JV TASK 24 – INVESTIGATION OF THE FATE OF MERCURY IN A COAL COMBUSTION PLUME USING A STATIC PLUME DILUTION CHAMBER

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INVESTIGATION OF THE FATE OF MERCURY IN A COAL COMBUSTION PLUME USING A STATIC PLUME DILUTION CHAMBER

EXECUTIVE SUMMARY

Background

The 1990 Clean Air Act Amendments (CAAAAs) required the U.S. Environmental Protection Agency (EPA) to determine whether the presence of mercury in the stack emissions from fossil fuel-fired electric utility power plants poses an unacceptable public health risk. In December 2000, the EPA issued an intent to regulate for mercury from coal-fired boilers.

Mercury is emitted by both natural and human processes and cycles through atmospheric, aquatic, and terrestrial environments. The chemical form of mercury affects its transport through air, land, and water, as well as its chemical and biological behavior. Therefore, determination of speciation in a cooling coal combustion plume is important in estimating mercury fate and effects through atmosphere. However, little is known about the mercury transformations that occur in the plume. The mercury transformations that occur in the plume determine the rate and the form of mercury deposited in lakes and streams. Therefore, a logical step in mercury research is to apply what we know and extend this understanding slightly beyond the system to the plume region. Frontier Geosciences has developed a static plume dilution chamber (SPDC) that was designed to simulate plume conditions in the atmosphere. The SPDC is a 0.5-m³ chamber designed to simulate light and rain events and allow extensive mercury sampling as a function of time. A schematic of the SPDC is shown in Figure ES-1.

The SPDC has the potential to simulate plume and atmospheric effects on mercury that may help researchers understand the mercury chemistry in the plume. Based on previous results, this device has the potential to provide researchers with a relatively inexpensive and simple tool that can be used to study mercury transformations occurring just beyond energy conversion systems in the atmosphere in a simulated plume environment. Although the results at three field tests have demonstrated positive results, the SPDC needed to be tested under controlled conditions.

The Energy & Environmental Research Center (EERC), with help from Frontier Geosciences, conducted pilot-scale tests to more fully evaluate the SPDC under more controlled conditions than what occur at a full-scale system. This work was done in partnership with the U.S. Department of Energy (DOE), EPRI, and EPA through the EERC's Center for Air Toxic Metals (CATM).

Goals and Objectives

The overall goal of the project was to further develop and then verify SPDC's ability to determine the physical and chemical transformations of mercury in combustion stack plumes. Specific objectives of the project were to perform controlled tests at the pilot scale using dynamic

Figure ES-1. Schematic of the static plume dilution chamber.

spiking of known mercury compounds (i.e., Hg^0 and HgCl_2) to prove the ability of the SPDC to determine the following:

- Whether mercury condenses onto particulate matter in a cooling plume
- Whether there is reduction of Hg^{2+} to Hg^0 occurring in hygroscopic aerosols
- Whether condensed Hg^{2+} on particles is photochemically reduced to Hg^0
- Whether or not the Solid Ontario Hydro mercury speciation method (SOH) provides the same results as the Ontario Hydro (OH) mercury speciation method.

Approach

The overall test matrix is shown in Table ES-1. As can be seen in the table, the primary variables for the tests were particulate collection device (the level of particulate matter in the SPDC), mercury spiking, the use of light source, and the use of simulated rain. In addition, a test was completed to determine if ozone has an effect on the mercury speciation in the SPDC (Test 10). The coal chosen for the tests was a Blacksville eastern bituminous coal.

Table ES-1. Test Matrix^{a,b,c}

Test No.	Fuel	Particulate Control Device	Hg Spike ^d	Light	Simulated Rain	Ozone, ppb
1	Natural gas	Baghouse	Hg ⁰	No	No	0
2	Natural gas	Baghouse	HgCl ₂	No	No	0
3	Blacksville	ESP ^e	None	No	No	0
4	Blacksville	ESP	None	Yes	Yes	0
5	Blacksville	ESP	None	Yes	No	0
6	Blacksville	ESP	HgCl ₂ + Hg ⁰	No	No	0
7	Blacksville	ESP	HgCl ₂ + Hg ⁰	Yes	Yes	0
8	Blacksville	ESP	HgCl ₂ + Hg ⁰	No	Yes	0
9	Blacksville	ESP	HgCl ₂ + Hg ⁰	Yes	No	0
10	Blacksville	ESP	HgCl ₂ + Hg ⁰	Yes	No	200
11	Blacksville	Baghouse	None	No	No	0
12	Blacksville	Baghouse	None	Yes	Yes	0
13	Blacksville	Baghouse	HgCl ₂ + Hg ⁰	No	No	0
14	Blacksville	Baghouse	HgCl ₂ + Hg ⁰	Yes	Yes	0
15	Blacksville	Baghouse	HgCl ₂ + Hg ⁰	No	Yes	0
16	Blacksville	Baghouse	HgCl ₂ + Hg ⁰	Yes	No	0

^a The dilution ratio for the chamber was between 140:1 and 200:1 for all runs.

^b Hg⁰ was monitored continuously (every 3–5 minutes).

^c Hg²⁺ and particulate-bound mercury were sampled at 6, 30, 60, and 120 minutes for each test.

^d The HgCl₂ and Hg⁰ spikes were nominally 12 or 15 µg/dm³.

^e Electrostatic precipitator.

For each test, the speciated flue gas mercury was measured using the OH and the SOH methods. After a known volume of flue gas was injected into the SPDC, speciated vapor-phase mercury measurements were made as a function of time using a Tekran mercury continuous emission monitor (CEM) with a KCl denuder. The test was then conducted based on the test matrix shown in Table ES-1. Following each test, rinses were made of the unit, and the mercury was measured in the rinses.

Results

To ensure valid results, reasonable mercury balances were obtained around the SPDC. For purposes of these tests, mercury balances ±25% were considered reasonable. The balances were made based on the following:

- The mercury into the SPDC:
 - Mercury in the injected flue gas
 - Mercury in the dilution air (filtered ambient air)
 - Mercury in the sampling makeup air (filtered ambient air)
- The mercury out of the SPDC:
 - Mercury leaving the system as part of the sampling activities
 - Mercury remaining in the chamber at the end of the test
 - Mercury in the rinses.

Except for the first test on the natural gas, all the mercury balances were within acceptable limits.

The SPDC results show a very rapid decrease in both Hg^{2+} and particulate-bound mercury. It appears the rate of decrease is so fast that it occurs even before the first real measurement can be made. Two explanations are possible for the rapid decrease. First, the Hg^{2+} could have been reduced to Hg^0 ; secondly, the Hg^{2+} may have collected along the walls of the SPDC and been removed by the rinses. If there was a reduction of Hg^{2+} to Hg^0 , then a corresponding increase in Hg^0 should have been observed. In fact, there was a corresponding increase in Hg^0 for almost all the tests. However, the increase in the mass of Hg^0 is substantially less than the decrease in the mass of Hg^{2+} . Although it appears there is some reduction of Hg^{2+} to Hg^0 , there are also clearly substantial wall effects within the SPDC chamber. The overall change in Hg species within the SPDC chamber for each of the tests is shown in Table ES-2. An example of these results is shown graphically in Figure ES-2.

Table ES-2. Change in Mercury Species in the SPDC

Test No.	Hg^{2+} Mass Change, ng	Hg^0 Mass Change, ng	Difference, ng
3	47.08	6.79	40.29
4	43.67	8.19	35.48
5	37.33	6.59	30.74
6	77.38	10.07	67.31
7	79.89	11.14	68.75
8	75.88	7.9	75.09
9	97.42	10.16	87.26
10	60.01	17.79	42.22
11	56.86	-2.93	59.79
12	56.17	2.30	53.87
13	88.55	9.62	78.93
14	87.05	11.54	75.51
15	82.45	12.70	69.75
16	104.72	0.04	104.68

Figure ES-2. The change in mercury speciation as a function of time for SPDC Test 4.

As part of the SPDC testing, a comparison of the SOH method was made to the OH method sampling for speciated mercury. Paired sampling trains were run. The statistical comparison for each mercury species from the methods are shown in Table ES-3. In comparing the two methods, the calculated t-statistic is less than the t-value for each of the measured mercury species. Therefore, the two methods are statistically similar not only for total mercury but Hg^{2+} , Hg^0 , and particulate-bound mercury as well.

Table ES-3. Statistical Comparison between the OH and SOH Mercury Sampling Methods

Statistical Component	Total Hg	Hg^0	Hg^{2+}	Particulate-Bound Hg
Mean Difference, ng	1.2500	0.2425	0.5188	0.6229
Mean Standard Deviation, ng	3.2490	1.0826	3.2486	1.1667
Calculated t-statistic	1.5390	1.4125	0.8958	0.6388
t-statistic (95% confidence)	1.7530	1.7530	1.7530	1.9430

Conclusions

Based on the SPDC tests, the following conclusions can be made:

- Good mass balances were obtained for the tests, $100 \pm 25\%$.

- The Hg^{2+} and the particulate-bound Hg decrease rapidly to at or near zero.
- For almost all tests, a rapid increase in Hg^0 occurred, however, the increase was not nearly to the degree that the Hg^{2+} and particulate bound-mercury decreased.
- The data appear to indicate that some reduction of Hg^{2+} to Hg^0 does take place in the SPDC (or plume?).
- The size of the SPDC (0.5 m^3) resulted in severe wall effects; a high percentage of the mercury goes to the wall to be removed later by the rinses.
- Because of the very rapid decrease in Hg^{2+} and the particulate-bound Hg, it was not possible to statistically evaluate the effects of light and rain on mercury chemistry in the SPDC.
- Because of the wall effects, a static SPDC is not a very useful tool in understanding mercury chemistry in combustion plumes. However, a dynamic system where flue gas is constantly flowing through the device and mixing with air may eliminate these effects.

Sampling Methodology

- The Tekran mercury CEM with KCl denuders appears to work well in speciating and measuring low levels of mercury.
- The SOH method statistically gives the same results as the OH mercury speciation method.

