	29.14	0.83	-19.1	12.46	6.80	28.71	68.07	1423702	47461856
Outlet											
Run 1	1.086	279.7	28.95	0.83	-0.9	12.40	6.80	30.26	70.90	2525435	92872092
Run 2	1.091	287.6	28.94	0.83	-0.9	12.33	7.00	28.90	73.28	2610333	94330960
Run 3	1.076	291.1	28.94	0.83	-0.9	11.10	8.60	28.71	72.68	2588900	93931712

Moisture Determination

Run No.	Delta H in. H2O	Meter Temp deg. F	Volume		Volume of Condensate ml	Volume Metered scf	Moisture %
			Metered cubic ft.	Meter Correction			
Inlet							
Run 1	0.87	76.8	60.111	0.997	181.5	57.535	12.94
Run 2	0.91	88.9	61.262	0.997	182.5	57.329	13.04
Run 3	0.85	74.3	59.333	0.997	176.1	57.033	12.70
Outlet							
Run 1	1.88	65.8	68.168	1.008	177.2	67.056	11.07
Run 2	1.87	74.2	68.303	1.008	185.0	66.108	11.65
Run 3	1.79	69.4	66.525	1.008	168.3	64.957	10.88

MidAmerican Energy Company
 George Neal South Station - Unit 4
 Particle-Bound Mercury Analysis - Summary of Results

Particle-Bound Mercury

Run No.	Front Half		Particle-Bound Mercury		* Removal Efficiency (percent)
	Analysis (ug)	Blank ** (ug)	Total (ug)	Concentration (ug/dscf) (lb/hr)	
Inlet					
Run 1	0.240	0.05	0.190	0.003302	0.000338
Run 2	0.094	0.05	0.044	0.000767	0.000081
Run 3	0.050	0.05	0.000	0.000000	0.000000
Average	0.128	0.05	0.078	0.001357	0.000140
Outlet					
Run 1	0.046	0.01	0.036	0.000537	0.000110
Run 2	0.089	0.01	0.079	0.001195	0.000249
Run 3	0.043	0.01	0.033	0.000508	0.000105
Average	0.059	0.01	0.049	0.000747	0.000155

** MDL's were used for blank values

* Efficiency =

1 -

$$\left[\frac{\text{Outlet lb/hr}}{2 * \text{Inlet lb/hr}} \right]$$

* 100

MidAmerican Energy Company
 George Neal South Station - Unit 4
 Oxidized Mercury Analysis - Summary of Results

Oxidized Mercury

Run No.	KCl Solution		Oxidized Mercury		* Removal Efficiency (percent)
	Analysis (ug)	Blank ** (ug)	Total (ug)	Concentration (ug/dscf) (lb/hr)	
Inlet					
Run 1	6.70	0.03	6.67	0.1159	n/a
Run 2	5.80	0.03	5.77	0.1006	n/a
Run 3	4.50	0.03	4.47	0.0784	n/a
Average	5.67	0.03	5.64	0.0983	n/a
Outlet					
Run 1	6.10	0.03	6.07	0.0905	22
Run 2	6.70	0.03	6.67	0.1009	1
Run 3	6.00	0.03	5.97	0.0919	0
Average	6.27	0.03	6.24	0.0944	8

** MDL's were used for blank values * Efficiency = $1 - \left[\frac{\text{Outlet lb/hr}}{2 * \text{Inlet lb/hr}} \right]$ * 100

MidAmerican Energy Company
 George Neal South Station - Unit 4
 Elemental Mercury Analysis - Summary of Results

Elemental Mercury

Run No.	HNO3-H2O2 Solution		H2SO4-KMnO4 Solution		Elemental Mercury		* Removal Efficiency (percent)
	Analysis (ug)	Blank ** (ug)	Analysis (ug)	Blank ** (ug)	Total (ug)	Concentration (ug/dscf)	
Inlet							
Run 1	0.25	0.25	8.90	0.04	8.86	0.1540	n/a
Run 2	0.25	0.25	11.00	0.04	10.96	0.1912	n/a
Run 3	0.25	0.25	4.80	0.04	4.76	0.0835	n/a
Average	0.25	0.25	8.23	0.04	8.19	0.1429	n/a
Outlet							
Run 1	0.25	0.25	8.20	0.04	8.16	0.1217	21
Run 2	0.25	0.25	10.00	0.04	9.96	0.1507	22
Run 3	0.25	0.25	8.10	0.04	8.06	0.1241	0
Average	0.25	0.25	8.77	0.04	8.73	0.1321	14

** MDL's were used for blank values

* Efficiency = 1 -

$$\left[\frac{\text{Outlet lb/hr}}{2 * \text{Inlet lb/hr}} \right]$$

* 100

MidAmerican Energy Company
 George Neal South Station - Unit 4
 Total Mercury Analysis - Summary of Results

Total Mercury

Run No.	Particle Bound		Oxidized (ug/dscm)	Oxidized (lb/hr)	Elemental		Total Mercury		* Removal Efficiency (percent)
	(ug/dscm)	(lb/hr)			(ug/dscm)	(lb/hr)	(ug/dscf)	(lb/hr)	
Inlet									
Run 1	0.00330	0.00034	0.11593	0.01188	0.15399	0.01578	0.2732	0.0280	n/a
Run 2	0.00077	0.00008	0.10065	0.01061	0.19118	0.02016	0.2926	0.0309	n/a
Run 3	0.00000	0.00000	0.07838	0.00820	0.08346	0.00873	0.1618	0.0169	n/a
Average	0.00136	0.00014	0.09832	0.01023	0.14288	0.01489	0.2426	0.0253	n/a
Outlet									
Run 1	0.00054	0.00011	0.09052	0.01853	0.12169	0.02492	0.2127	0.0436	22
Run 2	0.00120	0.00025	0.10090	0.02098	0.15066	0.03133	0.2528	0.0526	15
Run 3	0.00051	0.00011	0.09191	0.01903	0.12408	0.02570	0.2165	0.0448	0
Average	0.00075	0.00015	0.09444	0.01952	0.13214	0.02731	0.2273	0.0470	12

* Efficiency 1 - $\left[\frac{\text{Outlet lb/hr}}{2 * \text{Inlet lb/hr}} \right] * 100$

4.0 SAMPLING AND ANALYTICAL PROCEDURES

4.1 Flue Gas Sampling Locations

The original letter sent by the EPA requesting participation in this program asked that the test be performed at the inlet of the last emission control device and the outlet, or stack. George Neal South Station is configured in a manner such that it has two (2) separate inlet and duct runs to the ESP's.

During development of this test program, a web-site was developed to resolve frequently asked questions (FAQ). One of the questions that was posed, dealt with the matter of split ducts.

In answering this question, the EPA stated that only one (1) duct needed to be sampled, provided that process conditions are monitored. This interpretation was due to the fact that speciated mercury emissions are not expected to be stratified.

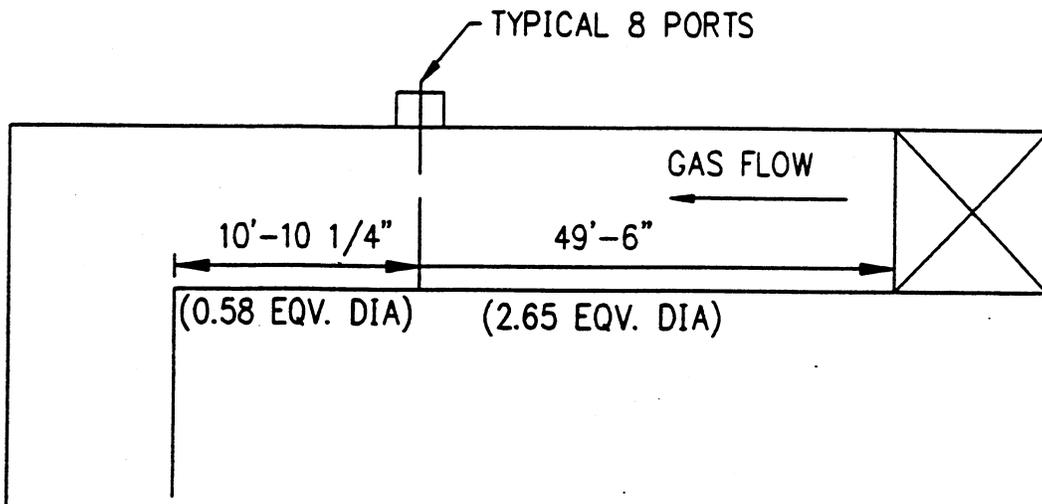
Consequently, this approach was used to conduct the inlet sampling. The only related process parameter that is monitored is the gas flow volumes from the CEM system in the stack. Gas flow volumes from the mercury test at the stack location were compared to the CEMS and found to be within 2.04%. Then the gas flow volumes from the mercury tests at the inlet location were doubled and then compared to the CEMS and found to be within an average of 4.32%. This concluded to us that the inlet ducts were relatively equal as far as gas flow volumes.

Figures 4-1 and 4-2 show the inlet flow schematics and traverse points. Figures 4-3 and 4-4 show the stack schematics and traverse points.

4.2 Coal Sampling Locations

Unit 4 has a total of seven coal mills. Each mill has one storage silo supplying two feeders per mill. Coal exits the silo and is fed into the mill by the two belt driven feeders. There is one coal sampling location per mill, located at one of the two feeders. During each test run, three coal samples weighing approximately two pounds each were extracted from each online mill. The samples were taken at 40-minute intervals, beginning at the start of the test run. A minimum of 18, two-pound samples were extracted per test run. At the conclusion of the test run, these samples were composited using a manual

divider and canvas, and split into two equally divided representative samples. One sample was retained for future reference and one was supplied to the test team for analysis.



DUCT 18'-8" x 18'-8"

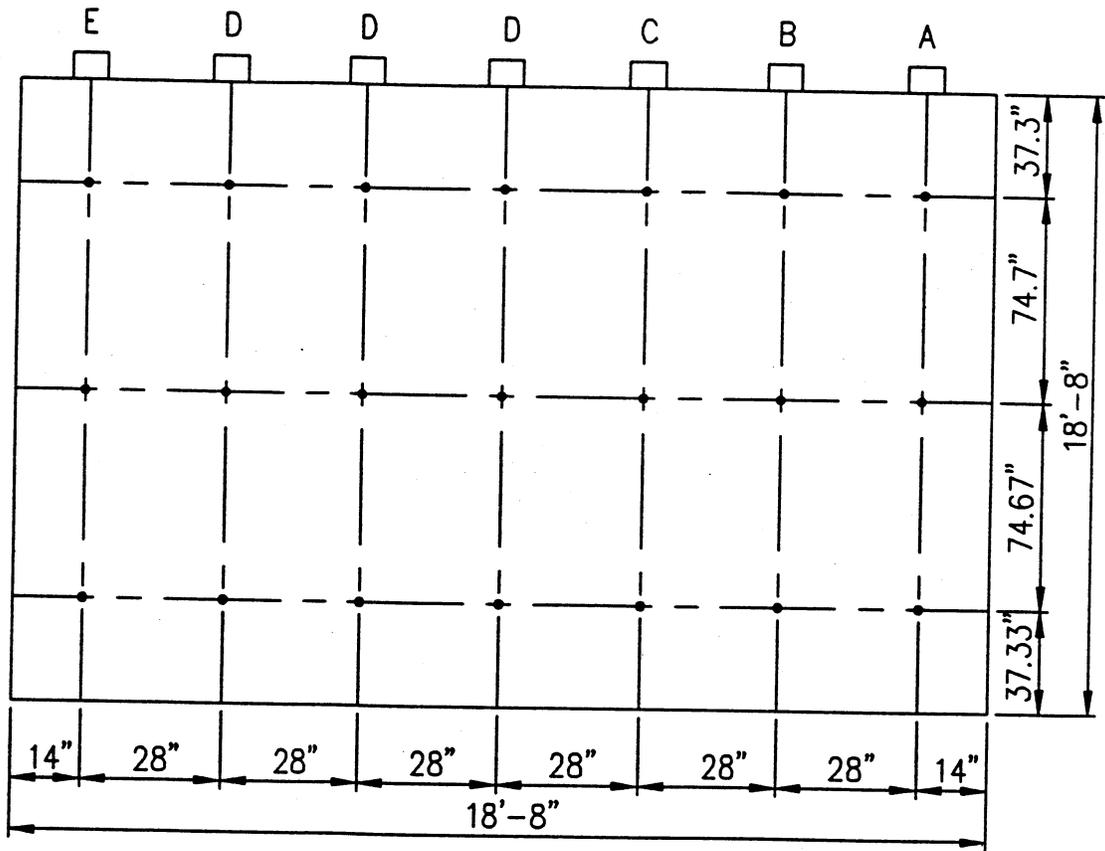
8 SAMPLE PORTS
 24 SAMPLE POINTS
 AREA = 348.6 ft²