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1.0 INTRODUCTION

The 1990 Clean Air Act Amendments (CAAAAs) required the U.S. Environmental Protection Agency (EPA) to determine whether the presence of mercury in the stack emissions from fossil fuel-fired electric utility power plants poses an unacceptable public health risk. EPA's conclusions and recommendations were presented in the *Mercury Study Report to Congress* (1) and the *Utility Air Toxics Report to Congress* (1). The first report addressed both the human health and environmental effects of anthropogenic mercury emissions, while the second addressed the risk to public health posed by the emission of mercury and other hazardous air pollutants from steam-electric generating units. Although these reports did not state that mercury controls on coal-fired electric power stations would be required, they did indicate that EPA views mercury as a potential threat to human health. In December 2000, the EPA did issue an intent to regulate for mercury from coal-fired boilers.

Mercury is emitted by both natural and human processes and cycles through atmospheric, aquatic, and terrestrial environments. Forms of mercury that appear most important in these environments are elemental mercury (Hg^0), inorganic or oxidized mercury (primarily Hg^{2+}), and methylmercury. The chemical form of mercury affects its transport through air, land, and water, as well as chemical and biological behavior. Elemental mercury is known to circulate in the atmosphere for about 1 to 2 years before it is deposited (2). Oxidized mercury in the atmosphere can be deposited directly to water bodies or transported from land by runoff and enter ponds, streams, rivers, lakes, etc. The water bodies contain microorganisms that have the metabolic capability to carry out chemical reactions that bind Hg^{2+} to methyl groups, producing methylmercury.

Although in-stack mercury speciation measurements are essential to develop and test control technologies and to provide data for input into atmospheric deposition models, the determination of speciation in a cooling coal combustion plume is more relevant for use in estimating mercury fate and effects. However, little is known about the mercury transformations that occur in the plume. The mercury transformations that occur in the plume determine the rate and the form of mercury deposited in lakes and streams. Although substantial research has been done in the past on mercury transformations within energy conversion systems—determining the concentrations of speciated mercury at the stack and doing ground-level atmospheric measurements—little has been done to determine the mercury chemistry, kinetics, and thermodynamics in the flue gas plume.

Therefore, a logical step in mercury research is to apply what we know to extend this understanding slightly beyond the system to the plume region. Frontier Geosciences has developed a static plume dilution chamber (SPDC) that was designed to simulate plume conditions in the atmosphere. The SPDC has the potential to simulate plume and atmospheric effects on mercury that may help researchers understand the mercury chemistry in the plume. Based on previous results, this device has the potential to provide researchers with a relatively inexpensive and simple tool that can be used to study mercury transformations occurring just beyond energy conversion systems in the

atmosphere in a simulated plume environment. Although the results at three field tests have demonstrated positive results (3), the SPDC needed to be tested under controlled conditions with mercury measurement methods that have been shown to correctly speciate mercury.

The Energy & Environmental Research Center (EERC), with help from Frontier Geosciences, conducted pilot-scale tests to more fully evaluate the SPDC under more controlled conditions than what occurs at a full-scale system. This work was done in partnership with the U.S. Department of Energy (DOE), EPRI, and EPA through the EERC's Center for Air Toxic Metals (CATM).

2.0 GOALS AND OBJECTIVES

The overall goal of the project was to further develop and then verify SPDC's ability to determine the physical and chemical transformations of mercury in combustion stack plumes. Specific objectives of the project were to perform controlled tests at the pilot scale using dynamic spiking of known mercury compounds (i.e., Hg^0 and HgCl_2) to prove the ability of the SPDC to determine the following:

- Whether mercury condenses onto particulate matter in a cooling plume
- Whether there is reduction of Hg^{2+} to Hg^0 occurring in hygroscopic aerosols
- Whether condensed Hg^{2+} on particles is photochemically reduced to Hg^0

3.0 FACILITY DESCRIPTION

3.1 Description of SPDC

The SPDC was a 1/2-cubic meter Teflon-coated reactor, as shown in Figure 1. Light was shone into the SPDC using a 1000-watt tungsten halogen quartz lamp. A total of ten ports with Teflon fittings allowed for plume input and sampling. The SPDC temperature was controlled by an insulated heating blanket. For these tests, the SPDC was maintained at a temperature of 300°F. A photograph of the unit is shown in Figure 2. It should be noted that the mist chambers shown in Figure 1 have been replaced with a Tekran mercury CEM (continuous emission monitor), KCl denuders, and a filter.

Prior to use, the SPDC is thoroughly cleaned, and fresh filter air is then pumped through the unit. After several complete exchanges of air, some of the air in the SPDC is evacuated until a negative pressure of about 5 psi is obtained. A known amount of flue gas is then introduced into the SPDC using a heated isokinetic sampling probe, as shown in Figure 1, for a set period of time (either 1 or 2 minutes). The flue gas in all cases was drawn from a location well downstream of the particulate control device.

Figure 1. Schematic of the static plume dilution chamber (SPDC).

Figure 2. Photograph of the SPDC.

Following the injection of flue gas into the SPDC, filtered fresh air was allowed into the chamber until the SPDC was at an equilibrium with the ambient pressure. This resulted in good mixing of the flue gas in the chamber. The dilution of the flue gas with ambient air was to simulate plume dilution. The gas within the SPDC was allowed to react for a known amount of time (approximately 1 hour). The maximum dilution ratio depended on the incoming flue gas concentration, and although it could be as great as 500:1 (typical for a plume 5–10 km distant from the stack), for these tests, the dilution ratio was between 80 and 200 to 1.

After a predetermined aging time, a sample of the gas in the SPDC was withdrawn from three different locations and the mercury species measured using a Tekran mercury CEM system. For all samples, total mercury, Hg^{2+} , Hg^0 , and particulate-bound mercury were measured. Particulate-bound mercury and Hg^{2+} loss to the walls were recovered by pressure spray washing first with double-deionized water, then followed by 0.01%/3% v/v HCl/KCl solution. The wall wash was filtered in real time through a 0.2- μm cellulose nitrate filter. This allowed for the determination of both particulate and dissolved mercury in the aqueous phase.

Real-time rainwater washout simulation was done while the flue gas plume was introduced into the SPDC. For these tests simulated rainwater (10^{-9} molar H_2SO_4) was continuously sprayed into the SPDC falling through the plume. After a known amount of time, the rainwater was collected at the bottom of the SPDC and immediately filtered through 0.2- μm cellulose nitrate filters to separate dissolved versus particulate-bound mercury in the rainwater sample. The gas remaining in the SPDC was then sampled to determine Hg^0 , Hg^{2+} , particulate-bound mercury, and mercury wall loss.

3.2 Description of the Pilot-Scale Particulate Test Combustor (PTC)

The pilot-scale tests were conducted using the EERC PTC, with either an electrostatic precipitator (ESP) or a baghouse as the downstream particulate control device. This combustor has been extensively used by the EERC for a variety of work over the years, including tests to evaluate a catalytic fabric filter for NO_x reduction, projects to evaluate mercury measurement methods and control technologies, and projects for removing fine particulate matter. The following is a short description of the pilot-scale facilities.

The PTC is a 580-MJ/hr (550,000-Btu/hr) pulverized coal-fired unit designed to generate fly ash and flue gas chemistry representative of that produced in a full-scale utility boiler. Coal is introduced to the primary air stream via a screw feeder and ejector. An electric air preheater is used for precise control of the combustion air temperature. The PTC instrumentation permits system temperatures, pressures, flow rates, flue gas constituent concentrations, and ESP operating data to be monitored continuously and recorded on a data logger.

The PTC (shown in Figure 3) is designed to operate in conjunction with either an ESP or a fabric filter. The ESP is a single-wire, tubular ESP, with a specific collection area of 125 $\text{ft}^2/1000 \text{ acfm}$ ($0.41 \text{ m}^2/\text{m}^3$) at 149°C (300°F) and a plate spacing of 27.9 cm (11 in.). Since the flue gas flow rate for the PTC is 3.67 scmm (130 scfm), the gas velocity through the ESP is 1.5 m/min (5 ft/min). The ESP has an electrically isolated plate that is grounded through an ammeter,

Figure 3. Schematic of the PTC.

allowing continual monitoring of the actual plate current to ensure consistent operation of the ESP from test to test. A 0–100 kV (0–10 mA) power supply with sparking control is used to provide power to the ESP.

The pulse-jet baghouse used for these tests has a 0.508-m (20-in.) internal chamber that is heat-traced and insulated, with the flue gas introduced near the bottom. Three 3.96 m × 12.7 cm (13 ft × 5 in.) bags provide an air-to-cloth ration of 1.22 m/min (4 ft/min), based on typical flue gas production rate of 5.66 actual m³ (200 acfm) at 149°C (300°F). Each bag was cleaned separately with its own diaphragm pulse valve. So that difference in pressure drop for each test condition could be quantified, the bags were cleaned on a timed basis rather having the cleaning cycle be initiated by the pressure drop. Once bag cleaning was initiated, all three bags were pulsed in rapid succession on-line. The bags were composed of PTFE with GORE-TEX[®] expanded membrane.

3.3 Description of Sampling Procedures

As shown in Table 1, very extensive measurements were made on the device to determine total mercury, particulate-bound mercury, and gas-phase mercury (i.e., Hg⁰ and Hg²⁺). Included as one of the sampling methods was the newly developed solid sorbent Ontario Hydro method (SOH). This method was compared to the OH wet-chemistry method. The results comparing the OH and SOH method are discussed later in this report. The advantage of the solid sorbent method is simplicity and ease of use.

Table 1. Sampling Methods for SPDC and Flue Gas Mercury

Method	Matrix	Measurement
Gold Traps	Plume air	Total Hg
Quartz Filter	Plume air	Particulate Hg
KCl Annular Denuder with Tekran	Plume air	Gas-phase Hg ²⁺ and total Hg
Tekran Mercury Analyzer ^a	Plume air	Total Hg
KCl Annular Denuder – Thermal Desorption into CVAFS ^b	Plume air	Gas-phase Hg ²⁺
Quartz Filter Behind Denuder – Thermal Desorption into CVAFS	Plume air	Particulate-bound Hg
Deionized Water Rinse	Water	Surface loss
3% KCl Rinse	Water	Surface loss
Simulated Rainwater (molar H ₂ SO ₄)	Water	Washout of particulate-bound Hg and gas-phase Hg ²⁺
Cellulose Nitrate Filter	Water	Dissolved/particulate-bound Hg
Ontario Hydro Method	Flue gas	Hg ⁰ , Hg ²⁺ , and particulate-bound Hg
Solid Ontario Hydro Method (SOH)	Flue gas	Hg ⁰ , Hg ²⁺ , and particulate-bound Hg
Sorbent Ontario Hydro Method	Flue gas	Hg ⁰ and Hg ²⁺

^a Continuous emission monitor.

^b Cold-vapor atomic fluorescence spectroscopy.

3.3.1 The SOH Method

Because of the advantages of the SOH method for mercury speciation (including simplicity, no hazardous solutions, precision, sensitivity, accuracy, and cost), part of the scope of the project was to help validate the SOH method by intercomparing it to the accepted OH mercury speciation method. Flue gas measurements were made using both the Ontario Hydro method and the SOH, simultaneously.

The principle of the SOH method has been described previously in Prestbo and Bloom (4) and Prestbo and Tokos (5). It is a sampling method for mercury, which quantifies not only total mercury, but also the speciation between gas-phase Hg²⁺ and Hg⁰. It also identifies to some degree the fraction of mercury that is bound to particulate matter. The SOH sampling system employs a series of heated, solid-phase adsorbent traps to speciate mercury. In principle, flue gas Hg²⁺ is adsorbed by a KCl-impregnated quartz wool sorbent trap. Hg⁰ is collected by an iodated carbon sorbent after passing through the KCl/quartz sorbent.

In addition, the SOH method that was applied at the EERC collected samples using isokinetic principles. A miniparticulate filter consisting of a small quartz-fiber filter disk inserted into a quartz tube on a pure nickel support screen was used to collect fly ash to measure particulate-bound mercury (6). The analysis of the mercury on the fly ash was done using thermal desorption at 800°C (1472°F), passing through a heated MnO converter, gold preconcentration and, finally, measured using CVAFS detection.

3.3.2 Ontario Hydro Mercury Speciation Method

Speciated mercury analyses will be performed using the OH mercury speciation method, which is the method selected by EPA for its information collection request (ICR). A schematic of the sample train is shown in Figure 4. The development, testing, and verification of this method were done on the EERC PTC. The OH method is a modification of EPA Method 29 in which three aqueous 1 N KCl impinger solutions are substituted for one of the HNO₃-H₂O₂ solutions. The OH sampling method provides for the measurement of particulate-bound mercury and Hg⁰ and Hg²⁺ concentration in the flue gas. The impinger solutions are analyzed for mercury by cold-vapor atomic absorption (CVAA) techniques. The analyses of the OH impinger solutions, reagent, field blanks, and field spikes will be done on-site. In this way, results are known within 24 hours, and changes to the test plan can be made while the sampling team is on-site. In addition, if problems do occur, corrective action can be taken and sampling redone, if necessary.

The OH method has been extensively tested at the EERC and has been shown to provide the best mercury speciation data for coal-fired boilers (7, 8). The method is currently being evaluated by American Society for Testing and Materials (ASTM) Subcommittee D22.03.01. A detailed

Figure 4. Schematic of the OH mercury speciation method.

description of the OH method in the ASTM format is available on the EPA Web site at <http://www.epa.gov/ttn/emc> under “Preliminary Methods.”

3.3.3 Tekran Mercury CEM

The mercury CEM used for these tests was the Tekran Model 2537A. The analyzer is based on the principle of atomic fluorescence (AF), which provides an inherently more sensitive signal than atomic absorption. The system uses a gold-impregnated silica support for preconcentrating the mercury and separating it from potential interferences that degrade sensitivity. The total time for the entire process is about 2.5 min. The analyzer is automated and has been used successfully for a number of years to measure ambient mercury. In addition, during the last 3 years, it has been used to measure mercury in flue gas streams from combustion systems. The system is calibrated using Hg^0 as the primary standard. The Hg^0 is contained in a closed vial, which is held in a thermostatic bath. The temperature of the mercury is monitored, and the amount of mercury is calculated using vapor pressure calculations. Typically, the calibration of the unit has proven stable over a 24-hr period. The Tekran CEM has a wide detection limit, from 0.1 ng/Nm³ to 50 µg/Nm³.

The instrument was originally designed to reliably measure ambient levels of vapor phase Hg^0 . A front-end sampler for the vapor-phase CEM instrument that differentiates between Hg^0 and Hg^{2+} has recently been developed. This was accomplished through the use of a programmable, temperature-regulated annular denuder coated with KCl that quantitatively removes vapor-phase Hg^{2+} from the air stream and does not collect Hg^0 . The Hg^0 is collected downstream and analyzed at 5-minute intervals in the conventional manner with the Tekran Model 2537A instrument. After a set period of time, zero air is passed through the denuder as it is rapidly heated. The accumulated Hg^{2+} is reduced to Hg^0 and moves to the analyzer where it is quantified. In this configuration, the Tekran instrument provides average concentrations of both Hg^0 and Hg^{2+} . The active length of the annular denuder is 23 cm. The denuder is constructed of quartz, with the active collection area modified to better support the KCl coating. Experiments thus far indicate the denuder needs to be recoated approximately once a month.

3.4 Mercury Spiking Systems

The tests required that known amounts of Hg^0 and Hg^{2+} be spiked into the flue gas stream. This was done using mercury-spiking systems previously developed at the EERC. These systems have been shown to accurately spike either Hg^0 or HgCl_2 into the PTC (7).

The spiking system for Hg^{2+} used mercury(II) chloride (HgCl_2) and is shown in Figure 5. As can be seen in Figure 5, the HgCl_2 is contained in three vessels on a fritted platform which allows N_2 to pass through. A mass flow controller is set at 35.6 cm³/min, which controls the amount of N_2 sweeping through the diffusion vessels. The dilution N_2 is preheated externally to 350°F (177°C) and kept at a constant flow rate of 20 scfh by using a flowmeter. The temperature of the oven is kept at 220°F (104°C).

Figure 5. Schematic of the HgCl_2 spiking system.

The Hg^0 spiking system is shown in Figure 6. The Hg^0 is contained in a single diffusion vessel with three levels, allowing the N_2 to pass through. A mass flow controller is set at $34.0 \text{ cm}^3/\text{min}$, controlling the amount of N_2 sweeping the vessel. A dilution flow of N_2 is controlled by a flowmeter set at 20 scfh and preheated in the oven before it is mixed. The oven temperature is set at 84°C (184°F), and the injection arm is set at 160°C (320°F). Calibration curves for both systems are shown in Figures 7 and 8, and as can be seen in Figures 7 and 8, the mercury concentration exiting the spiking systems is linear with the amount of sweep gas.

4.0 APPROACH/WORK PLAN

The overall test matrix is shown in Table 2. As can be seen in the table, the primary variables for the tests were the particulate collection device (the level of particulate matter in the SPDC), mercury spiking, the use of light source, and the use of simulated rain. In addition, a test was completed to determine if ozone has an effect on the mercury speciation in the SPDC (Test 10). The coal chosen for the tests was a Blacksville eastern bituminous coal. A typical analysis of the coal is shown in Table 3. This coal was chosen for the project because of the extensive mercury research that has been done by the EERC, Consol, and others utilizing this coal. In addition, the EERC had enough of this coal on-site for the proposed tests; therefore, it was not necessary to purchase the coal.

The first two tests were conducted firing natural gas. The purpose of these tests was primarily shakedown. The shakedown was to ensure everything was working as intended both from the standpoint of the SPDC operation and the mercury-spiking systems.

Figure 6. Schematic of the Hg^0 spiking system.

Figure 7. Calibration curve for the HgCl_2 spiking system.

Figure 8. Calibration for Hg⁰ spiking system.

Following the two shakedown tests firing natural gas, Tests 3 through 10 (using the tubular ESP) were to determine if Hg²⁺ adheres to the particulate matter, reduced by light, or removed by simulated rain. It was expected that the ESP would remove about 95% of the particulate matter. The remaining tests (Tests 11 through 16) were conducted using a baghouse. The baghouse was expected to remove >99.5%. The baghouse tests were conducted to determine what the fate of the Hg²⁺ was in the SPDC when very little particulate matter is present. The baghouse test was conducted at the same conditions used for the ESP test.

5.0 OPERATIONAL PROCESS DATA

5.1 Particulate Test Combustor, Baghouse, and ESP

In general, the PTC operated well during the test. As shown in Table 4, the O₂ and SO₂ levels were relatively constant throughout the entire test program. However, the loss on ignition (LOI) was somewhat higher than in previous tests firing Blacksville coal. The LOI measurements were 13.64% for a sample collected from the ESP hopper and 9.08% for an ash sample collected in the baghouse hopper later in the test program. Typically, tests with Blacksville coal in the PTC resulted in LOI levels of about 3%–5% (9). The same two fly ash samples used to determine the percent LOI were also analyzed for mercury. The results showed that the concentration of mercury from the ESP

Table 2. Test Matrix ^{a,b,c}

Test No.	Fuel	Particulate Control Device	Hg Spike ^d	Light	Simulated Rain	Ozone, ppb
1	Natural gas	Baghouse	Hg ⁰	No	No	0
2	Natural gas	Baghouse	HgCl ₂	No	No	0
3	Blacksville	ESP	None	No	No	0
4	Blacksville	ESP	None	Yes	Yes	0
5	Blacksville	ESP	None	Yes	No	0
6	Blacksville	ESP	HgCl ₂ + Hg ⁰	No	No	0
7	Blacksville	ESP	HgCl ₂ + Hg ⁰	Yes	Yes	0
8	Blacksville	ESP	HgCl ₂ + Hg ⁰	No	Yes	0
9	Blacksville	ESP	HgCl ₂ + Hg ⁰	Yes	No	0
10	Blacksville	ESP	HgCl ₂ + Hg ⁰	Yes	No	200
11	Blacksville	Baghouse	None	No	No	0
12	Blacksville	Baghouse	None	Yes	Yes	0
13	Blacksville	Baghouse	HgCl ₂ + Hg ⁰	No	No	0
14	Blacksville	Baghouse	HgCl ₂ + Hg ⁰	Yes	Yes	0
15	Blacksville	Baghouse	HgCl ₂ + Hg ⁰	No	Yes	0
16	Blacksville	Baghouse	HgCl ₂ + Hg ⁰	Yes	No	0

^a The dilution ratio for the chamber was between 140:1 and 200:1 for all runs.

^b Hg⁰ was monitored continuously (every 3–5 minutes).

^c Hg²⁺ and particulate-bound mercury was sampled at 6, 30, 60, and 120 minutes for each test.

^d The HgCl₂ and Hg⁰ spikes were nominally 12 or 15 µg/dm³.