NOT TO SCALE

Burns
 &
McDonnell

MIDAMERICAN ENERGY
 GEORGE NEAL SOUTH STATION
 UNIT #4
 INLET CROSS SECTION

Figure 4-1

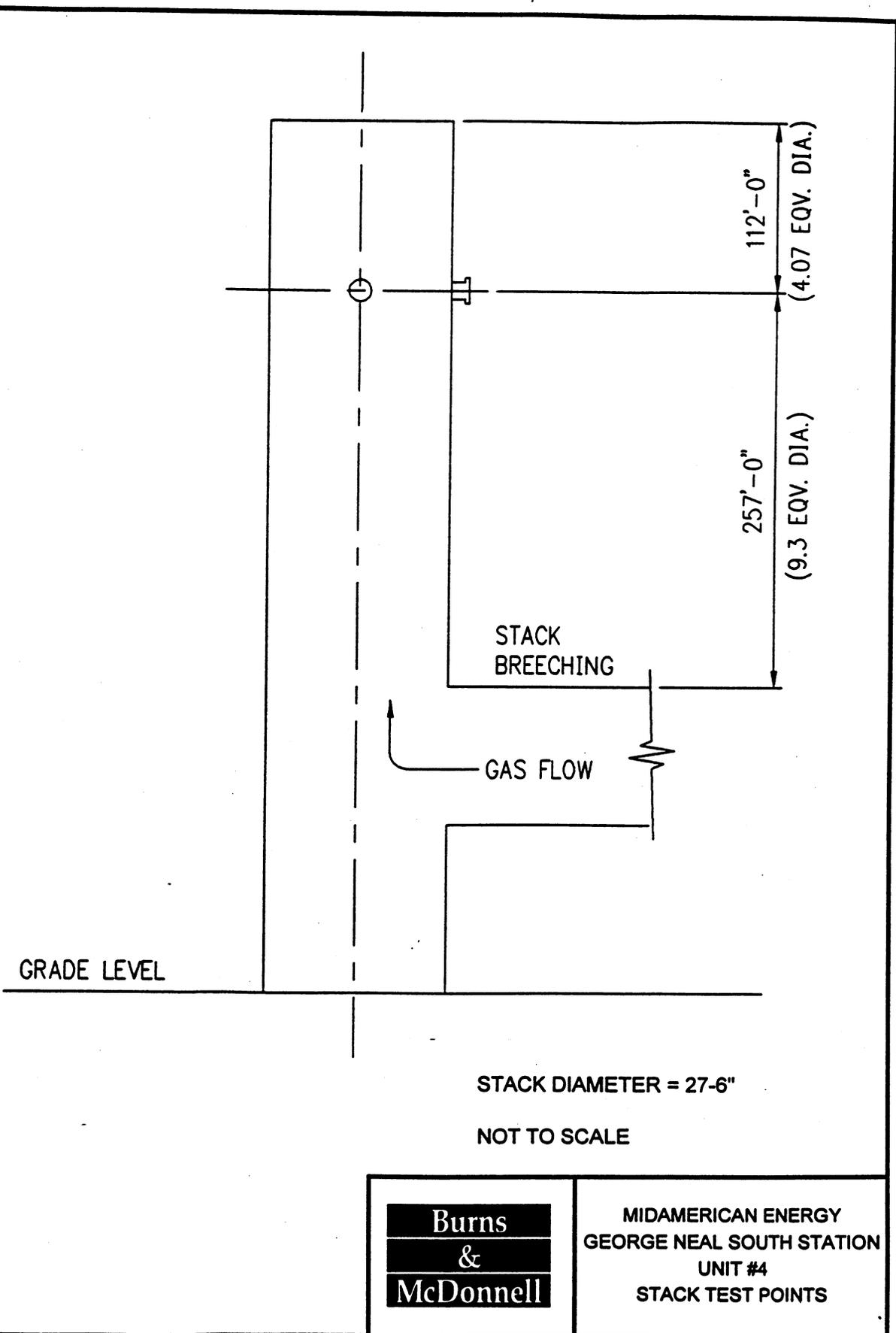


8 SAMPLE PORTS
 24 SAMPLE POINTS
 AREA = 348.6 ft²
 NOT TO SCALE

**Burns
 &
 McDonnell**

MIDAMERICAN ENERGY
 GEORGE NEAL SOUTH STATION
 UNIT #4
 INLET TEST POINTS

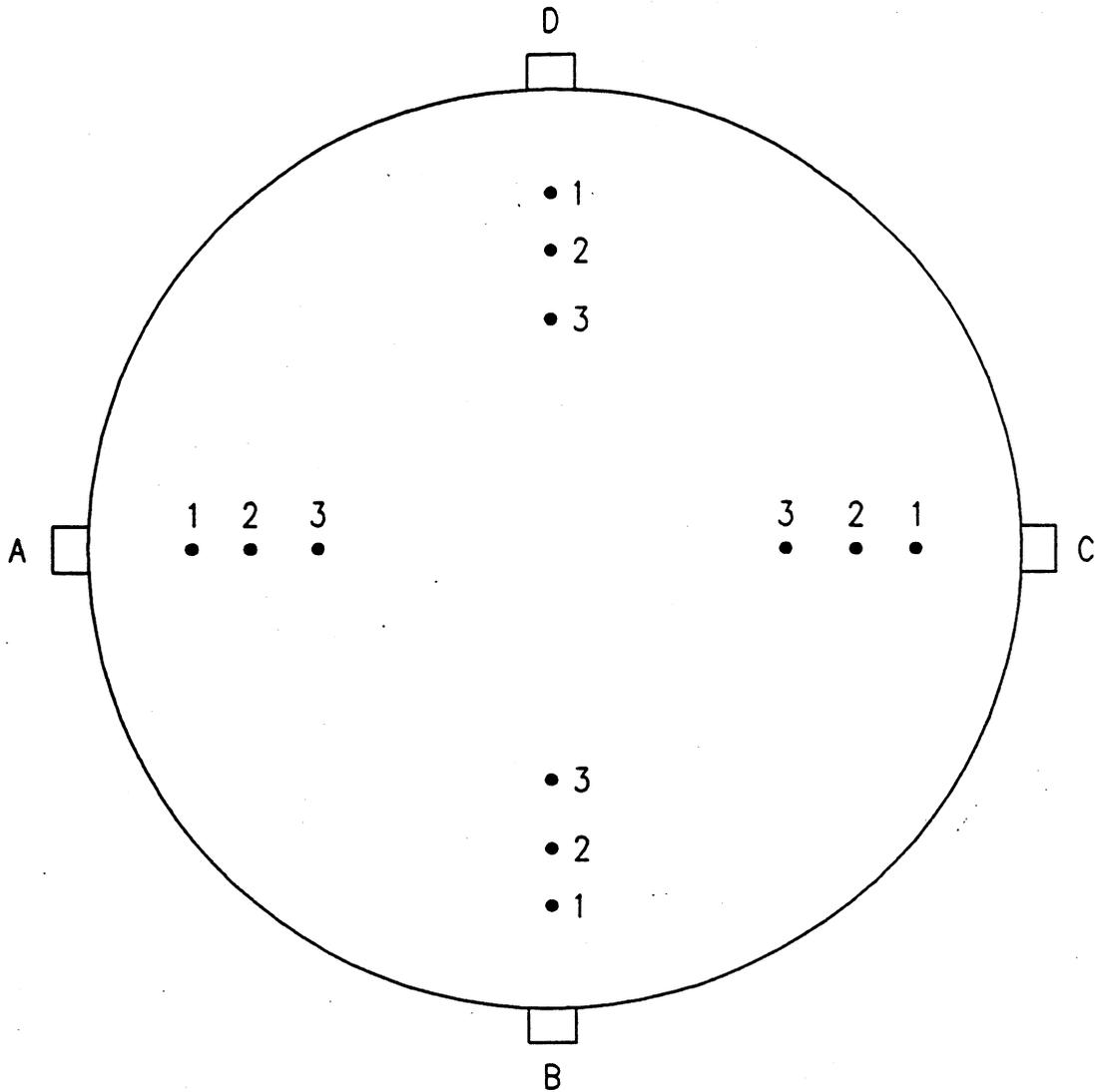
Figure 4-2



**Burns
&
McDonnell**

MIDAMERICAN ENERGY
GEORGE NEAL SOUTH STATION
UNIT #4
STACK TEST POINTS

Figure 4-3



<u>POINTS</u>	<u>DISTANCE FROM INSIDE WALL</u>	<u>DIAMETER STACK = 27'-6"</u> <u>AREA OF FLUE = 593.7 FT</u>
1	1.21 FT	
2	4.02 FT	
3	8.14 FT	

**Burns
&
McDonnell**

MIDAMERICAN ENERGY
GEORGE NEAL SOUTH STATION
UNIT #4
STACK TEST POINTS

Figure 4-4

4.3 Emissions Test Methods

The test method used for the mercury emissions testing was the Ontario-Hydro Test Method. The following is the latest draft form that was available at the time of the test. Procedures for U.S. EPA Reference Method 3A are also included at the end of this section.

April 8, 1999
DRAFT

Standard Test Method for Elemental, Oxidized, Particle-Bound, and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method)¹

1. Scope

1.1 This method applies to the determination of elemental, oxidized, particle-bound, and total mercury emissions from coal-fired stationary sources.

1.2 This method is applicable to elemental, oxidized, particle-bound, and total mercury concentrations ranging from approximately 0.5 to 100 $\mu\text{g/dscm}$.

1.3 This method describes equipment and procedures for obtaining samples from effluent ducts and stacks, equipment and procedures for laboratory analysis, and procedures for calculating results.

1.4 This method is applicable for sampling elemental, oxidized, and particle-bound mercury at the inlet and outlet of emission control devices and for calculating control device mercury collection efficiency.

1.5 Method applicability is limited to flue gas stream temperatures within the thermal stability range of the sampling probe and filter components.

1.6 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.8 This standard assumes users are familiar with EPA stack-gas sampling procedures as stated in EPA Methods 1-4, Method 5, and Method 17.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

¹ This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

² *Annual Book of ASTM Standards*, Vol. 11.01.

- D1356 Definitions of Terms Relating to Atmospheric Sampling and Analysis³
D 2986 Evaluation of Air-Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test³
D 3154 Test Method for Average Velocity in a Duct (Pitot Tube Method)³
D 3685 Particulates Independently or for Particulates and Collected Residue Simultaneously in Stack Gases³
E 1 Specification for ASTM Thermometers⁴

2.2 Other Standards:⁵

- EPA Method 1 – Sample and Velocity Traverses for Stationary Sources
EPA Method 2 – Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)
EPA Method 3 – Gas Analysis for the Determination of Dry Molecular Weight
EPA Method 4 – Determination of Moisture Content in Stack Gases
EPA Method 5 – Determination of Particulate Emissions from Stationary Sources
EPA Method 12 – Determination of Inorganic Lead Emissions from Stationary Sources
EPA Method 17– Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method)
EPA Method 29 – Determination of Metals Emissions from Stationary Sources
EPA Method 101A – Determination of Particle-Bound and Gaseous Mercury Emissions from Sewage Sludge Incinerators
EPA Method 301 – Field Validation of Pollutant Measurement Methods from Various Waste Media
EPA SW 846 7470 – Mercury in Liquid Waste – Manual Cold Vapor Technique
EPA Water and Waste 600/4-79-020 – Methods for Chemical Analysis of Water and Wastes

3. Terminology

3.1 Definitions other than those given below in Sections 3.2, 3.3, and 3.4 are listed in ASTM D 1356.

3.2 Definitions of Terms specific to the standard:

3.2.1 *elemental mercury*—mercury in its zero oxidation state, Hg⁰.

³ *Annual Book of ASTM Standards*, Vol. 11.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Available from the U.S. Environmental Protection Agency's Emission Measurement Technical Information Center or Code of Federal Regulations (40 CFR Part 60, Appendix A or 40 CFR Part 61, Appendix B).

3.2.2 *oxidized mercury*—mercury in its mercurous or mercuric oxidation states: Hg_2^{2+} and Hg^{2+} , respectively.

DRAFT

3.2.3 *elemental mercury catch*—mercury collected in the acidified hydrogen peroxide ($\text{HNO}_3\text{-H}_2\text{O}_2$) and potassium permanganate ($\text{H}_2\text{SO}_4\text{-KMnO}_4$) impinger solutions employed in this method. This is gaseous Hg^0 .

3.2.4 *oxidized mercury catch*—mercury collected in the aqueous potassium chloride (KCl) impinger solution employed in this method. This is gaseous Hg^{2+} .

3.2.5 *particle-bound mercury catch*—mercury associated with the particulate matter collected in the front half of the sampling train.

3.2.6 *front half of the sampling train*—all mercury collected on and upstream of the sample filter.

3.2.7 *total mercury*—all mercury (solid-bound, liquid, or gaseous) however generated or entrained in the flue gas stream (i.e., summation of elemental, oxidized, and particle-bound mercury).

3.3 Symbols:

- A = cross-sectional area of stack, m^2 (ft^2)
- B_{ws} = water vapor in the gas stream, proportion by volume
- ΔH = average pressure differential across the orifice meter, kPa (in. H_2O)
- Hg_{ash} = concentration of mercury in sample filter ash, $\mu\text{g/g}$
- Hg^{p} = concentration of particle-bound mercury, $\mu\text{g/dscm}$
- Hg^0 = concentration of elemental mercury, $\mu\text{g/dscm}$
- Hg^{2+} = concentration of oxidized mercury, $\mu\text{g/dscm}$
- IR = instrument reading from mercury analyzer, $\mu\text{g/L}$
- L_p = leakage rate observed during the posttest leak check, m^3/min (cfm)
- L_a = maximum-acceptable leakage rate
- M_s = molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole)
- M_w = molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)
- N = Normal conditions, defined as 0°C and 1 atmosphere pressure (in the U.S. N and standard conditions are the same in SI units)
- P_{bar} = barometric pressure at the sampling site, kPa (in. Hg)
- P_s = absolute stack gas pressure, kPa (in. Hg)
- P_{std} = standard absolute pressure, 101.3 kPa (29.92 in. Hg)
- R = ideal gas constant, $0.008314 \text{ kPa}\cdot\text{m}^3/\text{K}\cdot\text{g-mole}$ (21.85 in. $\text{Hg}\cdot\text{ft}^3/^\circ\text{R}\cdot\text{lb-mole}$)
- T_m = absolute average dry gas meter temperature, K ($^\circ\text{R}$)
- T_s = absolute stack temperature, K ($^\circ\text{R}$)
- T_{std} = standard absolute temperature, 293 K (528 $^\circ\text{R}$)
- V_D = total digested volume, mL
- V_m = volume of gas sample as measured by dry gas meter, dcm (dscf)

- $V_{m(std)}$ = volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf)
- $V_{w(std)}$ = volume of water vapor in the gas sample, corrected to standard conditions, scm (scf)
- W_{ash} = total mass of ash on sample filter, g
- W_{lc} = total weight of liquid collected in impingers and silica gel, g (lb)
- Y = dry gas meter calibration factor
- θ = total sampling time, min
- θ_1 = sampling time interval, from the beginning of a run until the first component change, min

corrected to standard
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4. Summary of Test Method

4.1 A sample is withdrawn from the flue gas stream isokinetically through a probe/filter system, maintained at 120°C or the flue gas temperature, whichever is greater, followed by a series of impingers in an ice bath. Particle-bound mercury is collected in the front half of the sampling train. Oxidized mercury is collected in impingers containing a chilled aqueous potassium chloride solution. Elemental mercury is collected in subsequent impingers (one impinger containing a chilled aqueous acidic solution of hydrogen peroxide and three impingers containing chilled aqueous acidic solutions of potassium permanganate). Samples are recovered, digested, and then analyzed for mercury using cold-vapor atomic absorption (CVAAS) or fluorescence spectroscopy (CVAAS).