Table 3. Analysis of the Blacksville Coal

Mercury, ppm (dry)	0.0916
Chlorine, ppm (dry)	758
Proximate Analysis	
Moisture, %	1.20
Volatile Matter, %	37.80
Fixed Carbon, %	53.30
Ash, %	7.71
Ultimate Analysis	
Hydrogen, %	4.62
Carbon, %	74.70
Nitrogen, %	2.09
Sulfur, %	2.03
Oxygen, %	7.65
Heating Value, Btu/lb	13,541

Table 4. PTC Operational Data^{1,2}

Run No.	Date	Temp., ³ °F	SO ₂ ppm	CO ppm	CO ₂ %	O ₂ %	NO _x ppm
1 & 2 ⁴	3/7/2000	305	21	1.9	9.2	4.4	165
			23	0.1	1.0	1.2	6
3	3/8/2000	309	1216	6.5	14.0	5.1	603
			42	0.6	0.5	0.7	61
4	3/8/2000	310	1312	3.8	14.4	4.4	643
			19	0.2	0.2	0.3	18
5	3/10/2000	307	1386	3.9	13.9	1.4	633
			48	0.1	0.2	0.2	15
6	3/13/2000	310	1279	3.2	13.5	4.7	642
			43	0.8	0.7	0.5	21
7	3/13/2000	310	1205	6.6	13.1	5.4	678
			42	1.4	0.4	0.5	12
8	3/13/2000	305	1284	4.9	13.8	5.0	719
			46	0.8	0.6	0.6	27
9	3/14/2000	300	1240	2.6	13.7	5.4	690
			46	11.1	0.6	0.5	20
10	3/14/2000	305	1221	3.8	13.8	5.1	671
			65	1.1	0.7	1.0	33
11	3/15/2000	305	1251	2.0	13.4	5.8	664
			36	0.2	0.4	0.4	38
12	3/15/2000	301	1334	5.3	13.7	4.9	660
			43	0.5	0.4	0.5	26
13	3/16/2000	303	1329	1.6	13.8	5.1	646
			42	0.4	0.5	0.6	16
14	3/16/2000	302	1324	4.5	13.5	4.6	637
			37	0.2	0.4	0.4	16
15	3/16/2000	302	1290	3.3	14.6	4.9	670
			54	0.3	0.6	0.6	34
16	3/17/2000	300	1295	2.3	13.9	5.5	633
			47	0.7	0.6	0.5	18

¹ Each run is the average followed by the standard deviation.

² Data taken at the outlet of the particulate control device.

³ Temperature at the sampling location at the outlet of the ESP/baghouse. Sampling location was approximately 10 ft downstream of the control device.

⁴ Tests on natural gas (all others on coal).

and baghouse hoppers were 0.252 and 0.606 µg/g, respectively. This is exactly the same concentration in the ESP hopper ash that was obtained in a project using the PTC firing Blacksville coal when the LOI was only 3.22% (9), indicating that the higher LOI did not have much of an effect on the overall mercury results.

As indicated earlier, the ESP used for these tests was a single-wire pilot unit that was not very efficient. This was particularly true for Tests 9 and 10, where the ESP was severely sparking and the power supply was tripping out. An attempt was made to maintain the power supply at 50 kV; however, because of sparking problems, it was generally operated from 30 to 40 kV. For the baghouse tests, the pressure drop across was easily controlled. By using an on-line medium pressure-pulsing system, the pressure drop was controlled between 4 and 8 in. H₂O with a cleaning cycle of about 2 hours.

Using an average inlet dust loading of 1.404 grains/scf, Table 5 shows the particulate collection efficiency for all the tests both with the ESP and baghouse. It is clear from Table 5 that the particulate collection efficiency was much better when using the baghouse.

Table 5. Particulate Collection Efficiencies for the Tests

Test No.*	Type of Particulate Collector	Outlet Dust Loading, gr/scf	Particulate Collection Efficiency, %
3	ESP	0.3679	73.80
4	ESP	0.0737	94.75
5	ESP	0.0685	95.13
6	ESP	0.0590	95.80
7	ESP	0.0682	95.14
8	ESP	0.0921	93.44
9	ESP	0.9012	35.82
10	ESP	0.6633	52.77
11	Baghouse	0.0044	99.69
12	Baghouse	0.0043	99.70
13	Baghouse	0.0040	99.72
14	Baghouse	0.0105	99.25
15	Baghouse	0.0060	99.57
16	Baghouse	0.0114	99.19

* Tests 1 and 2 were with natural gas.

5.2 SPDC Operation

Once the system was set up and shakedown tests were completed to resolve some minor issues (primarily associated with applying a partial vacuum to the SPDC), the SPDC system worked as expected. It also appeared that the Tekran mercury CEM in conjunction with the KCl denuders worked well in providing speciated mercury results. Using this system eliminated the need for some

of the more difficult sampling methods, as used by Frontier Geosciences in the past, such as mist chambers and ion-exchange resins. The operational data for the SPDC for each of the tests are shown in Table 6.

6.0 RESULTS AND DISCUSSION

6.1 Mercury Balance

To ensure that the results are valid, reasonable mercury balances must be obtained around the SPDC. For purposes of these tests, mercury balances $\pm 25\%$ were considered reasonable. The components of the mercury balance into the SPDC are listed below:

- Mercury in the injected flue gas
- Mercury in the dilution air (filtered ambient air)
- Mercury in the sampling makeup air (filtered ambient air)

Table 6. SPDC Operational Conditions

Run No.	SPDC Pressure, psi	Inlet Gas Temperature, °F	Sample Injection Time, min	Gas Flow Rate,* L/min	Dilution Ratio
1	-5.0	300	1	2.79	179.2
2	-5.0	300	1	2.79	179.2
3	-5.0	300	2	2.80	89.3
4	-4.8	300	2	2.79	89.6
5	-5.0	300	2	2.80	89.3
6	-5.0	300	1	2.80	178.6
7	-4.7	300	1	2.79	179.2
8	-5.0	300	1	2.80	178.6
9	-4.5	300	1.45	2.80	123.2
10	-2.3	300	1	2.81	177.9
11	-5.0	300	2	2.80	89.3
12	-4.3	300	2	2.79	89.6
13	-4.9	300	1	2.80	178.6
14	-4.7	300	1	2.80	178.6
15	-5.0	300	1	2.79	179.2
16	-5.0	300	1	2.80	178.6

* The gas flow rate is defined at 21 °C (70 °F).

The mercury going into the SPDC must balance with the following components:

- Mercury leaving the system as part of the sampling activities
- Mercury remaining in the chamber at the end of the test
- Mercury in the rinses

The total mercury balance results are shown in Tables 7 and 8. The mercury concentration in the ambient air was measured to be 3 ng/m³. As can be seen, the balances were quite good as the results with just several exceptions were within $\pm 25\%$, indicating valid results.

6.2 Flue Gas Spiking

The spiking systems were tested in the first two natural gas tests. In the first test, a nominal 10 $\mu\text{g}/\text{Nm}^3$ Hg^0 was spiked into the flue gas at the outlet of the ESP. The results showed that 98% was measured as Hg^0 . In Test 2, 10 $\mu\text{g}/\text{Nm}^3$ HgCl_2 was spiked into the system's flue gas at the outlet of the ESP. The results for this test showed about 80% was measured as Hg^{2+} and 20% as Hg^0 , indicating some reduction occurred. For the coal tests, nominally 12 $\mu\text{g}/\text{Nm}^3$ of each species was spiked into the flue gas. Averaging the baseline mercury measurements (Tests 4, 5, 11, and 12) gives a baseline total mercury concentration of 9.35 ± 1.47 . Using this value, the spiking results (Tests 6–9 and Tests 10–16) are shown in Table 9. From these data, it appears that for Tests 13 to 15, the mercury spiking was closer to 15 $\mu\text{g}/\text{Nm}^3$ each.

Table 7. Total Mercury Balance Data

SPDC Run No.	Hg In			Hg Out		
	Hg-Injected Flue Gas ng	Hg in Dilution Air ng	Hg in Sampling Makeup Air ng	Hg in SPDC End of Test ng	Hg Removed by Sampling ng	Hg in Rinses ng
1	28.7	0.94	0.17	37.79	4.08	3.29
2	29.1	0.94	0.14	11.67	1.04	12.44
3	56.3	0.94	0.10	10.71	0.69	25.44
4	48.0	0.93	0.16	11.08	1.08	29.58
5	42.8	0.93	0.20	9.82	1.33	34.64
6	96.2	0.94	0.18	25.31	3.29	56.00
7	100.0	0.94	0.18	27.64	4.02	48.59
8	99.2	0.94	0.18	27.37	3.21	43.47
9	130.9	0.94	0.18	14.70	2.13	91.03
10	90.6	0.94	0.18	22.83	3.00	64.94
11	59.9	0.93	0.18	0.79	3.00	43.74
12	58.3	0.93	0.18	5.95	0.66	36.41
13	112.9	0.94	0.18	31.32	3.71	69.29
14	112.8	0.94	0.18	34.42	3.97	41.58
15	108.7	0.94	0.18	35.94	4.17	46.37
16	130.5	0.94	0.18	24.08	2.84	88.14

Table 8. Total Mercury Balance

SPDC Run No.	Total Hg in to SPDC, ng	Total Hg out of SPDC, ng	Mercury Balance (out/in) %
1	29.82	47.70	160.0
2	30.16	27.70	91.8
3	57.37	39.39	68.7
4	49.08	46.18	94.1
5	43.97	48.34	109.9
6	97.34	87.15	89.5
7	101.12	84.70	83.8
8	100.33	78.50	78.2
9	132.05	110.40	83.6
10	91.74	93.31	101.7
11	61.04	47.37	77.6
12	59.37	47.46	79.9
13	114.02	106.87	93.7
14	113.88	84.41	74.1
15	109.85	90.92	82.8
16	131.66	117.60	89.3

Table 9. Mercury Spiking Results

SPDC Run No.	Total Hg, $\mu\text{g}/\text{Nm}^3$	Baseline Hg $\mu\text{g}/\text{Nm}^3$	Amount Hg Spiked $\mu\text{g}/\text{Nm}^3$
6	34.37	9.35	25.02
7	35.84	9.35	26.49
8	35.43	9.35	26.08
9	32.25	9.35	22.90
10	32.26	9.35	22.91
13	40.32	9.35	30.97
14	40.27	9.35	30.92
15	38.97	9.35	28.62
16	46.62	9.35	37.27

6.3 Flue Gas Mercury Speciation

The mercury speciation results using the OH method are shown in Table 10. Based on the volume of flue gas sampled, the expected mass loading to the SPDC for each of the mercury species was then calculated. The flue gas results for the baseline tests (Tests 3 and 11) are typical for tests at the outlet of the particulate collection device firing Blacksville coal in the PTC (8). As would be

Table 10. Speciated Mercury Measured in the Flue Gas and Expected Loading to the SPDC

SPDC Run	Mercury Concentration in Flue Gas				Volume Flue Gas Injected to SPDC, L	Expected Mercury Loading to SPDC from Flue Gas			
	Total Hg, $\mu\text{g}/\text{Nm}^3$	Hg^0 , $\mu\text{g}/\text{Nm}^3$	Hg^{2+} , $\mu\text{g}/\text{Nm}^3$	Hg_p , $\mu\text{g}/\text{Nm}^3$		Total Hg, ng	Hg^0 , ng	Hg^{2+} , ng	Hg_p , ng
1	10.29	10.05	0.23	0.02	2.79	28.7	28.0	0.6	0.1
2	10.42	2.11	8.30	0.01	2.79	29.1	5.9	23.2	0.0
3	10.06	0.54	8.68	1.70	5.60	56.3	3.0	48.6	9.5
4	8.60	0.46	7.88	0.26	5.58	48.0	2.6	44.0	1.5
5	7.65	0.46	6.81	0.38	5.60	42.8	2.6	38.1	2.1
6	34.37	5.85	27.96	0.57	2.80	96.2	16.4	78.3	1.6
7	35.84	6.81	28.73	0.30	2.79	100.0	19.0	80.2	0.8
8	35.43	7.61	27.19	0.63	2.80	99.2	21.3	76.1	1.8
9	32.25	1.26	24.10	6.89	4.06	130.9	5.1	97.8	28.0
10	32.26	1.72	21.65	8.89	2.81	90.6	4.8	60.8	25.0
11	10.70	0.44	10.23	0.04	5.60	59.9	2.5	57.3	0.2
12	10.44	0.36	10.07	0.01	5.58	58.3	2.0	56.2	0.1
13	40.32	8.45	31.85	0.02	2.80	112.9	23.7	89.2	0.1
14	40.27	9.12	31.14	0.02	2.80	112.8	25.5	87.2	0.1
15	38.97	9.38	29.56	0.04	2.79	108.7	26.2	82.5	0.1
16	46.62	8.91	37.69	0.02	2.80	130.5	24.9	105.5	0.1

expected, because of the higher particulate collection efficiency, less particulate-bound mercury was in the outlet flue gas for the baghouse test compared to the ESP tests. This result was substantiated by the lower concentration of mercury in the ESP hopper ash compared to the baghouse, 0.252 and 0.606 $\mu\text{g}/\text{g}$, respectively. It should also be noted that although equal amounts of Hg^0 and Hg^{2+} were spiked into the flue gas, there was substantial oxidation of the Hg^0 to Hg^{2+} . This has also been observed in previous tests (8).

6.4 SPDC Results

The SPDC mercury speciation results for each of the 14 tests (excluding the two natural gas tests) are shown in Figures 9 through 22. These figures show the vapor-phase mercury speciation as a function of time within the SPDC chamber. It is assumed that at time zero, the total amount of Hg^{2+} and particulate-bound mercury is the amount expected (from Table 10) based on the flue gas mercury speciation measurements. For the calculations incorporated into the graphs in Figures 9 through 22, it is assumed that all of the mercury in the dilution and makeup air is Hg^0 . It can be seen, in all of the figures, that there is a very rapid decrease in both Hg^{2+} and particulate-bound mercury. It appears that the rate of decrease is so fast that it occurs even before the first real measurement can be made. The overall change in Hg^{2+} mass within the SPDC chamber for each of the tests is shown in Table 11. There are two possible explanations for the rapid decrease. First, the Hg^{2+} could have been reduced to Hg^0 , and secondly, the Hg^{2+} may have collected along the walls of the SPDC and been removed

Figure 9. The change in mercury speciation as a function of time for SPDC Test 3.

Figure 10. The change in mercury speciation as a function of time for SPDC Test 4.

Figure 11. The change in mercury speciation as a function of time for SPDC Test 5.

Figure 12. The change in mercury speciation as a function of time for SPDC Test 6.

Figure 13. The change in mercury speciation as a function of time for SPDC Test 7.

Figure 14. The change in mercury speciation as a function of time for SPDC Test 8.

Figure 15. The change in mercury speciation as a function of time for SPDC Test 9.

Figure 16. The change in mercury speciation as a function of time for SPDC Test 10.

Figure 17. The change in mercury speciation as a function of time for SPDC Test 11.

Figure 18. The change in mercury speciation as a function of time for SPDC Test 12.

Figure 19. The change in mercury speciation as a function of time for SPDC Test 13.

Figure 20. The change in mercury speciation as a function of time for SPDC Test 14.

Figure 21. The change in mercury speciation as a function of time for SPDC Test 15.

Figure 22. The change in mercury speciation as a function of time for SPDC Test 16.

Table 11. Overall Change in SPDC Vapor-Phase Oxidized Mercury

SPDC Run	Hg²⁺ in Flue Gas, ng	Hg²⁺ Gas Sampled, ng	Hg²⁺ in Chamber, ng	Hg²⁺ Total, ng	Hg²⁺ Mass Change, ng
3	48.61	0.08	0.25	0.33	47.28
4	43.97	0.02	0.28	0.30	43.67
5	38.14	0.09	0.72	0.81	37.33
6	78.29	0.20	0.71	0.91	77.38
7	80.16	0.08	0.20	0.28	79.89
8	76.13	0.04	0.22	0.25	75.88
9	97.85	0.09	0.35	0.43	97.42
10	60.84	0.25	0.58	0.83	60.01
11	57.29	0.09	0.34	0.43	56.86
12	56.19	0.02	0.00	0.02	56.17
13	89.18	0.11	0.52	0.63	88.55
14	87.19	0.02	0.12	0.14	87.05
15	82.47	0.02	0.00	0.02	82.45
16	105.53	0.13	0.68	0.81	104.72

by the rinses. If there was a reduction of Hg²⁺ to Hg⁰, then a corresponding increase in Hg⁰ should have been observed.

In fact, there was a corresponding increase in Hg⁰ for almost all of the tests, as shown in Table 12. However, the increase in the mass of Hg⁰ is substantially less than the decrease in mass of Hg²⁺. Although it appears there is some reduction of Hg²⁺ to Hg⁰, there are also clearly substantial wall effects within the SPDC chamber. As shown by the good mercury balances, the mercury that is attracted to the walls of the SPDC are removed in the rinses of the chamber conducted at the end of each test. It is interesting to note that the percentage of mercury in the rinses measured at the end of the test was relatively constant from test to test. The average was 67%, with a standard deviation of only 12%.

Unfortunately, because of the very rapid changes that occurred in the SPDC, it is not possible to do a statistical evaluation to determine if the variables (light, simulated rain, ozone) had an effect on mercury speciation. Simply looking at the figures and the data, it appears that simulated rain does result in a more thorough removal of the Hg²⁺ and particulate-bound mercury as they become zero almost immediately. The addition of ozone (Test 10) did show a very high level of particulate-bound mercury in the flue gas following the ESP. However, for this test, the ESP was not operating at peak efficiency, and the dust loading was much higher than in previous tests. The same situation existed for Test 9 without ozone, and that test also showed much higher particulate-bound mercury. Therefore, it is not possible to make any conclusions as to the effect of ozone. The measurements made around the SPDC are shown in Appendix A.

Table 12. Hg⁰ Mass Balance for SPDC Tests

SPDC Run No.	Hg ⁰ In				Hg ⁰ Out			Hg ⁰ Mass Change, ng
	Hg ⁰ Flue Gas, ng	Hg ⁰ Makeup Air, ng	Hg ⁰ Dilution Air, ng	Hg ⁰ Total, ng	Hg ⁰ gas Sampled, ng	Hg ⁰ Chamber, ng	Hg ⁰ Total, ng	
3	3.02	0.10	0.94	4.06	0.59	10.25	10.84	6.79
4	2.57	0.16	0.94	3.66	1.05	10.80	11.85	8.19
5	2.58	0.20	0.94	3.71	1.24	9.05	10.29	6.59
6	16.38	0.17	0.94	17.49	3.05	24.50	27.55	10.07
7	19.00	0.18	0.94	20.12	3.91	27.35	31.26	11.14
8	21.31	0.18	0.94	22.43	3.18	27.15	30.33	7.90
9	5.12	0.18	0.94	6.24	2.04	14.35	16.39	10.16
10	4.83	0.18	0.94	5.96	2.55	21.20	23.75	17.79
11	2.46	0.18	0.94	3.58	0.20	0.44	0.65	-2.93
12	2.01	0.18	0.94	3.13	0.57	4.86	5.43	2.30
13	23.66	0.18	0.94	24.78	3.60	30.80	34.40	9.62
14	25.54	0.18	0.94	26.66	3.95	34.25	38.20	11.54
15	26.17	0.18	0.94	27.29	4.14	35.85	39.99	12.70
16	24.95	0.18	0.94	26.07	2.71	23.40	26.11	0.04

6.5 Comparison of the SOH Method to the Ontario Hydro Method

As part of the SPDC testing, a comparison of the SOH method was made to the OH method sampling for speciated mercury. Paired sampling trains were run. The results of the sampling are shown in Table 13 along with the statistical comparison for each mercury species. For the two methods, the calculated t-statistic is less than the t-value for each of the measured mercury species. Therefore, the two methods are statistically similar not only for total mercury but Hg²⁺, Hg⁰, and particulate-bound mercury as well. The draft final report completed by Frontier Geosciences comparing the two methods is found in Appendix B.

7.0 QUALITY CONTROL

The EERC is committed to delivering consistent and high-quality research that meets our client's needs and expectations. In order to ensure that the goals of this project are realized, an organizationwide quality management system (QMS), authorized and supported by EERC managers, is in effect and governs all programs within the organization. The EERC established and formalized a QMS and quality control (QC) procedures in August 1988. The *Quality Manual* defines the requirements and the organizational responsibilities for each major element of the QMS and references the supporting documents needed to provide a comprehensive program.