5. Significance and Use

5.1 The measurement of particle-bound, oxidized, elemental, and total mercury in stationary-source flue gases provides data that can be used for dispersion modeling, deposition evaluation, human health and environmental impact assessments, emission reporting, compliance determinations, etc. Particle-bound, oxidized, and elemental mercury measurements before and after control devices may be necessary for optimizing and evaluating the mercury removal efficiency of emission control technologies.

6. Interferences

There are no known interferences, but certain biases may be encountered (See Section 16).

7. Apparatus

7.1 *Sampling Train*—similar to ASTM D 3685, EPA Method 5/EPA Method 17 and EPA Method 29 trains, as illustrated in Fig. 1.

7.1.1 *Probe Nozzle (Probe Tip)*—Glass nozzles are required unless alternate nozzles are constructed of materials that are free from contamination and will not interact with the sample. Probe fittings constructed of polytetrafluoroethylene (PTFE), polypropylene, etc., are required instead of metal fittings to prevent contamination.

7.1.2. *Probe Liner*—If the sample train is to be in EPA Method 5 configuration (out-of-stack filtration), the probe liner must be constructed of quartz or borosilicate glass. If an EPA Method 17 (in-stack filtration) sampling configuration is used, the probe liner may be constructed of borosilicate glass, quartz or, depending on the flue gas temperature, PTFE.

7.1.3 *Pitot Tube*—Type S pitot tube. Refer to Section 2.2 of EPA Method 2 for a description.

7.1.4 *Differential Pressure Gauges*—inclined manometers or equivalent devices. Refer to Section 2.1 of EPA Method 2 for a description.

7.1.5 *Filter Holder* — constructed of borosilicate glass or PTFE-coated stainless steel with a PTFE filter support or other nonmetallic, noncontaminating support. Do not use a glass frit or stainless steel wire screen. A silicone rubber or PTFE gasket, designed to provide a positive seal against leakage from outside or around the filter, may be used.

7.1.6 *Connecting Umbilical Tube*—heated PTFE tubing. This tube must be heated to a minimum of 120°C to help prevent water and acid condensation. (The umbilical tube is defined as any tubing longer than 0.5 m that connects the filter holder to the impinger train).

7.1.7 *Probe and Filter Heating System*

7.1.7.1 *EPA Method 5 Configuration*—For EPA Method 5 configuration, the temperature of the flue gas, sample probe, and the exit of the sample filter must be monitored using temperature sensors capable of measuring temperature to within 3°C (5.4°F). The heating system must be capable of maintaining the sample gas temperature of the probe and exit of the sample filter to within $\pm 15^\circ\text{C}$ ($\pm 27^\circ\text{F}$) of the flue gas temperature. Regardless of the flue gas temperature, to prevent water and acid condensation, at no time must the probe temperature, sample filter exit gas temperature, or the temperature of the connecting umbilical cord be less than 120°C.

7.1.7.2 *EPA Method 17 Configuration*—For EPA Method 17 configuration, the sample filter is located in the duct and, therefore, naturally maintained at the flue gas temperature. The heating system is only required to maintain the probe and connecting umbilical cord to at least 120°C. If the flue gas temperature is less than 120°C, then EPA Method 5 configuration must be used.

7.1.8 *Condensing/Absorbing System*—consists of eight impingers immersed in an ice bath and connected in series with leak-free ground glass fittings or other noncontaminating leak-free fittings. (At no time is silicon grease or other greases to be used for this method). The first, second, fourth, fifth, sixth, and eighth impingers are of the Greenburg–Smith design modified by replacing the standard tip with a 1.3-cm (0.5-in.)-ID straight glass tube extending to about 1.3 cm (0.5 in.) from the bottom of the flask. The third and seventh impingers are also Greenburg–Smith design, but with the standard tip including the glass impinging plate. The first, second, and third impingers contain aqueous 1 N potassium chloride (KCl) solution. The fourth impinger contains

an aqueous solution of 5% v/v nitric acid (HNO_3) and 10% v/v hydrogen peroxide (H_2O_2). The fifth, sixth, and seventh impingers contain an aqueous solution of 4% w/v potassium permanganate ($KMnO_4$) and 10% v/v sulfuric acid (H_2SO_4). The last impinger contains silica gel or an equivalent desiccant. Refer to Note 1.

Note 1—When flue gas streams are sampled with high moisture content ($>20\%$), additional steps must be taken to eliminate carryover of impinger contents from one sample type to the next. These steps must include use of oversized impinger(s) or use of an empty impinger between the KCl and $HNO_3-H_2O_2$. If a dry impinger is used, it must be rinsed as discussed in Section 13.2 of this method and the rinse added to the preceding impinger.

7.1.9 Metering System—vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within $3^\circ C$ ($5.4^\circ F$), and a dry gas meter or controlled orifice capable of measuring volume to within 2%.

7.1.10 Barometer—barometer capable of measuring atmospheric pressure to within 0.33 kPa (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case, the station value (which is the absolute barometric pressure) shall be requested. An adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of negative 0.33 kPa (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

7.1.11 Gas Density Determination Equipment—temperature sensor and pressure gauge, as described in Section 2.3 and 2.4 of EPA Method 2. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the sensor tip extends beyond the leading edge of the probe sheath and does not touch any metal. Alternative temperature sensor configurations are described in Section 2.1.10 of EPA Method 5. If necessary, a gas analyzer can be used to determine dry molecule weight of the gas (refer to EPA Method 3).

7.2 Digestion Apparatus

7.2.1 Dry Block Heater or Hot Water Bath—a heater capable of maintaining a temperature of $95^\circ C$ is required for digestion of samples, similar to that described in EPA SW846 Method 7470.

7.2.2 Ice Bath

7.2.3 Digestion Flasks—Use 50- to 70-mL tubes or flasks with screw caps that will fit a dry block heater. For a water bath, 300-mL biological oxygen demand bottles for SW846 Method 7470 are to be used. In addition, borosilicate glass test tubes, 35- to 50-mL volume, with rack are needed.

7.2.4 Microwave or Convection Oven and PTFE Digestion Vessels—120 mL, or equivalent digestion vessels with caps equipped with pressure relief valves for the dissolution of

ash, along with a capping station or the equivalent to seal the digestion vessel caps. Use a vented microwave or convection oven for heating. In addition, polymethylpentene (PMP) or equivalent volumetric flasks are recommended for the digested ash solutions.

Use a vented
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7.3 Analytical Equipment—dedicated mercury analyzer or equivalent apparatus for the analysis of mercury via CVAAS. Alternatively, CVAFS may be used. 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.

8. Reagents and Materials

8.1 Purity of Reagents—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II in ASTM Specification D 1193.

8.3 Reagents:

8.3.1 Boric Acid (H_3BO_3)—purified reagent grade.

8.3.2 Hydrochloric Acid (HCl)—trace metal-grade concentrated hydrochloric acid, with a specific gravity of 1.18.

8.3.3 Hydrofluoric Acid (HF)—concentrated hydrofluoric acid, 48%–50%.

8.3.4 Hydrogen Peroxide (H_2O_2)—30%^{v/v} hydrogen peroxide.

8.3.5 Hydroxylamine Sulfate ($NH_2OH \cdot H_2SO_4$)—solid.

8.3.6 Mercury Standard Solution—a certified (1000 $\mu\text{g/mL}$) mercury standard.

8.3.7 Nitric Acid (HNO_3)—trace metal-grade concentrated nitric acid with a specific gravity of 1.42.

⁶ “Reagent Chemicals, American Chemical Society Specifications,” Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see “Reagent Chemicals and Standards,” by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the “United States Pharmacopeia.”

8.3.8 *Potassium Chloride (KCl)*—solid.

8.3.9 *Potassium Permanganate (KMnO₄)*—solid.

8.3.10 *Potassium Persulfate (K₂S₂O₈)*—solid.

8.3.11 *Stannous Chloride (SnCl₂ · 2H₂O)*—solid.

8.3.12 *Sulfuric Acid (H₂SO₄)*—trace metal-grade concentrated sulfuric acid, with a specific gravity of 1.84.

8.4 *Materials:*

8.4.1 *Indicating Silica Gel*—with a size of 6–16 mesh.

8.4.2 *Crushed or Cubed Ice.*

8.4.3 *Sample Filters*—quartz fiber filters, without organic binders, exhibiting at least 99.95% efficiency (<0.05% penetration) for 0.3- μ m dioctyl phthalate smoke particles and containing less than 0.2 μ g/m² of mercury. Test data provided by filter manufacturers and suppliers stating filter efficiency and mercury content are acceptable. However, if no such results are available, determine filter efficiency using ASTM Test Method D 2986, and analyze filter blanks for mercury prior to emission testing. Filter material must be unreactive to sulfur dioxide (SO₂) or sulfur trioxide (SO₃).⁷

8.4.4 *Filter Papers*—for filtration of digested samples. The filter paper must have a particle retention of >20 μ m and filtration speed of >12 sec.

8.4.5 *Nitrogen Gas (N₂)*—carrier gas of at least 99.998% purity. Alternatively, argon gas may be used.

8.4.6 *Soda Lime*—indicating 4- to 8-mesh absorbent for trapping carbon dioxide.

8.4.7 *Sample Containers*—glass with PTFE-lined lids.

8.5 *Sampling Reagents*

8.5.1 *KCl Absorbing Solution (1 mol/L)*—Dissolve 74.56 g of KCl in 500 mL of reagent water in a 1000-mL volumetric flask, swirl to mix, and dilute to volume with water. Mix well. A new batch of solution must be made prior to each field test.

⁷ Felix, L.G.; Clinard, G.I.; Lacey, G.E.; McCain, J.D. "Inertial Cascade Impactor Substrate Media for Flue Gas Sampling," U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, Publication No. EPA-600/7-77-060; June 1977, 83 p.

8.5.2 *HNO₃-H₂O₂ Absorbing Solution (5%^{v/v} HNO₃, 10%^{v/v} H₂O₂)*—Add slowly, with stirring, 50 mL of concentrated HNO₃ to a 1000-mL volumetric flask containing approximately 500 mL of water, and then add carefully, with stirring, 333 mL of 30%^{v/v} H₂O₂. Dilute to volume with water. Mix well. A new batch of solution must be made prior to each field test.

8.5.3 *H₂SO₄-KMnO₄ Absorbing Solution (4%^{w/v} KMnO₄, 10%^{v/v} H₂SO₄)*—Mix carefully, with stirring, 100 mL of concentrated H₂SO₄ into approximately 800 mL of water. When mixing, be sure to follow standard acid to water addition procedures and safety precautions associated with strong acids. Then add water, with stirring, to make 1 L. This solution is 10%^{v/v} H₂SO₄. Dissolve, with stirring, 40 g of KMnO₄ into 10%^{v/v} H₂SO₄, and add 10%^{v/v} H₂SO₄, with stirring, to make 1 L. (Warning—See 9.1.1). H₂SO₄-KMnO₄ absorbing Solution must be made daily.

8.6 *Rinse Solutions for Sample Train*

8.6.1 *0.1 N HNO₃ Solution*—A certified reagent grade 0.1 N HNO₃ solution can be purchased directly or can be made by slowly adding 12.5 mL of concentrated HNO₃ to a 2000-mL volumetric flask containing approximately 500 mL of water, then diluting with water to volume.

8.6.2 *10%^{w/v} HNO₃ Solution*—Mix carefully, with stirring, 100 mL of concentrated HNO₃ into approximately 800 mL of water. When mixing, be sure to follow standard acid to water addition procedures and safety precautions associated with strong acids. Then add water, with stirring, to make 1 L.

8.6.3 *10%^{w/v} Hydroxylamine Sulfate*—Add 100 g hydroxylamine sulfate to a 1000-mL volumetric flask containing approximately 500 mL of water. After the hydroxylamine sulfate has been dissolved, dilute with water to volume.

8.7 *Sample Digestion Reagents:*

8.7.1 *Boric Acid Solution (4%^{w/v})*—Dissolve 4 g H₃BO₃ in water, and dilute to 100 mL.

8.7.2 *Aqua Regia (HCl:HNO₃ 3:1)*—Add 3 parts concentrated HCl to 1 part concentrated HNO₃. Note that this should be made up in advance and allowed to form a dark orange color. This mixture should be loosely capped, as pressure will build as gases form.

8.7.3 *Saturated Potassium Permanganate Solution (5%^{w/v})*—Mix 5 g KMnO₄ into water, dilute to 100 mL, and stir vigorously.

8.7.4 *Potassium Persulfate Solution (5%^{w/v})*—Dissolve 5 g K₂S₂O₈ in water, and dilute to 100 mL.

8.8 *Analytical Reagents:*

8.8.1 *Hydrochloric Acid Solution (10%^{v/v})*—Add 100 mL concentrated HCl to water, and dilute to 1 L. Be sure to follow all safety precautions for using strong acids. **DRAFT**

8.8.2 *Stannous Chloride Solution (10%^{w/v})*—Dissolve 100 g in 10%^{v/v} HCl, and dilute with 10%^{v/v} HCl to 1 L. Difficulty in dissolving the stannous chloride can be overcome by dissolving in a more concentrated HCl solution (such as 100 mL of 50%^{v/v} HCl) and diluting to 1 L with water. Note that care must be taken when adding water to a strong acid solution. Add a lump of mossy tin (~0.5 g) to this solution.

8.9 *Mercury Standards:*

8.9.1 *10 mg/L Hg Stock Solution*—Dilute 1 mL of 1000 mg/L Hg standard solution to 100 mL with 10%^{v/v} HCl.