Table 13. Statistical Comparison of the SOH Method to the OH Method

Run No.	Total Hg			Hg ⁰			Hg ²⁺			Particulate-Bound Hg		
	OH	SOH	Diff.	OH	SOH	Diff.	OH	SOH	Diff.	OH	SOH	Diff.
1	10.30	12.80	-2.50	10.10	12.80	-2.70	0.23	0.03	0.20	0.26	0.01	0.25
2	10.40	16.20	-5.80	2.10	2.10	0.00	6.40	14.10	-7.70	0.38	0.08	0.30
3	10.90	8.10	2.80	0.54	0.58	-0.04	8.70	7.50	1.20	0.57	0.60	-0.03
4	8.60	8.50	0.10	0.46	0.30	0.16	7.90	8.20	-0.30	0.30	0.10	0.20
5	7.70	8.30	-0.60	0.46	0.35	0.11	6.80	7.90	-1.10	0.63	0.91	-0.28
6	34.40	37.20	-2.80	5.90	4.10	1.80	28.00	32.50	-4.50	6.89	3.72	3.17
7	35.80	33.50	2.30	6.80	7.10	-0.30	28.70	26.30	2.40	8.89	8.14	0.75
8	35.40	32.90	2.50	7.60	5.30	2.30	27.20	26.70	0.50			
9	32.30	28.10	4.20	1.30	0.62	0.68	24.10	23.80	0.30			
10	32.30	24.60	7.70	1.70	0.81	0.89	21.70	15.60	6.10			
11	10.70	9.50	1.20	0.44	0.08	0.36	10.20	9.40	0.80			
12	10.40	9.00	1.40	0.36	0.14	0.22	10.10	8.80	1.30			
13	40.30	36.30	4.00	8.50	7.80	0.70	31.90	28.50	3.40			
14	40.30	38.10	2.20	9.10	9.80	-0.70	31.10	28.30	2.80			
15	39.00	39.50	-0.50	9.40	9.00	0.40	29.60	30.50	-0.90			
16	46.60	42.80	3.80	8.90	8.90	0.00	37.70	33.90	3.80			
	Mean Diff.		1.2500			0.2425			0.5188			0.6229
	Mean Std. Dev.		3.2490			1.0826			3.2486			1.1667
	Calc. t-Stat.		1.5390			1.4125			0.8958			0.6388
	t-Statistic (95% confidence)		1.7530			1.7530			1.7530			1.9430

Compliance with this manual and its supporting documents ensures that the EERC adequately fulfills governmental and private clients' requirements relating to quality and compliance with applicable regulations, codes, and protocols. This project is required to follow the *Quality Manual*, project-specific quality assurance (QA) procedures, and all revisions. The EERC QA manager implements and oversees all aspects of QA/QC for all research, development, and demonstration projects and will review the QA/QC components of this project. The project manager is responsible for ensuring that project-specific QA/QC protocols are followed.

The EERC maintains a wide range of laboratories and equipment for solid, liquid, and gaseous characterization of the physical, chemical, mineralogical, biological, hydrological, and geological properties of natural and synthetic materials and processes. Laboratory procedures and instrument calibrations follow nationally recognized or approved standards and methods put forth by EPA, ASTM, National Institute of Standards and Technology (NIST), and other agencies. Each laboratory manager is responsible for ensuring that the applicable QA/QC procedures in this project are implemented. It is expected that the strongest QA/QC evaluation of the SPDC tests will be based on

a mercury mass balance calculation. Sufficient QA/QC data will be generated to assess both accuracy and precision of the SPDC data. This includes duplicate samples of total gas- and particulate-phase mercury. The following QA/QC requirements are specific to this project.

7.1 QA/QC for the Ontario Hydro Mercury Speciation Method

All data sheets, volumetric flasks, and petri dishes used for sample recovery were marked with preprinted labels. All labels included identifying data, including date, time, run number, sample port location, and the name of the sampler.

Prior to being used for the sampling, all glassware was washed with hot, soapy water, then rinsed with deionized water three times, soaked in 10% V/V nitric acid for a minimum of 4 hours, rinsed an additional three times with deionized water, and dried. The glassware was then stored in closed containers until it was used.

As part of the QA/QC procedures during the pilot-scale tests, both field blanks and field spikes were completed. The results are shown in Tables 14 and 15. A field blank is to determine if there is any mercury contamination. If the field blank shows contamination above instrument background, steps need to be taken to eliminate or reduce the contamination to below background levels. Field spikes are completed to ensure that adequate levels of accuracy are maintained. These samples are made up independently of the chemist doing the analyses. The spikes are required to be within 15% of the true value. If the values are not within the specified limits, the instrument is recalibrated and the samples reanalyzed. For these tests, with the exception of one sample, and that one was very close, all field spikes gave recoveries of $100\% \pm 15\%$. A much more detailed discussion of QA/QC is presented in the details of the method found at the EPA web site, <http://www.epa.gov/ttn/emc/prelim.html>.

Table 14. Field Blank Results for OH Method

Blanks	KCl	H ₂ O ₂	KMnO ₄
1	<0.03	0.08	0.04
2	<0.03	0.09	<0.03
3	0.05	<0.03	

Table 15. Field Spike Results for the OH Method

Solution	Expected, μg/L	Measured, μg/L	Recovery, %
KMnO ₄	10	8.80	88.0
KMnO ₄	10	8.55	85.5
KMnO ₄	10	10.15	101.2
KCl	10	9.91	99.1
KCl	10	10.01	100.1
KCl	10	10.40	104.0
H ₂ O ₂	5.2	5.67	109.0
H ₂ O ₂	2	1.66	83.0
H ₂ O ₂	2	2.28	114.0
H ₂ O ₂	5	4.40	88.0
H ₂ O ₂	10	9.95	99.5
H ₂ O ₂	4.8	4.39	91.5

7.2 Quality Control for the SOH Sampling

The following section summarizes QC measures undertaken to ensure that the analysis of the SOH method samples was completed under very good control. The results are summarized in the table which follows and are discussed in the text below. Included in the text is the "data quality objective" or DQO, which is essentially the quality objectives or control limits expected for the results of the SOH method to be considered good.

The results reported below include lab analysis replicates, system blanks, matrix spikes, and standard reference material (SRM) recoveries. QC data are extracted from various sample analysis data sheets. The file names and extensions are included in the table below.

Replicate Analysis. Replicate sample analysis provides the means to measure the precision of the CVAFS analytical method. In most cases, replicate analysis occurred on the same analysis day, using the same instrument. However, in many instances, replicate sample analysis occurred on different days and in some cases with different instruments. For samples analyzed in duplicate ($n = 2$), a relative percent difference (RPD) was calculated as a percentage using the formula:

$$\text{RPD} = \text{absolute value of } [A - B] / \text{average } (A + B)$$

If a sample was analyzed in replicate where $n > 2m$, then a relative standard deviation (RSD) is calculated as a percentage using the following formula where s = standard deviation and m = mean:

$$RSP = s/m$$

The laboratory replicate data are shown in Table 16 have an average RPD of 2.2%. Therefore, the projected DQO for lab analysis duplicates of $\pm 20\%$ was readily achieved.

Matrix Limits. The sample analysis matrix spike is a measure of laboratory analysis accuracy and is the addition of 1.00-ng standard spike to a bubbler which also includes a sample aliquot. The recovery of the 1.00-ng spike was calculated as a percent recovery using the formula:

$$\% \text{ Recovery} = \frac{(\text{ng/aliquot of the spike} + \text{ng/aliquot of the sample})}{(\text{ng/aliquot of the sample} + 1.00 \text{ ng})}$$

The results of the matrix spike were excellent ($\sigma = 5.7\%$) and are shown in Table 17. Therefore, the DQO for the sample analysis matrix spike of ± 2.5 was always met during this study.

Sample Blanks. The blanks for both the KCl denuders and the SOH traps were all less than 0.1 ng per trap, as shown in Table 18.

Table 16. SOH QC Replicate Results

Sample ID	Rep. 1	Rep. 2	Mean	RPD
ICA-SOH-7-0307 AD	389.2	395.0	392.1	1.5%
KCLA-SOH-10-0307 AD	378.9	384.6	381.7	1.5%
KCLA-2030800 AD	177.0	180.7	178.9	2.1%
IC-A-11030800 AD	47.8	48.4	48.1	1.4%
KCLB-7031300 AD	150.3	153.4	151.8	2.0%
PHG-2031400 AD	60.6	63.3	61.9	4.5%

Table 17. SOH Sample Analysis Matrix Spikes

Sample ID	Recovery, %
ICA-SOH-7-0307 AS + 1.0 ng	98.0
KCLA-SOH-10-0307 AS + 1.0 ng	92.8
KCLA-2030800 AS + 1.0 ng	106.4
IC-A-11030800 AS + 1.0 ng	107.7
KCLB-7031300 AS + 1.0 ng	105.0
PHG-2031400 AS + 1.0 ng	95.5
KCLA-5031500 AS + 1.0 ng	100.7

Table 18. SOH Blanks

Blank No.	KCl Denuder, ng/trap	SOH Trap, ng/trap
1	0.0086	0.0223
2	0.0020	0.0032
3	0.0019	0.0105
4	0.0015	0.0044
5	0.0010	0.0087
6	0.0025	0.0069
7	0.0032	0.0017
8	0.0068	0.0196
9	0.0017	0.0836
10	0.0041	0.0041
11	0.0014	0.0047
Mean	0.0032	0.0154
Std. Dev.	0.0024	0.0236

8.0 CONCLUSIONS

Based on the SPDC tests, the following conclusions can be made:

- Good mass balances were obtained for the tests, $100 \pm 25\%$.
- The Hg^{2+} and the particulate-bound Hg decrease rapidly to at or near zero.
- There was for almost all tests a rapid increase in Hg^0 ; however, not nearly to the degree that the Hg^{2+} and particulate bound-mercury decreased.
- The data appear to indicate that some reduction of Hg^{2+} to Hg^0 does take place in the SPDC (or plume?).
- The size of the SPDC (0.5 m^3) resulted in severe wall effects; a high percentage of the mercury goes to the wall to be removed later by the rinses.
- Because of the very rapid decrease in Hg^{2+} and the particulate-bound Hg, it was not possible to statistically evaluate the effects of light and rain on mercury chemistry in the SPDC.
- Because of the wall effects, a static SPDC is not a very useful tool in understanding mercury chemistry in combustion plumes. However, a dynamic system where flue gas is constantly flowing through the device and mixing with air may eliminate these effects.

Conclusion sampling methodology are as follows:

- The Tekran mercury CEM with KCl denuders appeared to work well in speciating and measuring low levels of mercury.
- The SOH method statistically gave the same results as the Ontario Hydro mercury speciation method.

9.0 REFERENCES

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APPENDIX A

SPDC DATA

Run # 000307-1 with Nat'l Gas, Baghouse, and Hg(0) Spiking

Date	Time	Vol l	Hg ⁰ ng/m ³	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/7/2000	14:35	3.96	98.422	0.0	n/a	n/a				
3/7/2000	14:39	3.99	79.839	4.0	3.98	80.2	0.55	0.57	<0.009	0
3/7/2000	14:43	4.00	78.996	8.0	3.95	79.9	0.55	0.57	<0.009	0
3/7/2000	14:47	4.00	79.192	12.0	3.92	80.8	0.55	0.57	<0.009	0
3/7/2000	14:51	4.00	77.626	16.0	3.89	79.8	0.55	0.57	<0.009	0
3/7/2000	14:55	4.00	76.805	20.0	3.86	79.6	0.55	0.57	<0.009	0
3/7/2000	14:59	4.00	75.104	24.0	3.83	78.5	0.55	0.57	<0.009	0
3/7/2000	15:03	4.00	74.637	28.0	3.80	78.6	0.55	0.57	<0.009	0
3/7/2000	15:07	4.00	72.803	32.0	3.77	77.3	0.55	0.57	<0.009	0
3/7/2000	15:11	4.00	71.815	36.0	3.74	76.9	0.55	0.57	<0.009	0
3/7/2000	15:15	4.00	69.745	40.0	3.71	75.3	0.07	0.08	<0.009	0
3/7/2000	15:19	3.99	69.763	44.0	3.68	75.9	0.07	0.08	<0.009	0
3/7/2000	15:23	4.00	68.357	48.0	3.65	74.9	0.07	0.08	<0.009	0
3/7/2000	15:27	3.99	68.287	52.0	3.62	75.5	0.07	0.08	<0.009	0

Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3
	Hg total	PHg	Hg0	Hg2+	Hg total	PHg	Hg0	Hg2+
OH	57	0.1	56	1.3	65	0.1	64	0.7
SOH	71.7	NA	71.5	0.14				

Run # 000307-2 with Nat'l Gas, Baghouse, and HgCl2 Spiking

Date	Time	Vol l	Hg ⁰ ng/m ³	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/7/2000	19:36	3.78	19.4	0.0	n/a	n/a				
3/7/2000	19:40	4.00	20.6	4.0	3.984	20.7	3.68	3.72	<0.009	0
3/7/2000	19:44	4.00	21.3	8.0	3.952	21.6	3.68	3.72	<0.009	0
3/7/2000	19:48	4.00	20.8	12.0	3.921	21.2	3.68	3.72	<0.009	0
3/7/2000	19:52	3.64	21.0	16.0	3.890	21.6	1.20	1.25	<0.009	0
3/7/2000	19:56	4.00	21.0	20.0	3.859	21.8	1.20	1.25	<0.009	0
3/7/2000	20:00	4.00	20.9	24.0	3.828	21.9	1.20	1.25	<0.009	0
3/7/2000	20:04		20.9	28.0	3.797	21.9	0.50	0.54	0.83	0.89
3/7/2000	20:08		20.9	32.0	3.767	21.9	0.50	0.54	0.83	0.89
3/7/2000	20:12		20.9	36.0	3.737	21.9	0.50	0.54	0.83	0.89
3/7/2000	20:16		20.9	40.0	3.707	21.9	0.50	0.54	0.83	0.89
3/7/2000	20:20		20.9	44.0	3.678	21.9	0.50	0.54	0.83	0.89

Tekran 079 sample Hg(0) during this period - values not acceptable
Assumption here is that Hg(0) stayed constant

Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	58	0	12	47	74	0	12	63
SOH	91	NA	12	79				

Run # 000308-3 with Bit. Coal, ESP, and no Spiking

Date	Time	Vol l	[Hg ⁰] ng/m3	Dilution Time	Dilution Corr. Factor	Corrected Hg(0) ng/m ³	Corrected Hg(2+) ng/m ³	Corrected Hg(2+) ng/m ³
3/8/2000	12:32	3.87	26.8	0.0	n/a	n/a		
3/8/2000	12:36	4.00	22.4	4.0	3.984	22.5	5.97	6.04
3/8/2000	12:40	4.00	21.1	8.0	3.952	21.4	5.97	6.04
3/8/2000	12:44	4.00	20.8	12.0	3.921	21.2	5.97	6.04
3/8/2000	12:48	4.00	19.7	16.0	3.890	20.2	0.48	0.50
3/8/2000	12:52	4.00	20.5	20.0	3.859	21.3	0.48	0.50
3/8/2000	12:56	4.00	19.9	24.0	3.828	20.8	0.48	0.50
3/8/2000	13:00	3.99	19.5	28.0	3.797	20.5	0.48	0.50

Hg ²⁺	ng/m ³	PHg	ng/m ³
RGM-0308-3A T78	8.01	P-0308-3A	1.057
RGM-0308-3A dup T79	3.94	P-0308-3A-dup	0.926
M-0308-3B T78	0.48	P-0308-3B	0.404

Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	122	19	6.0	97	116	19	6.3	91
SOH	90	NA	6.5	84				

SPDC RUN 000308-4 with Bit. Coal, ESP, no Spikeing, with rainwater and light

Date	Time	Vol l	Hg ⁰ ng/m ³	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³
19:26	3.99	18.7	0.0	n/a	18.7	18.7	<0.007	0
19:30	4.00	19.3	4.0	3.984	19.4	19.4	<0.007	0
19:34	4.00	18.9	8.0	3.952	19.1	19.1	<0.007	0
19:38	4.00	19.1	12.0	3.921	19.5	19.5	<0.007	0
19:42	4.00	2.1	16.0	3.890	2.1	20.0	0.52	0.54
19:46	4.00	0.5	20.0	3.859	0.5	20.0	0.52	0.54
19:50	3.99	0.5	24.0	3.828	0.6	20.0	0.52	0.54
19:54	4.00	0.5	28.0	3.797	0.5	20.0	0.52	0.54
19:58	3.99	19.3	32.0	3.767	20.5	20.5	0.50	0.55
20:02	3.99	20.0	36.0	3.737	21.4	21.4	0.50	0.55
20:06	4.00	20.0	40.0	3.707	21.6	21.6	0.50	0.55
20:10	3.99	19.8	44.0	3.678	21.6	21.6	0.50	0.55
20:14	4.00	11.3	48.0	3.648	12.4	21.6	0.50	0.55

	Hg ²⁺ ng/m ³	PHg ng/m ³
RGM-0308-4A T78	-0.136	0.082
RGM-0308-4B T79	0.519	0.082
RGM-0308-4C T78	0.503	-0.101

Sampling Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m ³ Hg total	ng/m ³ PHg	ng/m ³ Hg0	ng/m ³ Hg2+	ng/m ³ Hg total	ng/m ³ PHg	ng/m ³ Hg0	ng/m ³ Hg2+
OH	96	2.9	5.1	88	95	1.5	4.2	90
SOH	95	0.1	3.3	92				

SPDC RUN 5 with light and ozone ESP no spiking

Date	Time	Vol l	Hg ⁰ ng/m ³	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³	Adjusted PHg ng/m ³
3/10/2000	17:25	2.50	24.5	0.0	n/a	24.5	24.5	2.58	2.64	<0.009	0
3/10/2000	17:27	2.50	23.5	2.5	2.494	23.56	23.6	2.58	2.64	<0.009	0
3/10/2000	17:30	2.50	19.714	5.0	2.481	19.86	19.9	2.58	2.64	<0.009	0
3/10/2000	17:32	2.50	10.136	7.5	2.469	10.26	19.6	2.58	2.64	<0.009	0
3/10/2000	17:35	2.50	5.855	10.0	2.457	5.96	19.6	2.58	2.64	<0.009	0
3/10/2000	17:37	2.50	3.562	12.5	2.444	3.64	19.6	2.58	2.64	<0.009	0
3/10/2000	17:40	2.50	1.986	15.0	2.432	2.04	19.6	2.58	2.64	<0.009	0
3/10/2000	17:42	2.50	1.428	17.5	2.420	1.48	19.6	2.58	2.64	<0.009	0
3/10/2000	17:45	2.50	18.694	20.0	2.408	19.41	19.4	0.20	0.21	0.119	0.127
3/10/2000	17:47	2.50	3.615	22.5	2.396	3.77	18.7	0.20	0.21	0.119	0.127
3/10/2000	17:50	2.50	1.031	25.0	2.384	1.08	18.7	0.20	0.21	0.119	0.127
3/10/2000	17:52	2.50	0.999	27.5	2.372	1.05	18.7	0.20	0.21	0.119	0.127
3/10/2000	17:55	2.50	0.356	30.0	2.360	0.38	18.7	0.20	0.21	0.119	0.127
3/10/2000	17:57	2.50	0.425	32.5	2.349	0.45	18.7	0.20	0.21	0.119	0.127
3/10/2000	18:00	2.50	0.31	35.0	2.337	0.33	18.7	0.20	0.21	0.119	0.127
3/10/2000	18:02	2.50	0.757	37.5	2.325	0.81	18.7	0.20	0.21	0.119	0.127
3/10/2000	18:05	2.50	16.714	40.0	2.314	18.06	18.1	1.31	1.44	0.083	0.091
3/10/2000	18:07	2.50	1.964	42.5	2.302	2.13	18.1	1.31	1.44	0.083	0.091
3/10/2000	18:10	2.50	0.548	45.0	2.291	0.60	18.1	1.31	1.44	0.083	0.091
3/10/2000	18:12	2.50	0	47.5	2.279	0.00	18.1	1.31	1.44	0.083	0.091
3/10/2000	18:15	2.50	0	50.0	2.268	0.00	18.1	1.31	1.44	0.083	0.091
3/10/2000	18:17	2.50	0	52.5	2.256	0.00	18.1	1.31	1.44	0.083	0.091
3/10/2000	18:20	2.50	0	55.0	2.245	0.00	18.1	1.31	1.44	0.083	0.091
3/10/2000	18:22	2.50	0	57.5	2.234	0.00	18.1	1.31	1.44	0.083	0.091

	Hg ²⁺ ng/m ³		PHg ng/m ³
RGM-0310-5A T78	2.584	P-0310-5A	-0.0349
RGM-0310-5B T78	0.202	P-0310-5B	0.1194
RGM-0310-5C T78	1.305	P-0310-5C	0.0826