8.9.2 *100 µg/L Hg Stock Solution*—Dilute 1 mL of 10 mg/L Hg stock solution to 100 mL with 10%^{v/v} HCl.

8.9.3 *Working Hg Standards*—Prepare working standards of 1.0, 5.0, 10.0, and 20.0 µg/L Hg from the 100-µg/L stock solution by diluting 1, 5, 10, and 20 mL each to 100 mL with 10%^{v/v} HCl.

Note 1—If samples to be analyzed are less than 1.0 µg/L Hg, working standards should be prepared at 0.05, 0.1, 0.5, and 1.0 µg/L Hg from a 10-µg/L Hg standard solution.

8.9.4 *Quality Control Standard (QC)*—A quality control standard is prepared from a separate Hg standard solution. The QC standard should be prepared at a concentration of approximately one-half the calibration range.

8.10 *Glassware Cleaning Reagents*—Prior to any fieldwork, all glassware should be cleaned according to the guidelines outlined in EPA Water and Waste 600/4-79-020, Section 4, pages 4-5.

9. Hazards

9.1 *Warning:*

9.1.1 Pressure may build up in the solution storage bottle because of a potential reaction between potassium permanganate and acid. Therefore, these bottles should not be fully filled and should be vented to relieve excess pressure and prevent explosion. Venting must be in a manner that will not allow contamination of the solution.

9.1.2 Hazards to personnel exist in the operation of the cold-vapor atomic absorption spectrophotometer. Refer to the manufacturer's instruction manual before operating the instrument.

9.1.3 Sample digestion with hot concentrated acids creates a safety problem. Observe appropriate laboratory procedures for working with concentrated acids. **DRAFT**

9.2 *Precaution:*

9.2.1 The determination of microquantities of mercury species requires meticulous attention to detail. Good precision is generally unattainable without a high level of experience with stack-sampling procedures. Precision may be improved by knowledge of, and close adherence to, the suggestions that follow.

9.2.1.1 All glassware used in the method must be cleaned thoroughly prior to use in the field, as described in Section 8.10 of this method.

9.2.1.2 Use the same reagents and solutions in the same quantities for a group of determinations and the corresponding solution blank. When a new reagent is prepared or a new stock of filters is used, a new blank must be taken and analyzed.

10. **Sampling**

10.1 *Preparation for Test:*

10.1.1 *Preliminary Stack Measurements*—Select the sampling site, and determine the number of sampling points, stack pressure, temperature, moisture, dry molecular weight, and range of velocity head in accordance with procedures of ASTM Test Method D 3154 or EPA Methods 1 through 4.

10.1.2 Select the correct nozzle diameter to maintain isokinetic sampling rates based on the range of velocity heads determined in 10.1.1.

10.1.3 Ensure that the proper differential pressure gauge is selected for the range of velocity heads (refer to EPA Method 2, Section 2.2).

10.1.4 It is suggested that an EPA Method 17 configuration be used; however, if an EPA Method 5 setup is to be used, then select a suitable probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack to minimize probe length when a large duct or stack is sampled.

10.1.5 *Sampling Time and Volume*—The total sampling time for this method should be at least 2 but not more than 3 hours. Use a nozzle size that will guarantee an isokinetic gas sample volume between 1.0 dry cubic meters corrected to standard conditions (dscm) and 2.5 dscm. If traverse sampling is done (necessary for sampling at electric utilities), use the same points for sampling that were used for the velocity traverse as stated in Section 10.1.1 of this method. Each traverse point must be sampled for a minimum of 5 minutes.

11. **Preparation of Apparatus**

11.1 *Pretest Preparation:*

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11.1.1 Weigh several 200- to 300-g portions of silica gel in airtight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container on each container. Alternatively, the silica gel can be weighed directly in the impinger immediately prior to the train being assembled.

11.1.2 Desiccate the sample filters at $20^{\circ} \pm 5.6^{\circ}\text{C}$ ($68^{\circ} \pm 10^{\circ}\text{F}$) and ambient pressure for 24 to 36 hours, weigh at intervals of at least 6 hours to a constant weight (i.e., $<0.5\text{-mg}$ change from previous weighing), and record results to the nearest 0.1 mg. Alternatively, the filters may be oven-dried at 105°C (220°F) for 2 to 3 hours, desiccated for 2 hours, and weighed.

11.1.3 Clean all sampling train glassware as described in Section 8.10 before each series of tests at a single source. Until the sampling train is assembled for sampling, cover all glassware openings where contamination can occur.

11.2 *Preparation of Sampling Train:*

11.2.1 Assemble the sampling train as shown in Figure 1.

11.2.2 Place 100 mL of the KCl solution (see Section 8.5.1 of this method) in each of the first, second, and third impingers, as indicated in Figure 1.

11.2.3 Place 100 mL of the $\text{HNO}_3\text{-H}_2\text{O}_2$ solution (Section 8.5.2 of this method) in the fourth impinger, as indicated in Figure 1.

11.2.4 Place 100 mL of the $\text{H}_2\text{SO}_4\text{-KMnO}_4$ absorbing solution (see Section 8.5.3 of this method) in each of the fifth, sixth, and seventh impingers, as indicated in Figure 1.

11.2.5 Transfer approximately 200 to 300 g of silica gel from its container to the last impinger, as indicated in Figure 1.

11.2.6 Prior to final train assembly, weigh and record the weight of each impinger. This information is required to calculate the moisture content of the sampled flue gas.

11.2.7 To ensure leak-free sampling train connections and to prevent possible sample contamination problems, use PTFE tape, PTFE-coated O-rings, or other noncontaminating material.

11.2.8 Place a weighed filter in the filter holder using a tweezer or clean disposable surgical gloves.

11.2.9 Install the selected nozzle using a noncontaminating rubber-type O-ring or equivalent when stack temperatures are less than 260°C (500°F) and an alternative gasket

) material when temperatures are higher. Other connecting systems, such as PTFE ferrules or ground glass joints, may also be used on the probe and nozzle.

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11.2.10 Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

11.2.11 Place crushed or cubed ice around the impingers.

11.2.12 *Leak-Check Procedures.* Follow the leak-check procedures given in Section 4.1.4.1 (Pretest Leak Check), Section 4.1.4.2 (Leak Checks During the Sample Run), and Section 4.1.4.3 (Posttest Leak Checks) of EPA Method 5 or 17.

Note 2—If the flue gas temperature at the sampling location is greater than 260°C (above the temperature where PTFE or rubber-type seals can be used), the posttest leak check is determined beginning at the front end of the probe (does not include nozzle or sample filter holder for EPA Method 17).

12. Calibration and Standardization

12.1 *Sampling Train Calibration:*

) 12.1.1 *Probe Nozzle*—Refer to Sections 2.1.1 of either EPA Method 5 or 17.

12.1.2 *Pitot Tube*—Refer to Section 4 of EPA Method 2.

12.1.3 *Metering System*—Refer to Section 5.3 of either EPA Method 5 or 17.

12.1.4 *Probe Heater*—Refer to Section 7.1.7.1 and 7.1.7.2 of this method.

12.1.5 *Temperature Gauges*—Refer to Section 4.3 of EPA Method 2.

12.1.6 *Leak Check of the Metering System*—Refer to Section 5.6 of EPA Method 5 or Section 5.5 of EPA Method 17.

12.1.7 *Barometer*—Calibrate the barometer to be used against a mercury barometer.

12.2 *Atomic Absorption or Atomic Fluorescence Spectrometer Calibration*—Perform instrument setup and optimization according to the manufacturer's specifications. Cold-vapor generation of mercury is performed via addition of stannous chloride solution to reduce oxidized mercury to its elemental state. The mercury-laden solution is then purged with a carrier gas into the atomic absorption cell. This procedure is used to calibrate the instrument using 10%^{v/v} HCl as the blank along with the standards described in Section 8.9.3. Calibration is verified by analyzing the QC standard prepared according to Section 8.9.4 of this method.

13. Procedures

13.1 *Sampling Train Operation:*

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13.1.1 Maintain an isokinetic sampling rate within 10% of true isokinetic. For an EPA Method 5 configuration, maintain sample filter exit gas stream temperatures and probe within $\pm 15^{\circ}\text{C}$ of the flue gas temperature at the sampling location. However, at no time, regardless of the sample configuration, must the sample filter, probe, or connecting umbilical cord temperature be lower than 120°C .

13.1.2 Record the data, as indicated in Figure 2, at least once at each sample point but not less than once every 5 minutes.

13.1.3 Record the dry gas meter reading at the beginning of a sampling run, the beginning and end of each sampling time increment, before and after each leak check, and when sampling is halted.

13.1.4 Level and zero the manometer. Periodically check the manometer level and zero, because it may drift during the test period.

13.1.5 Clean the port holes prior to the sampling run.

13.1.6 Remove the nozzle cap. Verify that the filter and probe heating systems are up to temperature and that the pitot tube and probe are properly positioned.

Note 3—For an EPA Method 5 configuration, prior to starting the gas flow through the system, the sample filter exit gas temperature may not be at the hot box temperature. However, if the system is set up correctly, once flow is established, the sample filter exit gas temperature will quickly come to equilibrium.

13.1.7 Start the pump. Position the nozzle at the first traverse point with the nozzle tip pointing in the direction of flow. Seal the openings around the probe and port hole to prevent unrepresentative dilution of the gas stream. Read the pitot tube manometer, start the stopwatch, open and adjust the control valve until the isokinetic sampling rate is obtained (refer to Section 4.1.5 from either EPA Method 5 or 17 for information on isokinetic sampling rate computations), and maintain the isokinetic rate at all points throughout the sampling period.

13.1.8 When sampling at one traverse point has been completed, move the probe to the next traverse point as quickly as possible. Close the coarse adjust valve, and shut the pump off when transferring the probe from one sample port to another. Exclude the time required to transfer the probe from one port to another from the total sampling time.

13.1.9 Traverse the stack cross section, as required by EPA Method 1.

13.1.10 During sampling, periodically check and, if necessary, adjust the probe and filter exit sample gas temperatures, as well as the zero of the manometer.

13.1.11 Add more ice, if necessary, to maintain a temperature of $<20^{\circ}\text{C}$ (68°F) at the condenser/silica gel outlet.

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13.1.12 Replace the filter assembly if the pressure drop across the filter becomes such that maintaining isokinetic sampling is no longer possible. Conduct a leak check (refer to EPA Method 5 or 17, Section 4.1.4.2) before installing a new filter assembly. The total particulate weight and determination of particle-bound mercury includes all filter assembly catches.

13.1.13 In the unlikely event depletion of KMnO_4 via reduction reactions with flue gas constituents other than elemental mercury occurs, it may render it impossible to sample for the desired minimum time. This problem is indicated by the complete bleaching of the purple color of the acidified permanganate solution. If the purple color is lost in the first two H_2SO_4 - KMnO_4 impingers, then the sample must be repeated. If the gas stream is known to contain large amounts of reducing constituents (i.e., >2500 ppm SO_2) or breakthrough has occurred in previous sampling runs, then the following modification is suggested: the amount of HNO_3 - H_2O_2 ($10\% \text{V/V}$) in the fourth impinger should be doubled, and/or a second HNO_3 - H_2O_2 impinger should be used to increase the oxidation capacity for reducing gas components prior to the H_2SO_4 - KMnO_4 impingers.

13.1.14 Use a single train for the entire sample run, except when simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct or when equipment failure necessitates a change of trains.

13.1.15 At the end of a sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, record the final dry gas meter reading, and conduct a posttest leak check, as described in Section 4.1.4.3 of EPA Method 5. Also, leak-check the Pitot lines as described in EPA Method 2, Section 3.1. The lines must pass the leak check to validate the velocity head data.

13.1.16 Calculate percent isokinetic to determine whether the run was valid or another test run should be performed (refer to Section 14.6 of this method).

13.2 *Sample Recovery:*

13.2.1 Allow the probe to cool before proceeding with sample recovery. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle, and place a rinsed, noncontaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling; a vacuum can form in the filter holder, with the undesired result of drawing liquid from the impingers onto the filter.

13.2.2 Before moving the sampling train to the cleanup site, remove the probe from the sampling train, and cap the open outlet. Be careful not to lose any condensate that may be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger, and cap the impinger. Cap the filter holder outlet and impinger inlet. Use

noncontaminating caps, such as ground-glass stoppers, plastic caps, serum caps, or PTFE tape, to close these openings.

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13.2.3 Alternatively, the following procedure may be used to disassemble the train before the probe and filter holder/oven are completely cooled. Initially disconnect the filter holder outlet/impinger inlet, and loosely cap the open ends. Then disconnect the probe from the filter holder or cyclone inlet, and loosely cap the open ends. Cap the probe tip, and remove the umbilical cord as previously described.

13.2.4 Transfer the probe and filter-impinger assembly to a clean area that is protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly, and note any abnormal conditions.

13.2.5 The impinger train sample recovery scheme is illustrated in Figure 3.

13.2.6 *Container 1 (Sample Filter)*—Carefully remove the sample filter from the filter holder so as not to lose any ash, weigh filter and ash, and place the filter in a labeled petri dish container. To handle the filter, use either acid-washed polypropylene or PTFE-coated tweezers or clean, disposable surgical gloves rinsed with water and dried. If it is necessary to fold the filter, make certain the particulate cake is inside the fold. Transfer any particulate matter or filter fibers that adhere to the filter holder gasket to the filter in the petri dish. A dry (acid-cleaned) nonmetallic bristle brush should be used to remove any remaining particulate matter. Do not use any metal-containing materials when recovering this train. Immediately cover and seal the labeled petri dish.

13.2.7 *Container 2/2a (All Rinses in Front of the Sample Filter)*

13.2.7.1 *Case 1: Includes Gravimetric Particulate Determination in Addition to Mercury*

Quantitatively recover particulate matter and any condensate from all components prior to the sample filter. A nonmetallic brush may be used for removing particulate matter. All front-half components (all components prior to the sample filter) are then rinsed with acetone as outlined in EPA Method 5 or 17. The acetone rinse is then placed into a container (Container 2a) for which the tare weight has been recorded. Container 2a, with a ribbed watch glass over the top, is placed in a fume hood until the acetone has completely evaporated. After the front-half components have been rinsed with acetone, then rinse these components with 0.1 N HNO₃. The 0.1 N HNO₃ rinse is placed in Container 2.

13.2.7.2 *Case 2: Mercury Determination Only (No Acetone Rinse)*

Quantitatively recover particulate matter and any condensate from all components prior to the sample filter. A nonmetallic brush may be used for removing particulate matter. The front-half components are then rinsed with 0.1 N HNO₃, and this rinse is placed in Container 2.

13.2.8 *Container 3 (Impingers 1 through 3, KCl Impinger Contents and Rinses):*

13.2.8.1 Dry the exterior surfaces of Impingers 1, 2, and 3. Then weigh and record the weight of each impinger (to the nearest 0.5 g). DRAFT

13.2.8.2 Clean the filter support, the back half of the filter housing, and connecting glassware by thoroughly rinsing with 0.1 N HNO₃. Pour the rinse into a glass sample Container 3.

13.2.8.3 Add a 5% w/v KMnO₄ solution to each impinger until a purple color remains.

13.2.8.4 Pour all of the liquid from the three KCl impingers into Container 3.

13.2.8.5 Rinse the impingers and connecting glassware with 10% v/v HNO₃. Although unlikely, if deposits remain on the impinger surfaces, remove them by doing another 10% v/v HNO₃ rinse that has a very small amount (several drops) of 10% w/v hydroxylamine sulfate solution added to each of the KCl impingers. Add these rinses to Container 3. If the solution in Container 3 becomes clear, add a small amount of the 5% w/v KMnO₄ solution until a pink or slightly purple color is obtained. Check again after 90 min to ensure the purple color remains.

13.2.8.6 Perform a final rinse of the impingers and connecting glassware with 0.1 N HNO₃, and add to Container 3.

13.2.8.7 Do a final rinse of all glass components with water which is discarded.

13.2.8.8 Mark the height of the fluid level in Container 3, seal, and clearly label the contents.

13.2.9 *Container 4 (Impinger 4, HNO₃-H₂O₂ Impinger Contents and Rinses):*

13.2.9.1 Dry the exterior surfaces of Impinger 4. Then weigh and record the weight of this impinger (to the nearest 0.5 g).

13.2.9.1 Pour the HNO₃-H₂O₂ absorbing solution into sample Container 4.

13.2.9.2 Rinse the H₂O₂-HNO₃ impinger and connecting glassware a minimum of two times with 0.1 N HNO₃, and pour the rinses into Container 4. Do a final rinse with water and discard water.

13.2.10 *Container 5 (Impingers 5 through 7, H₂SO₄-KMnO₄ Impinger Contents and Rinses):*

13.2.10.1 Dry the exterior surfaces of Impingers 5, 6, and 7. Then weigh and record the weight of each impinger (to the nearest 0.5 g).

13.2.10.2 Pour all of the liquid from the three H₂SO₄-KMnO₄ impingers into a glass sample Container 5.

13.2.10.3 Rinse the H_2SO_4 - $KMnO_4$ impingers and connecting glassware a minimum of two times with 0.1 N HNO_3 , and pour the rinses into Container 5. If deposits remain on the impinger surfaces, after the two rinses, remove them by doing a third rinse with 0.1 N HNO_3 and several drops hydroxylamine sulfate. On a drop by drop basis add more hydroxylamine sulfate until the deposit are removed. Add these rinses to Container 5. If the solution in Container 5 becomes clear, add small amounts of H_2SO_4 - $KMnO_4$ solution until a pink or slightly purple color is obtained.

13.2.10.4 Perform a final 0.1 N HNO_3 rinse of the impingers and connecting glassware follow by a water rinse. The 0.1 N HNO_3 rinse is added to Container 5, and the water rinse is discarded.

13.2.10.5 Mark the height of the fluid level, seal the container, and clearly label the contents.

Note 4—As stated earlier in the warning in Section 9.1.1, pressure can build up in the sample storage flask because of the potential reaction of $KMnO_4$ with acid. Do not fill the container completely, and take precautions to relieve excess pressure.

13.2.11 *Container 6 (Impinger 8, Silica Gel Impinger Contents):*

13.2.11.1 Dry the exterior surfaces of Impinger 8. Then weigh and record the weight of this impinger (to the nearest 0.5 g).

13.2.11.2 Note the color of the indicating silica gel to determine whether it has been completely spent, and make a notation of its condition. If spent, the silica gel must be either regenerated or disposed of.

13.2.12 *Solution Blanks (Containers 7-11)*

Solution blanks are taken each time new reagents are prepared.

13.2.12.1 *Container 7 (0.1 N HNO_3 Blank)*—Place 50 mL of the 0.1 N HNO_3 solution used in the sample recovery process into a properly labeled container. Seal the container.

13.2.12.2 *Container 8 (1 N KCl Blank)*—Place 50 mL of the 1 N KCl solution used as the impinger solution into a properly labeled container. Seal the container.

13.2.12.3 *Container 9 (5% ν HNO_3 -10% ν H_2O_2 Blank)*—Place 50 mL of the HNO_3 - H_2O_2 solution used as the nitric acid impinger reagent into a properly labeled container. Seal the container.

13.2.12.4 *Container 10 (H_2SO_4 - $KMnO_4$ Blank)*—Place 50 mL of the H_2SO_4 - $KMnO_4$ solution used as the impinger solution in the sample recovery process into a properly labeled container. Refer to Note 4 in Section 13.2.10.5 of this method.

13.2.12.5 *Container 11 (10% w/v Hydroxylamine Sulfate Blank)*—Place 100 mL of hydroxylamine sulfate solution into a properly labeled sample container. Seal the container. **DRAFT**

13.2.13 *Container 12 (Sample Filter Blank)*—Once during each field test, place into a properly labeled petri dish three unused blank filters from the same lot as the sampling filters. Seal the petri dish.

13.2.14 After all of the samples have been recovered, they must be analyzed within 45 days.

13.2.15 After all impingers and connectors have been properly rinsed and the solutions recovered, the glassware should be cleaned according to the procedures in Section 8.10 or triple-rinsed with 10% v/v HNO₃ followed by a rinsing with water. If a new source is to be sampled or if there are any brown stains on the glassware, then the glassware must be cleaned according to procedures in Section 8.10 of this method. If multiple sites are to be sampled during a single mobilization, an exception to this procedure will be allowed. In this case, a triple rinsing of the glassware with 10% v/v HNO₃ solution followed by a water rinse prior to sampling can be used as an alternative to the procedures in Section 8.10. However, if there are any brown stains on the glassware, then the glassware must be cleaned according to procedures in Section 8.10 of this method.

13.3 *Sample Preparation:*

13.3.1 *Ash Sample (Containers 1 and 2)*

13.3.1.1 *Case 1: Includes Gravimetric Particulate Determination in Addition to Mercury*—The gravimetric particulate loading is determined from the mass of the ash on the filter (Container 1) and the residual particulate from the acetone rinse (Container 2a), as outlined in EPA Method 5 or 17. If a large amount of ash is on the filter, carefully remove the ash to create a raw ash sample from which a representative weighed aliquot can be taken for digestion. If the mass of ash collected on the filter is small (less than 0.5 g), digest the entire filter along with the ash. Dissolve the residual particulate from Container 2a using concentrated HNO₃. This solution is then added to Container 2 (0.1 N HNO₃ probe rinse). The ash material from Container 1 is then digested using the procedures described in Section 13.3.2 of this method. The same procedure is used to determine the mercury on the sample filter blank.

Use a modification of EPA SW 846 7470 to digest the sample in Container 2 prior to analysis. The main modification is that the volumes of reagents and sample have been reduced tenfold to reduce waste. This reduction in reagent volume is acceptable because modern dedicated mercury analyzers do not require the large volumes that previous manual methods required. Transfer a 10-mL aliquot of the sample to a digestion tube with a screw cap.

13.3.1.2 *Case 2: Mercury Determination Only*—The same procedures are followed as described previously in Section 13.3.1.1 with the exception that there is no Container 2a.

13.3.2 *Ash Digestion*—Accomplish the complete dissolution of ash by one of the following methods or an equivalent alternative method. The following methods are for the dissolution of inorganic samples, such as ash or sediments, when an analysis of trace elements including mercury is done.

13.3.2.1 *Microwave Digestion*—The use of this method assumes proper training in microwave digestion techniques. In addition, this method is tailored for a CEM (continuous emission monitor) microwave digestion system. A 0.5-g ash sample, accurately weighed to 0.0001 g, is placed in a PTFE microwave digestion vessel with 3 mL of concentrated HF, 3 mL of concentrated HNO₃, and 3 mL of concentrated HCl. The vessel is sealed and placed in the microwave (along with other vessels). The vessels are slowly heated to a pressure of 347 kPa (50 psi), which is held for 5 minutes, followed by heating to a pressure of 550 kPa (80 psi), which is held for 20 minutes. The vessels are allowed to cool to room temperature before venting. 15 mL of 4%^{w/v} boric acid is added to each vessel. The vessels are sealed and placed in the microwave again. The vessels are slowly heated back to a pressure of 347 kPa (50 psi) and held for 10 minutes. The vessels are again allowed to cool to room temperature before venting. The contents of each vessel are quantitatively transferred to a 50-mL PMP or polypropylene (PP) volumetric flask and diluted; note that care must be taken in adding water to a strong acid solution.

13.3.2.2 *Conventional Digestion*—The use of this method assumes proper training in PTFE bomb digestion techniques. Place a 0.5-g ash sample, accurately weighed to 0.0001 g, in a PTFE digestion vessel with 7 mL of concentrated HF and 5 mL of aqua regia. Seal the vessel, and place it in an oven or water bath at 90°C for a minimum of 8 hours (these may be heated overnight). Cool the vessel to room temperature before venting. Add 3.5 g of boric acid and 40 mL of water to each vessel. Seal the vessels, and place them in the oven or water bath for an additional 1 hour. Cool the vessels again to room temperature before venting. Quantitatively transfer the contents of each vessel to a 100-mL PMP, PP, or glass volumetric flask and dilute. Note that care must be taken in adding water to a strong acid solution.

13.3.3 *Preparation of Aqueous KCl Impinger Solution (Containers 3 and 8)*—Dilute sample in a 500-mL volumetric flask to volume with water, and mix. Use a modification of EPA SW 846 7470 to digest the sample prior to analysis. The main modification is that the volumes of reagents and sample have been reduced tenfold to reduce waste. This reduction in reagent volume is acceptable because modern dedicated mercury analyzers do not require the large volumes that previous manual methods required. Transfer a 10-mL aliquot of the sample to a digestion tube with a screw cap. Add 0.5 mL of concentrated H₂SO₄, 0.25 mL of concentrated HNO₃, and 1.5 mL of 5%^{w/v} KMnO₄ solution. Mix the solution, and allow it to stand for 15 minutes. Add 0.75 mL of 5%^{w/v} K₂S₂O₈ solution, and loosely cap the tube. Place the tube in a dry block heater or water bath equipped with a temperature probe, and heat to 95°C. Do not allow the temperature to exceed 95°C. Hold the sample at 95°C for 2 hours before allowing it to cool to room temperature. The purple color from the added KMnO₄ solution must remain throughout the digestion. Clearing of the solution during the heating indicates the depletion of KMnO₄. Prior to analysis, add 1 mL of 10%^{w/v} of solid hydroxylamine sulfate solution to the sample. The sample solution should remain clear after addition of hydroxylamine sulfate.

13.3.4 *Preparation of HNO₃-H₂O₂ Impinger Solution (Containers 4 and 9)*—Dilute sample in a 250-mL volumetric flask to volume with water, and mix. Treat the sample with a modified version of EPA SW 846 7470. Modifications to the method are necessary to properly treat the H₂O₂-containing impinger solution before the analysis with CVAAS. The modifications include the addition of HCl, the use of an ice bath during the KMnO₄ addition, and the slow addition of the KMnO₄. Transfer a 5-mL aliquot of the sample to a digestion tube with a screw cap. Add 0.25 mL of concentrated HCl, place the tube in an ice bath, and allow it to cool for 15 minutes. The destruction of H₂O₂ is accomplished by slow addition of saturated KMnO₄ solution in 0.25-mL increments along the inside of the digestion tube. The violence of this reaction requires careful, slow addition of the KMnO₄ for safety reasons and to avoid loss of analyte. Cool the sample for 15 minutes in between each addition, and mix the sample prior to each addition. After the first five additions, increase the increments to 0.5 mL. Carry out the addition of KMnO₄ until the solution remains purple, indicating complete reaction of the H₂O₂. Record the volume of saturated KMnO₄ solution added to the sample. Add 0.75 mL of 5%^{w/v} K₂S₂O₈ solution to the sample, and then cap the tube loosely. Place the tubes in a dry block heater or water bath equipped with a temperature probe, and heat to 95°C. Do not allow the temperature to exceed 95°C. Maintain the sample at 95°C for 2 hours before allowing it to cool to room temperature. Note that the purple color due to KMnO₄ must remain throughout the digestion. Clearing of the solution during the heating indicates the depletion of KMnO₄. Before doing the analysis, add 1 mL 10%^{w/v} of solid hydroxylamine sulfate solution to the sample. The sample should then become clear.

13.3.5 *Preparation of H₂SO₄-KMnO₄ Impinger Solution (Containers 5 and 10)*—Prepare the solution immediately prior to analysis. Dissolve by incrementally adding approximately 500 mg of solid hydroxylamine sulfate into the sample until a clear, colorless solution persists. Add the hydroxylamine slowly because of the violence of this reaction. Dilute the sample in a 500-mL volumetric flask to volume with water, and mix. Transfer a 10-mL aliquot of the sample to a digestion tube with a screw cap. Add 0.75 mL of 5%^{w/v} K₂S₂O₈ solution to the sample, and then cap the tube loosely. Place the tube in a dry block heater or water bath equipped with a temperature probe, and heat to 95°C. Do not allow the temperature to exceed 95°C. Hold the sample at 95°C for 2 hours before allowing it to cool to room temperature.

13.3.6 *Simplification of the Digestion*—If an acetone rinse was not used for gravimetric particulate determination or it is very clear, there is insignificant organic material present in the sampled gas stream; then the digestion procedure for the HNO₃-H₂O₂ and H₂SO₄-KMnO₄ impinger solutions may be simplified by omitting the persulfate digest (the addition of K₂S₂O₈ and heating). The persulfate digest is performed for the purpose of oxidizing certain organics. Because this method is specific to coal combustion systems where organic compounds are usually insignificant,⁸ this digest may be omitted because the H₂O₂ is sufficient to oxidize most compounds. The decision to omit this procedure should be made based on the gas stream being

⁸ "A Comprehensive Assessment of Toxic Emissions from Coal-Fired Power Plants: Phase I Results from the U.S. Department of Energy Study," Prepared for the U.S. Department of Energy Federal Energy Technology Center, Contract No. DE-FC21-93MC30097, Energy & Environmental Research Center, University of North Dakota, Grand Forks, ND, 1996.

sampled and/or verification that organics resistant to H₂O₂ oxidation are not present. If unsure whether organics are present or if an acetone rinse has been used, then the total digestion procedure is required.

13.3.7 *0.1 N HNO₃ and 10%^{w/v} Hydroxylamine Sulfate Rinse Solutions (Containers 7 and 11)*—These solutions can be analyzed directly for mercury without any preparation steps.

13.4 *Sample Analysis*—Analyze all of the prepared solutions by CVAAS or CVAFS following the guidelines specified by the instrument manufacturer.

13.4.1 *QA/QC*—For this method, it is important that both the sampling team and analytical people be very well trained in the procedures. This is a complicated method that requires a high-level of sampling and analytical experience. For the sampling portion of the QA/QC procedure, both solution and field blanks are required. It should be noted that if high-quality reagents are used and care is taken in their preparation and in the train assembly, there should be little, if any, mercury measured in either the solution or field blanks.

As stated in Section 13.2.12 of this method, solution blanks will be taken and analyzed every time a new batch of solution is prepared. If mercury is detected in these solution blanks, the concentration is subtracted from the measured sample results. The maximum amount that can be subtracted is either 10% of the measured result or 10 times the instrument detection limit, whichever is less. If the solution blanks are greater than this general guideline, then the results are not valid.

A field blank is performed by assembling a sample train, transporting it to the sampling location during the sampling period, and recovering it as a regular sample. These data are used to ensure that there is no contamination as a result of the sampling activities. A minimum of one field blank at each sampling location must be completed for each test site. Any mercury detected in the field blanks cannot be subtracted from the results. Whether or not the mercury detected in the field blanks is significant is determined based on the QA/QC procedures established prior to the testing.

The QA/QC for the analytical portion of this method is that every sample, after it has been prepared, is to be analyzed in duplicate with every tenth sample analyzed in triplicate. These results must be within 10% of each other. If this is not the case, then the instrument must be recalibrated and the samples reanalyzed. In addition, after every ten samples, a known spike sample must be analyzed. For the ash samples, a certified reference ash sample (may be purchased from NIST) is to be digested and analyzed at least once during the test program. It is also suggested that the QA/QC procedures developed for a test program include submitting, on occasion, spiked mercury samples to the analytical laboratory by either the prime contractor if different from the laboratory or an independent organization.

14. Flue Gas Calculations

14.1 *Dry Gas Volume*—Calculate the dry gas sample volume, $V_{m(std)}$, at standard conditions using Equation 1.

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$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m} \right) \left| \frac{P_{bar} + \Delta H}{P_{std}} \right| = K_1 V_m Y \frac{P_{bar} + \Delta H}{T_m} \quad [\text{Eq. 1}]$$

where:

- P_{bar} = barometric pressure at the sampling site, kPa (in. Hg)
- P_{std} = standard absolute pressure, 101.3 kPa (29.92 in. Hg)
- T_m = absolute average dry gas meter temperature (refer to Figure 2), K ($^{\circ}$ R)
- T_{std} = standard absolute temperature, 293 K (528 $^{\circ}$ R)
- V_m = volume of gas sample as measured by dry gas meter, dcm (dscf)
- $V_{m(std)}$ = volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf)
- Y = dry gas meter calibration factor
- ΔH = average pressure differential across the orifice meter (refer to Figure 2), kPa (in. Hg)
- K_1 = 2.894 K/kPa (17.64 $^{\circ}$ R/in. Hg)

Note 5—Equation 1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., leak checks conducted prior to component changes or following the test) exceeds the maximum acceptable leakage rate, L_a , equal to 0.00057 m³/min (0.02 cfm) or 4% of the average sampling rate, whichever is less. If the leakage rate observed during the posttest leak check, L_p , or an individual leakage rate observed during the leak check conducted prior to the “ith” component change ($i = 1, 2, 3, \dots, n$), L_i , exceeds L_a , then Equation 1 must be modified as follows:

(a) **Case I.** No component changes made during sampling run. In this case, replace V_m with the expression:

$$[V_m - (L_p - L_a)\theta]$$

where:

- L_p = leakage rate observed during the posttest leak check, m³/min (cfm)
- L_a = maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change—equal to 0.00057 m³/min (0.02 cfm) or 4% of the average sampling rate, whichever is less
- θ = total sampling time, min

(b) **Case II.** One or more component changes made during the sampling run. In this case, replace V_m with the expression:

$$\left[V_m - (L_1 - L_a)\theta_1 - \sum_{i=1}^n (L_i - L_a)\theta_i - (L_p - L_a)\theta_p \right]$$

where:

θ_i = sampling time interval, from the beginning of a run until the first component change, min

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and substitute only for those leakage rates (L_i or L_p) that exceed L_a .

14.2 *Volume of Water Vapor*—Calculate the volume of water vapor of the stack gas using Equation 2.

$$V_{w(std)} = \frac{W_{lc} R T_{std}}{M_w P_{std}} = K_2 W_{lc} \quad [\text{Eq. 2}]$$

where:

M_w = molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)

R = ideal gas constant, 0.008314 kPa-m³/K-g-mole (21.85 in. Hg-ft³/°R-lb-mole)

W_{lc} = total weight of liquid collected in impingers and silica gel (refer to Figure 2), g

$V_{w(std)}$ = volume of water vapor in the gas sample, corrected to standard conditions, scm (scf)

K_2 = 0.001336 m³/mL (0.04707 ft³/mL)

14.3 *Volume of Moisture*—Calculate the moisture content, B_{ws} , of the stack gas using Equation 3.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \quad [\text{Eq. 3}]$$

where:

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

15. Calculations for Particle-Bound, Oxidized, Elemental, and Total Mercury Concentrations:

15.1 Particle-Bound Mercury

15.1.1 *Case 1: Amount of Ash on the Filter is Greater Than 0.5 g*—Calculate the concentration of mercury in $\mu\text{g/g}$ in the ash sample (Hg_{ash}) using Equation 4:

$$\text{Hg}_{\text{ash}}, \mu\text{g/g} = (\text{IR})(\text{DF}) \quad [\text{Eq. 4}]$$

where:

IR = instrument reading, $\mu\text{g/L}$

DF = dilution factor = (total digested volume, L)/(mass of ash digested, g)

Calculate the amount of mercury in the probe rinse (Hg_{pr} , Container 2) in μg using Equation 5:

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$$Hg_{pr}, \mu\text{g} = (IR)(V_1) \quad [\text{Eq. 5}]$$

where:

IR = instrument reading, $\mu\text{g}/\text{L}$

V_1 = total volume of probe rinse sample from which sample aliquot was taken, L

Calculate the amount of mercury on the sample filter blank (Hg_{fb}) in the same way using Equation 6.

$$Hg_{fb}, \mu\text{g} = (IR)(V_2) \quad [\text{Eq. 6}]$$

where:

IR = instrument reading, $\mu\text{g}/\text{L}$

V_2 = total volume of sample filter blank digest, L

The total amount of particle-bound mercury (Hg_{tp}) is then determined using Equation 7:

$$Hg(\text{particle}), \mu\text{g} = (Hg_{ash})(W_{ash}) - Hg_{fb} + Hg_{pr} \quad [\text{Eq. 7}]$$

where:

W_{ash} = the total ash weight on filter, g

The concentration of particle-bound mercury ($\mu\text{g}/\text{dscm}$) in the gas stream is then determined using Equation 8:

$$Hg^p, \mu\text{g}/\text{dscm} = Hg(\text{particle})/V_{m(\text{std})} \quad [\text{Eq. 8}]$$

where:

$V_{m(\text{std})}$ = is the total volume of dry gas sampled at standard (normal) conditions, dscm

15.1.2 *Case 2: Amount of Ash on the Filter is Less Than 0.5 g*—The calculation is the same as in Case 1 except the entire sample (ash and filter) is digested; therefore, DF in Equation 4 is defined only by the total digested volume. Equations 5–7 remain the same.

15.2 Oxidized Mercury

15.2.1 *KCl Solution (Impingers 1–3)*—Calculate the concentration of mercury in $\mu\text{g}/\text{L}$ in the KCl impinger solutions using Equation 9:

$$Hg_{KCl}, \mu\text{g}/\text{L} = (IR)(DF) \quad [\text{Eq. 9}]$$

where:

) IR = instrument reading, $\mu\text{g/L}$

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- DF = dilution factor, $\frac{V_D + V(H_2SO_4) + V(HNO_3) + V(KMnO_4) + V(K_2S_2O_8) + V(NH_2OH)}{V_D}$ DRAFT
- V_D = total digested volume, 10 mL
- $V(H_2SO_4)$ = volume of added concentrated H_2SO_4 , 0.5 mL
- $V(HNO_3)$ = volume of added concentrated HNO_3 , 0.5 mL
- $V(KMnO_4)$ = volume of added 5%^{w/v} $KMnO_4$, 1.5 mL
- $V(K_2S_2O_8)$ = volume of added 5%^{w/v} $K_2S_2O_8$, 0.75 mL
- $V(NH_2OH)$ = volume of added 10%^{w/v} hydroxylamine sulfate, 1.0 mL

The amount of mercury in the KCl solution blank is calculated in the same way.

15.2.2 *Total Oxidized Mercury (Hg_O)*—is defined by method as the mercury measured in the KCl sample minus the mercury measured in the KCl solution blanks, as shown in Equation 10:

$$Hg_O, \mu g = (Hg_{KCl})(V_3) - (Hg_{Ob})(V_3) \quad [Eq. 10]$$

where:

- Hg_{KCl} = Mercury concentration measured in KCl aliquot, $\mu g/L$
- V_3 = Total volume of aqueous KCl from which sample aliquot was taken, L
- Hg_{Ob} = Mercury concentration measured in KCl solution blank aliquot, $\mu g/L$

The concentration of Hg^{2+} ($\mu g/dscm$) in the gas stream is then determined using Equation 11:

$$Hg^{2+}, \mu g/dscm = Hg_O/V_{m(std)} \quad [Eq. 11]$$

where:

$V_{m(std)}$ is the total volume of dry gas sampled at standard conditions, dscm

15.3 Elemental Mercury

15.3.1 *$HNO_3-H_2O_2$ Solution (Impinger 4)*—Calculate the concentration of mercury in $\mu g/L$ in the $HNO_3-H_2O_2$ impinger solution using Equation 12:

$$Hg_{H2O2}, \mu g/L = (IR)(DF) \quad [Eq. 12]$$

where:

- IR = instrument reading, $\mu g/L$
- DF = dilution factor = $\frac{V_D + V(HCl) + V(KMnO_4) + V(K_2S_2O_8) + V(NH_2OH)}{V_D}$
- V_D = total digested volume, 5 mL
- $V(HCl)$ = volume of added concentrated HCl, 0.25 mL

-) $V(\text{KMnO}_4)$ = volume of added saturated KMnO_4 , mL (volume need to turn sample to a purple color)
- $V(\text{K}_2\text{S}_2\text{O}_8)$ = volume of added 5%^{w/v} $\text{K}_2\text{S}_2\text{O}_8$, 0.75 mL (if used)
- $V(\text{NH}_2\text{OH})$ = volume of added 10%^{w/v} hydroxylamine sulfate, 1.0 mL

turn sample to a purple
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The amount of mercury in the $\text{HNO}_3\text{-H}_2\text{O}_2$ solution blank is calculated in the same way.

15.3.2 $\text{H}_2\text{SO}_4\text{-KMnO}_4$ Solution (Impingers 5-7)—Calculate the concentration of mercury in $\mu\text{g/L}$ in the $\text{H}_2\text{SO}_4\text{-KMnO}_4$ impinger solutions using Equation 13:

$$\text{Mercury, } \mu\text{g/L} = (\text{IR})(\text{DF}) \quad [\text{Eq. 13}]$$

where:

DF = dilution factor = $\frac{V_D + V(\text{K}_2\text{S}_2\text{O}_8)}{V_D}$

IR = instrument reading, $\mu\text{g/L}$

V_D = total digested volume, 5 mL

-) $V(\text{K}_2\text{S}_2\text{O}_8)$ = volume of added 5%^{w/v} $\text{K}_2\text{S}_2\text{O}_8$, 0.75 mL (if used)

The concentration of mercury in the $\text{H}_2\text{SO}_4\text{-KMnO}_4$ solution blank is calculated in the same way.

15.3.3 Total Elemental Mercury (Hg_E)—is defined by method as the mercury measured in the $\text{H}_2\text{SO}_4\text{-KMnO}_4$ impingers plus the mercury in the $\text{HNO}_3\text{-H}_2\text{O}_2$ impingers minus the solution blanks as shown in Equation 14:

$$\text{Hg}_E, \mu\text{g} = (\text{Hg}_{\text{H}_2\text{O}_2})(V_4) - (\text{Hg}_{\text{Eb1}})(V_4) + (\text{Hg}_{\text{KMnO}_4})(V_5) - (\text{Hg}_{\text{Eb2}})(V_5) \quad [\text{Eq. 14}]$$

where:

$\text{Hg}_{\text{H}_2\text{O}_2}$ = Mercury concentration measured in $\text{HNO}_3\text{-H}_2\text{O}_2$ aliquot, $\mu\text{g/L}$

V_4 = Total volume of aqueous $\text{HNO}_3\text{-H}_2\text{O}_2$ from which sample aliquot was taken, L

Hg_{Eb1} = Mercury concentration measured in $\text{HNO}_3\text{-H}_2\text{O}_2$ solution blank aliquot, $\mu\text{g/L}$

$\text{Hg}_{\text{KMnO}_4}$ = Mercury concentration measured in $\text{H}_2\text{SO}_4\text{-KMnO}_4$ aliquot, $\mu\text{g/L}$

V_5 = Total volume of aqueous $\text{H}_2\text{SO}_4\text{-KMnO}_4$ from which sample aliquot was taken, L

Hg_{Eb2} = Mercury concentration measured in $\text{H}_2\text{SO}_4\text{-KMnO}_4$ solution blank aliquot, $\mu\text{g/L}$

The concentration of Hg^{2+} ($\mu\text{g/dscm}$) in the gas stream is then determined using Equation 15:

$$\text{Hg}^0, \mu\text{g/dscm} = \text{Hg}_E / V_{m(\text{std})} \quad [\text{Eq. 15}]$$

where:

$V_{m(\text{std})}$ is the total volume of dry gas sampled at standard conditions, dscm

15.4 *Total Mercury*—Is defined by the method as the sum of the particulate bound mercury, oxidized mercury, and elemental mercury as shown in Equation 16:

$$\text{Hg}(\text{total}), \mu\text{g}/\text{dscm} = \text{Hg}^{\text{p}} + \text{Hg}^{2+} + \text{Hg}^0 \quad [\text{Eq. 16}]$$

16. Precision and Bias

16.1 Precision

16.1.1 Formal evaluation of the Ontario Hydro method was completed with dynamic spiking of Hg^0 and HgCl_2 into a flue gas stream.⁹ The results are shown in Table 1. The relative standard deviation for gaseous elemental mercury and oxidized mercury was found to be less than 11% for mercury concentrations greater than $3 \mu\text{g}/\text{Nm}^3$ and less than 34% for mercury concentrations less than $3 \mu\text{g}/\text{Nm}^3$. In all cases, the laboratory bias for these tests based on a calculated correction factor was not statistically significant. These values were within the acceptable range, based on the criteria established in EPA Method 301 (% RSD less than 50%).

16.1.2 The precision of particle-bound, oxidized, and elemental mercury sampling method data is influenced by many factors: flue gas concentration, source, procedural, and equipment variables. Strict adherence to the method is necessary to reduce the effect of these variables. Failure to assure a leak-free system, failure to accurately calibrate all indicated system components, failure to select a proper sampling location, failure to thoroughly clean all glassware, and failure to follow prescribed sample recovery, preparation, and analysis procedures can seriously affect the precision of the results.

16.2 Bias

16.2.1 Gaseous mercury species in flue gases that are capable of interacting with fly ash particles collected in the front half of the sampling train can produce a positive particle-bound mercury bias.

16.2.2 Particle-bound mercury existing in the flue gas may vaporize after collection in the front half of the sampling train because of continued exposure to the flue gas sample stream and reduced pressures during the sampling period. Such vaporization would result in a negative particle-bound mercury bias.

⁹ EPRI. "Evaluation of Flue Gas Mercury Speciation Methods," EPRI TR-108988, Electric Power Research Institute, Palo Alto, CA, Dec. 1997.

Table 1

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Results from Formal EPA Method 301 Evaluation Tests for the Ontario Hydro Method

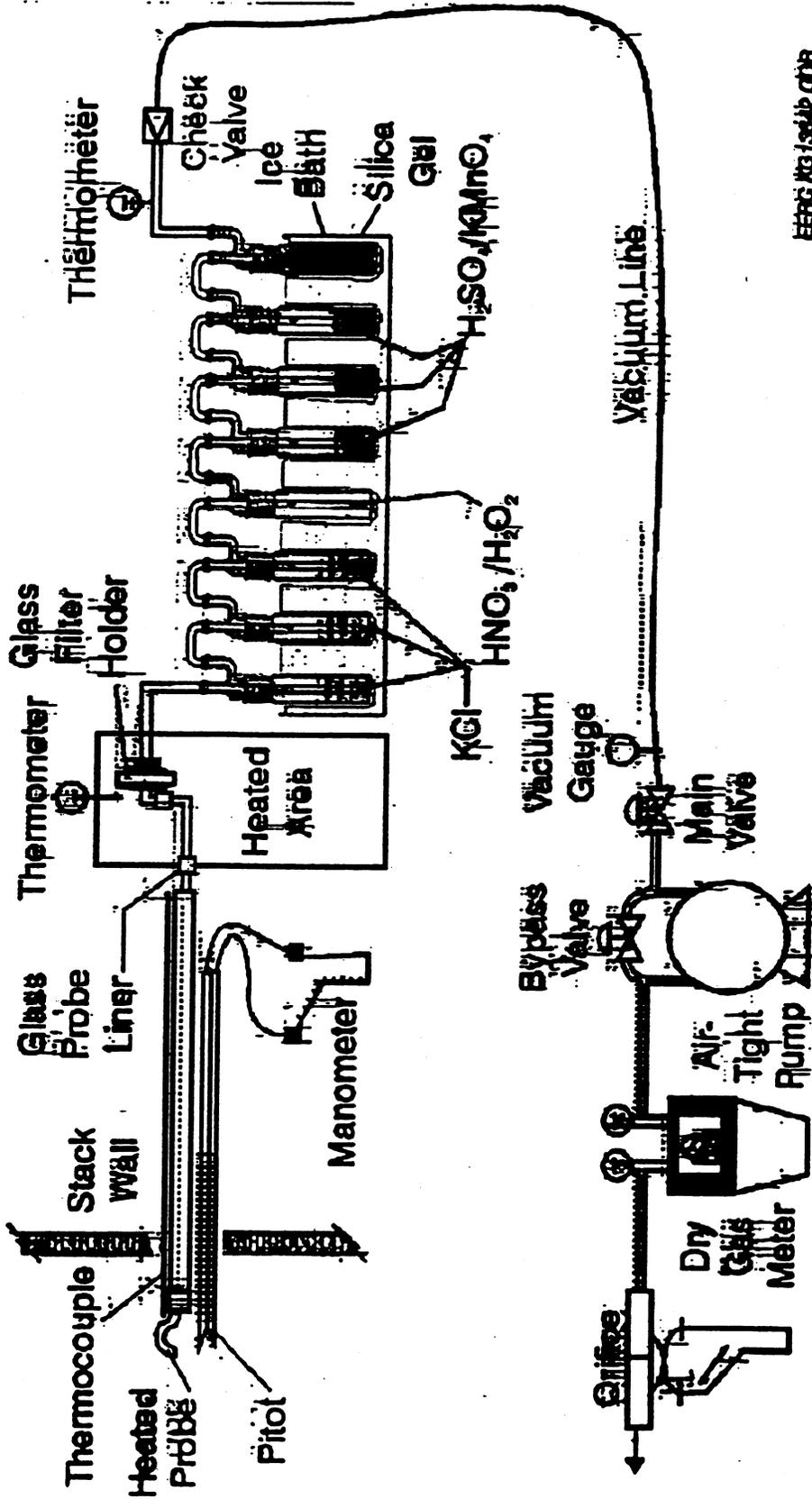
Ontario Hydro Method**	Total Vapor-Phase Mercury			Oxidized Mercury			Elemental Mercury		
	Mean, $\mu\text{g}/\text{Nm}^3$	Std. Dev.	RSD, %	Mean, $\mu\text{g}/\text{Nm}^3$	Std. Dev.	RSD, %	Mean, $\mu\text{g}/\text{Nm}^3$	Std. Dev.	RSD, %
Baseline	23.35	2.05	8.79	21.24	2.13	10.02	2.11	0.65	30.69
Hg ⁰ Spike (15.0 $\mu\text{g}/\text{Nm}^3$)	38.89	2.00	5.13	23.32	2.08	8.94	15.57	1.09	6.97
HgCl ₂ Spike (19.9 $\mu\text{g}/\text{Nm}^3$)	42.88	2.67	6.23	40.22	2.87	7.14	2.66	0.89	33.31

* For each mean result, there were 12 replicate samples (four quadrants)

** The correction factor in all cases was not statically significant and is not shown.

17. Keywords—Air toxics, mercury, sampling, speciation

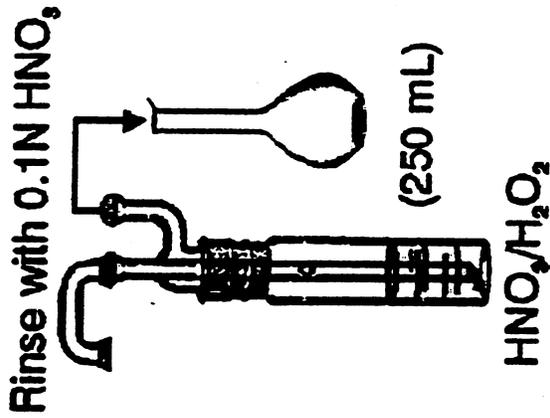
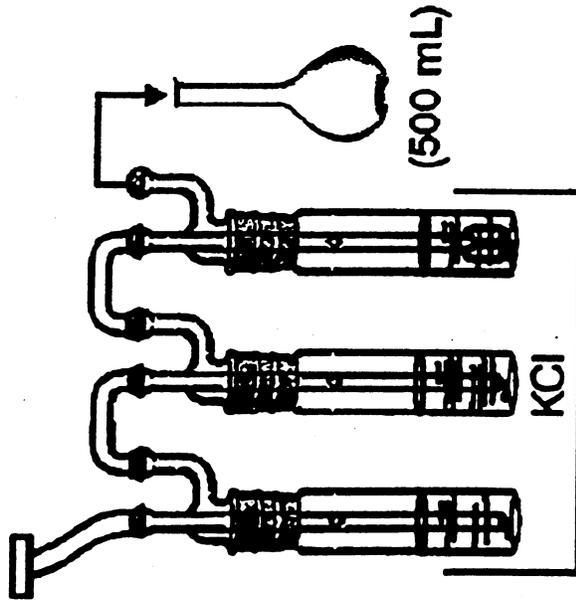
DRAFT



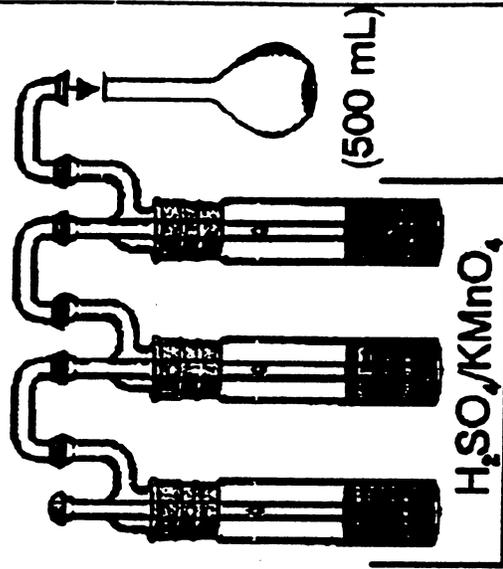
EEPC No 13542.cdr

FIG. 1. Schematic of Mercury-Sampling Train in the Method 5 configuration.

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



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




 Rinse All U-Tubes with 0.1N HNO₃

BIBLIOGRAPHY OF EPA METHODS REFERENCED

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- (1) Method 303F in *Standard Methods for the Examination of Water and Wastewater*, 15th Edition, 1980. Available from the American Public Health Association, 1015 18th Street N.W., Washington, D.C. 20036.
- (2) EPA Methods 6010, 6020, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841, *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SW-846, Third Edition. September 1988. Office of Solid Waste and Emergency Response, U. S. Environmental Protection Agency, Washington, D.C. 20460.
- (3) EPA Methods 1 through 5, *Code of Federal Regulations*, Title 40, Part 60, Appendix A, July 1, 1991.
- (4) EPA Method 101A, *Code of Federal Regulations*, Title 40, Part 61, Appendix B, July 1, 1991.
- (5) EPA Method 29, *Emission Measurement Technical Information Center*, EMTIC TM-029, April 25, 1996.
- (6) U.S. Environmental Protection Agency "Method 301 - Field Validation of Pollutant Measurement Method from Various Waste Media," In *Code of Federal Regulations*, Title 40, Parts 61 to 80. Washington, DC, USA, Office of the Federal Register, Part 63, appendix A, pp 324-331, July 1993.

USEPA REFERENCE METHOD 3A - OXYGEN AND CARBON DIOXIDE

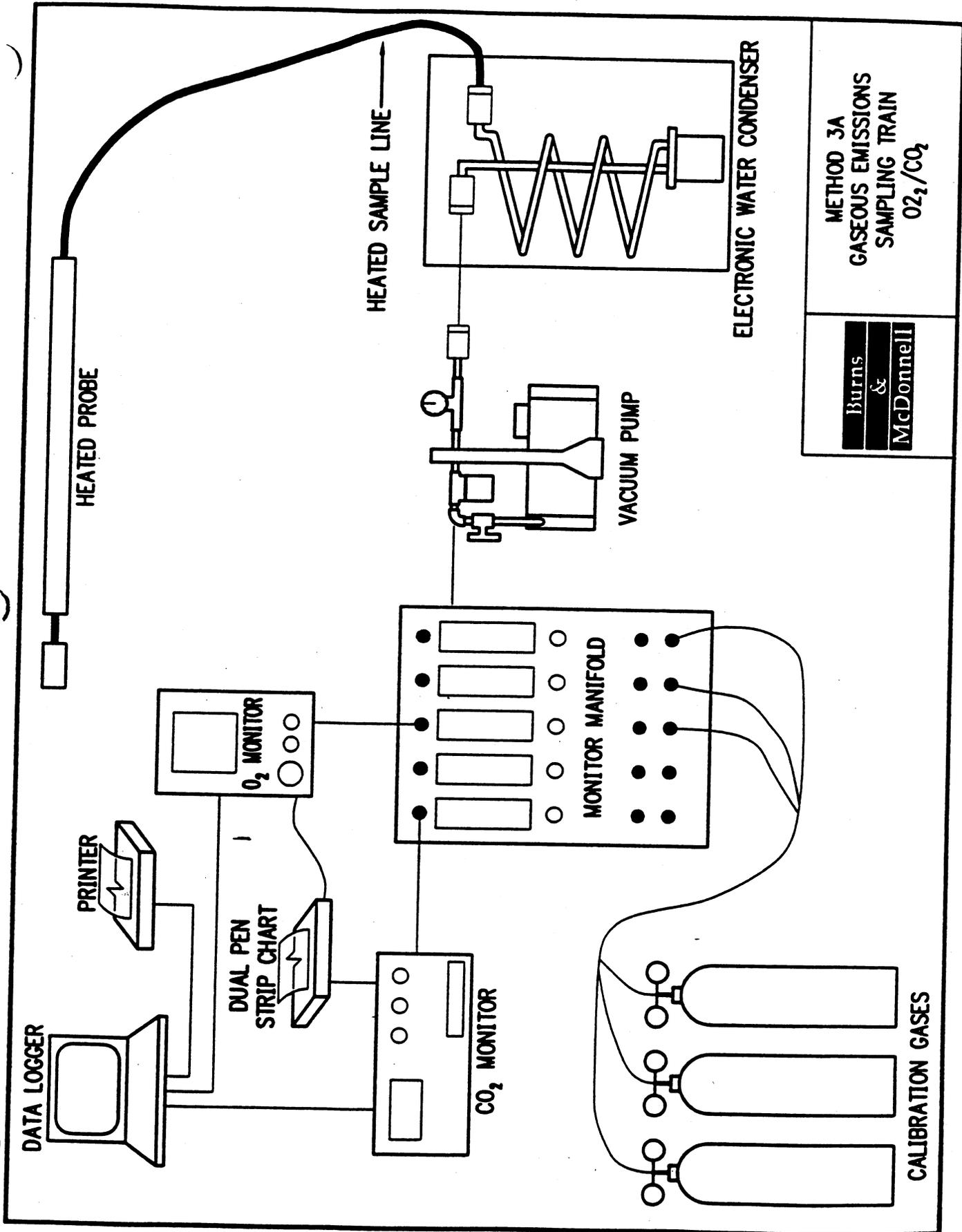
Sampling Equipment

A Beckman Corporation portable compliance monitor Model 755R O₂ analyzer and a Milton Roy portable compliance monitor Model 3400 CO₂, as shown in the exhibit, will be used for the sampling of oxygen and carbon dioxide, respectfully.

The sampling of the flue gas takes place via a porous filter connected to a T-type, stainless steel, heated probe. The sample is then transported to the sample conditioner then to the analyzer via a sample line constructed of Teflon® tubing. The sample is pulled by means of an Air Dimensions diaphragm pump with the gas flow being regulated by a Dwyer flow rate meter.

Protocol 1 calibration gases are used to calibrate the analyzers. One span gas along with ambient air will be used for the O₂ analyzer and two span gases will be used for the CO₂ analyzer. The O₂ gases will be based on a span value of 25 percent and will be in the 80-100 percent range for the high-level gas and in the 40-60 percent range for the mid-level gas. The CO₂ gases will be based on a span value of 20 percent and will be in the 80-100 percent range for the high-level gas and in the 40-60 percent range for the mid-range gas. Nitrogen gas will be used to zero the O₂ analyzer and CO₂ analyzer.

Interference gases will be introduced into the analyzers prior to use in the field. Three interference gases will be used, NO_x, CO, and SO₂ for this particular test.



METHOD 3A
GASEOUS EMISSIONS
SAMPLING TRAIN
O₂/CO₂

Burns
&
McDonnell

Sample Preparation

The sampling and conditioning portion of the system is designed to prevent loss of O₂ and CO₂ from the sample gas as follows:

The sample is drawn through a heated, filtered probe to remove any particulate and prevent it from reaching the monitor. A Teflon® sample line conveys the gas to a sample conditioner at a temperature above the dew point of the flue gas, thereby preventing condensation and subsequent losses of soluble sample components. An electronic water condenser-sample conditioner, which is a simple form of a heat exchanger, completes the sample conditioning. The object of this device is to reduce the temperature of the sample to a low enough level to allow the moisture in the sample to be regenerated in droplet form and to prevent condensation in the monitors.

The Beckman oxygen analyzer Model 755R provides a continuous readout of the oxygen content of the flowing gas sample. The determination is based on measurement of the magnetic susceptibility of the sample gas. Oxygen is strongly paramagnetic; other common gases, with only a few exceptions, are weakly diamagnetic. With the Model 755R, the volume magnetic susceptibility of the flowing gas sample is sensed in the detector/magnet assembly, which in turn displays the front panel reading on the analyzer.

The Model 3400 CO₂ infrared gas analyzer continuously analyzes a flowing gas sample based on the infrared absorption characteristics of gases to measure gas concentration. Use of an efficient single-beam design results in long-term stability. A single beam of infrared energy is modulated and passed through a sample cell containing the gas to be measured. The beam emerges attenuated by the amount of energy absorbed by the gases in the sample. Changes in the concentration of the gases result in changes of the intensity of the beam. The remaining energy in the beam is passed serially through two cavities of an infrared detector, a massflow sensor filled with gas of the type to be measured. Changes in the intensity of the beam change the pressure differential between the cavities and consequently the balance of the electrical bridge in the detector circuit. Electronic processing and linearization of the imbalance signal are used to generate an electrical output signal linearly proportional to the concentration of the gas measured.

Sampling Procedures

Sampling will be conducted from the test port located in the duct. Prior to any sampling, the analyzers will be set up by introducing the calibration gases at the back of the analyzers one at a time and setting the appropriate span and zero values. This data will be recorded on the computer data logger and the field data sheet.

The upscale calibration gases and nitrogen gas will be then introduced to the sampling system via a valve assembly at the outlet of the sampling probe in order to perform the sampling system bias check. These values will be recorded on the computer data logger and the field data sheet. A system bias less than five percent is considered valid. These values are then used to calculate the actual reference method gas concentrations used for the emission rates.

The upscale calibration gases and nitrogen gas will be then reintroduced to the sampling system to observe the time required to display a 95 percent stepchange in gas concentration on the computer logger. This response time will be recorded on the field data sheet.

The probe will be then positioned in the gas stream, heated at a controlled temperature ($248^{\circ}\text{F} \pm 25^{\circ}\text{F}$), and the sample pump turned on. The average steady-state concentrations of O_2 and CO_2 at the sample points will be recorded on the computer data logger. A post-test system calibration bias and drift test will be performed on the analyzers after each test run. Three (3) test runs will be performed for the source emissions testing.

5.0 QA/QC ACTIVITIES

5.1 Quality Control Procedures

Quality control is of the utmost importance to each member of the test team. The QA officer reviewed all aspects of this test program and communicated to the project manager any unacceptable finding and discrepancies. Unacceptable findings and discrepancies were corrected at once and re-submitted for approval by the QA officer.

Table 5-1 is provided and is used as a guideline pertaining to the frequency and allowable deviation from equipment calibrations. Raw equipment calibration data sheets are included for reference, in the appendix.

Prior to sampling, a velocity traverse and 30 minute moisture test was performed. This data was used to determine the proper nozzle size and amount of flue gas anticipated to be sampled.

At the conclusion of each test run, the isokinetic sample rate was calculated to determine if the sample rate was acceptable, and if another test run was needed to be performed.

Laboratory analytical procedures and internal quality control procedures are to be strictly adhered to. Table 5-2 shows the solution blanks that were required for this test program. The Ontario Hydro Method also calls for a field blank to be taken at each test location. At the conclusion of the test, the field blank sample was retrieved using the procedures as illustrated in Table 5-1.

5.2 Quality Assurance Audits

Prior to the initial test run, each sample location was required to run a dry gas meter calibration check.

The analytical laboratory was issued a mercury sample audit, supplied by Burns & McDonnell. This audit must be analyzed within ten (10) percent to be acceptable.

5.3 QA/QC Checks of Data Reduction

All averaging of data is rounded off to one extra decimal place beyond the reported value. The QA officer performed an independent check of all calculations; re-averaging the raw data and inputting the data into an applicable computer program.

Upon receipt of the coal analysis, calculations were performed to determine the amount of mercury available within the flue gas. The calculation was performed based on the average mercury content of the coal (0.09 ppm) and a typical coal feeder reading total (226,000 lbs coal/hr). This calculation indicated that the total mercury in the coal burned during the test runs was approximately 0.065 lb/hr. The highest total mercury emission estimated from Run 2 at on ESP inlet (0.021×2) was 0.062 lb/hr. This indicates that the majority of the mercury in the coal remained in the flue gas stream at the ESP inlet.

5.4 Sample Identification and Custody

All samples were properly tagged using a Sample ID Tag as shown in Figure 5-1. Each section of the form were completed. Non-applicable sections were noted as such. Identification of each bottle were crossed referenced to the sample recovery procedures, Table 5-3.

The field test manager was responsible for complete control of all field data sheets and samples. After collection and retrieval of the samples, an integrity seal, Figure 5-2, was affixed to the sample, and stored in a safe and secure location.

The chemical analysis and chain of custody form, Figure 5-3, was completed by the sample retrieval parties, and provided to the field manager.

TABLE 5-2
SOLUTION BLANKS

Note: Solution Blanks are taken each time new reagents are prepared.

0.1 N HNO ₃	0.1 N KCl	5% ^{v/v} HNO ₃ – 10% ^{v/v} H ₂ O ₂	H ₂ SO ₄ – KMnO ₄	10% ^{w/v} Hydroxylamine Sulfate	Sample Filter
Place 50 ml of solution in container	Place 50 ml of solution in container	Place 50 ml of solution in container	Place 50 ml of solution in container	100 ml	
Seal and Label Container SB1	Seal and Label Container SB2	Seal and Label Container SB3	Seal and Label Container SB4	Seal and Label Container SB5	Required only Once During Test – Place 1 Filter from the Lot in a Petri Dish, Seal and Label Container SBF6

**FIGURE 5-1
SAMPLE ID TAG**

Burns & McDonnell

CLIENT _____
PROJECT # _____
FACILITY _____
SAMP. LOC. _____
DATE _____
RUN NO. _____
SAMPLE I.D. _____
TARE WT. _____ gm

**FIGURE 5-2
SAMPLE CUSTODY SEAL**

Custody Seal

Burns & McDonnell	_____
	Date
	Signature

SAMPLING EQUIPMENT PRETEST AUDIT

Plant Name MID-America

Run By Kyle Blum Date 9/13/99

Facility Type Fossil Fuel Power Plant

Project No. 99-343-3

Test Loc. Unit 4 Inlet Duct

Meter No. 790

Dry Gas Meter Reading (Cubic Ft.)	Meter Temperature in Rankine (Fahrenheit + 460)	Lower and Upper Limits for Gamma
Initial <u>963.626</u>	In <u>531</u>	.97 * Y <u>.96709</u>
Final <u>971.467</u>	Out <u>530</u>	1.03 * Y <u>1.02691</u>

Vm = 7.841

Avg. Temp. 531 R

$$Y_c = \frac{\text{Run Time}}{V_m} \left| \frac{(0.0319) T_d}{P_{bar}} \right|^{1/2}$$

Y = .997

Delta H@ 1.79

$$Y_c = \frac{(10)}{(7.841)} \left| \frac{0.0319 (531)}{(29.15)} \right|^{1/2}$$

Pbar 29.15

Run Time 10 min

Yc = .9722

Acceptable Range for Gamma
$.97 * Y < Y_c < 1.03 * Y$
$(.9671) < .9722 < (1.0269)$

Gamma acceptable between lower and upper limits?

Yes

No

Thermocouples *	Allowable Deviation From Ambient	Ambient Temperature, Deg. F
Dry Gas Meter	+/- 5.4 Deg. F	
Impinger Exit	+/- 2.0 Deg. F	
Filter Box	+/- 5.4 Deg. F	
Stack **	+/- 1.5% Deg. F	

* Reference Thermometer Initial Ambient Temperature, _____

** 1.5% of the Minimum Absolute Stack Temperature _____

FIGURE 3-1

SAMPLING EQUIPMENT PRETEST AUDIT

Plant Name MID AMERICAN - NEAL SOUTH

Run By B. BOWLING Date 9/14/99

Facility Type COAL FIRE BOILER

Project No. 99-3433

Test Loc. STACK UNIT 4

Meter No. 663.1

Dry Gas Meter Reading (Cubic Ft.)	Meter Temperature in Rankine (Fahrenheit + 460)	Lower and Upper Limits for Gamma
Initial <u>247.559</u>	In <u>520</u>	.97 * Y <u>.978</u>
Final <u>255.047</u>	Out <u>521</u>	1.03 * Y <u>1.038</u>

Vm = 7.488

Avg. Temp. 521

$$Y_c = \frac{\text{Run Time}}{V_m} \left| \frac{(0.0319) T_d}{P_{bar}} \right|^{1/2}$$

Y = 1.008

Delta H@ 2.65

$$Y_c = \left(\frac{10}{7.488} \right) \left| \frac{0.0319 (521)}{28.95} \right|^{1/2}$$

Pbar 28.95

Run Time 10 MIN

Yc = 1.012

Acceptable Range for Gamma
$.97 * Y < Y_c < 1.03 * Y$
$(.978) < 1.012 < (1.038)$

Gamma acceptable between lower and upper limits? Yes No

Thermocouples *	Allowable Deviation From Ambient	Ambient Temperature, Deg. F
Dry Gas Meter	+ - 5.4 Deg. F	
Impinger Exit	+ - 2.0 Deg. F	
Filter Box	+ - 5.4 Deg. F	
Stack **	+ - 1.5% Deg. F	

* Reference Thermometer Initial Ambient Temperature, _____

** 1.5% of the Minimum Absolute Stack Temperature _____