SPDC RUN 6 with ESP, no light, no rain, with fluegas spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m3	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Corrected Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/13/2000	10:22	2.50	87.7	0.0	n/a	87.7	8.19	8.31	1.53	1.56
3/13/2000	10:25	2.50	84.5	2.5	2.494	84.7	8.19	8.31	1.53	1.56
3/13/2000	10:27	2.50	84.9	5.0	2.481	85.5	8.19	8.31	1.53	1.56
3/13/2000	10:30	2.50	82.8	7.5	2.469	83.9	8.19	8.31	1.53	1.56
3/13/2000	10:32	2.50	84.8	10.0	2.457	86.3	8.19	8.31	1.53	1.56
3/13/2000	10:35	2.50	92.5	12.5	2.444	94.7	8.19	8.31	1.53	1.56
3/13/2000	10:37	2.50	54.8	15.0	2.432	56.3	1.38	1.45	0.11	0.11
3/13/2000	10:40	2.50	50.4	17.5	2.420	52.1	1.38	1.45	0.11	0.11
3/13/2000	10:42	2.50	49.8	20.0	2.408	51.7	1.38	1.45	0.11	0.11
3/13/2000	10:45	2.50	41.3	22.5	2.396	43.1	1.38	1.45	0.11	0.11
3/13/2000	10:47	2.50	39.5	25.0	2.384	41.4	1.38	1.45	0.11	0.11
3/13/2000	10:50	2.50	39.4	27.5	2.372	41.5	1.38	1.45	0.11	0.11
3/13/2000	10:52	2.50	39.0	30.0	2.360	41.4	1.38	1.45	0.11	0.11
3/13/2000	10:55	2.50	38.0	32.5	2.349	40.5	1.38	1.45	0.11	0.11
3/13/2000	10:57	2.50	38.2	35.0	2.337	40.9	1.30	1.42	0.18	0.20
3/13/2000	11:00	2.50	38.1	37.5	2.325	41.0	1.30	1.42	0.18	0.20
3/13/2000	11:02	2.50	38.7	40.0	2.314	41.8	1.30	1.42	0.18	0.20
3/13/2000	11:05	2.50	38.1	42.5	2.302	41.4	1.30	1.42	0.18	0.20
3/13/2000	11:07	2.50	38.3	45.0	2.291	41.8	1.30	1.42	0.18	0.20
3/13/2000	11:10	2.50	37.6	47.5	2.279	41.3	1.30	1.42	0.18	0.20
3/13/2000	11:12	2.50	37.9	50.0	2.268	41.8	1.30	1.42	0.18	0.20
3/13/2000	11:15	2.50	36.9	52.5	2.256	40.9	1.30	1.42	0.18	0.20

	Hg ²⁺ ng/m ³	PHg ng/m ³
RGM-0313-6A	8.19	P-0313-6A 1.53
RGM-0313-6B	1.38	P-0313-6B 0.11
RGM-0313-6C	1.30	P-0313-6C 0.18

	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
Sampling Method	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	192	3.2	33	157	200	3.3	28	169
SOH	208	3.4	23	182				

SPDC RUN 7 with ESP, with light, with rain, with fluegas spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m3	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/13/2000	16:12	2.50	184.7	0.0	n/a	184.7	3.18	3.24	1.62	1.65
3/13/2000	16:15	2.50	64.3	2.5	2.494	64.5	3.18	3.24	1.62	1.65
3/13/2000	16:17	2.50	62.9	5.0	2.481	63.4	3.18	3.24	1.62	1.65
3/13/2000	16:20	2.50	63.3	7.5	2.469	64.1	3.18	3.24	1.62	1.65
3/13/2000	16:22	2.50	62.8	10.0	2.457	63.9	3.18	3.24	1.62	1.65
3/13/2000	16:25	2.50	68.8	12.5	2.444	70.4	3.18	3.24	1.62	1.65
3/13/2000	16:27	2.50	83.1	15.0	2.432	85.4	3.18	3.24	1.62	1.65
3/13/2000	16:30	2.50	82.0	17.5	2.420	84.7	3.18	3.24	1.62	1.65
3/13/2000	16:32	2.50	53.2	20.0	2.408	55.2	0.15	0.16	0.12	0.13
3/13/2000	16:35	2.50	52.7	22.5	2.396	55.0	0.15	0.16	0.12	0.13
3/13/2000	16:37	2.50	52.7	25.0	2.384	55.2	0.15	0.16	0.12	0.13
3/13/2000	16:40	2.50	52.2	27.5	2.372	55.0	0.15	0.16	0.12	0.13
3/13/2000	16:42	2.50	51.9	30.0	2.360	54.9	0.15	0.16	0.12	0.13
3/13/2000	16:45	2.50	51.9	32.5	2.349	55.2	0.15	0.16	0.12	0.13
3/13/2000	16:47	2.50	51.5	35.0	2.337	55.1	0.15	0.16	0.12	0.13
3/13/2000	16:50	2.50	51.3	37.5	2.325	55.1	0.15	0.16	0.12	0.13
3/13/2000	16:52	2.50	51.4	40.0	2.314	55.6	0.35	0.39	0.17	0.19
3/13/2000	16:55	2.50	51.1	42.5	2.302	55.5	0.35	0.39	0.17	0.19
3/13/2000	16:57	2.50	50.1	45.0	2.291	54.7	0.35	0.39	0.17	0.19
3/13/2000	17:00	2.50	50.1	47.5	2.279	54.9	0.35	0.39	0.17	0.19
3/13/2000	17:02	2.50	49.5	50.0	2.268	54.6	0.35	0.39	0.17	0.19
3/13/2000	17:05	2.50	49.2	52.5	2.256	54.6	0.35	0.39	0.17	0.19
3/13/2000	17:07	2.50	49.3	55.0	2.245	54.9	0.35	0.39	0.17	0.19
3/13/2000	17:10	2.50	48.9	57.5	2.234	54.7	0.35	0.39	0.17	0.19

	Hg ²⁺ ng/m ³	PHg ng/m ³
RGM-0313-7A	3.18	1.62
RGM-0313-7B	0.15	0.12
RGM-0313-7C	0.35	0.17

Sampling Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	200	1.7	38	160	194	1.1	39	153
SOH	187	0.6	40	147				

SPDC RUN 8 with ESP, no light, with rain, with fluegas spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m ³	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Corrected Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/13/2000	19:37	2.50	49.6	0.0	n/a	49.6	0.648	0.66	0.079	0.081
3/13/2000	19:40	2.50	50.5	2.5	2.494	50.6	0.648	0.66	0.079	0.081
3/13/2000	19:42	2.50	49.3	5.0	2.481	49.6	0.648	0.66	0.079	0.081
3/13/2000	19:45	2.50	49.7	7.5	2.469	50.3	0.648	0.66	0.079	0.081
3/13/2000	19:47	2.50	49.4	10.0	2.457	50.3	0.648	0.66	0.079	0.081
3/13/2000	19:50	2.50	49.7	12.5	2.444	50.8	0.648	0.66	0.079	0.081
3/13/2000	19:52	2.50	49.1	15.0	2.432	50.5	0.648	0.66	0.079	0.081
3/13/2000	19:55	2.50	49.8	17.5	2.420	51.4	0.648	0.66	0.079	0.081
3/13/2000	19:57	2.50	50.5	20.0	2.408	52.5	0.682	0.72	<0.009	0.000
3/13/2000	20:00	2.50	51.1	22.5	2.396	53.3	0.682	0.72	<0.009	0.000
3/13/2000	20:02	2.50	50.4	25.0	2.384	52.8	0.682	0.72	<0.009	0.000
3/13/2000	20:05	2.50	50.8	27.5	2.372	53.5	0.682	0.72	<0.009	0.000
3/13/2000	20:07	2.50	50.3	30.0	2.360	53.3	0.682	0.72	<0.009	0.000
3/13/2000	20:10	2.50	50.7	32.5	2.349	53.9	0.682	0.72	<0.009	0.000
3/13/2000	20:12	2.50	50.3	35.0	2.337	53.8	0.682	0.72	<0.009	0.000
3/13/2000	20:15	2.50	50.4	37.5	2.325	54.2	0.682	0.72	<0.009	0.000
3/13/2000	20:17	2.50	50.6	40.0	2.314	54.7	0.394	0.43	<0.009	0.000
3/13/2000	20:20	2.50	51.2	42.5	2.302	55.6	0.394	0.43	<0.009	0.000
3/13/2000	20:22	2.50	50.4	45.0	2.291	55.1	0.394	0.43	<0.009	0.000
3/13/2000	20:25	2.50	50.6	47.5	2.279	55.5	0.394	0.43	<0.009	0.000
3/13/2000	20:27	2.50	49.9	50.0	2.268	55.0	0.394	0.43	<0.009	0.000
3/13/2000	20:30	2.50	49.9	52.5	2.256	55.2	0.394	0.43	<0.009	0.000
3/13/2000	20:32	2.50	48.8	55.0	2.245	54.4	0.394	0.43	<0.009	0.000
3/13/2000	20:35	2.50	48.5	57.5	2.234	54.3	0.394	0.43	<0.009	0.000

Hg ²⁺	ng/m ³	PHg	ng/m ³
RGM-0313-8A	0.648	P-0313-8A	0.079
RGM-0313-8B	0.682	P-0313-8B	-0.082
RGM-0313-8C	0.394	P-0313-8C	-0.010

	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
Sampling Method	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	198	3.5	43	152	191	4.3	36	151
SOH	184	5.1	30	149				

SPDC RUN 9 with ESP, with light, no rain, with flue gas spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m3	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Corrected Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³	Adjusted PHg ng/m ³
3/14/2000	11:15	1.24	36.07	0.00	n/a	36.1	36.1	2.26	2.29	<0.009	0
3/14/2000	11:17	1.24	37.255	2.50	2.497	37.3	37.3	2.26	2.29	<0.009	0
3/14/2000	11:20	1.24	37.247	5.00	2.491	37.4	37.4	2.26	2.29	<0.009	0
3/14/2000	11:22	1.24	37.007	7.50	2.484	37.2	37.2	2.26	2.29	<0.009	0
3/14/2000	11:25	1.24	36.983	10.00	2.478	37.3	37.3	2.26	2.29	<0.009	0
3/14/2000	11:27	1.24	37.086	12.50	2.472	37.5	37.5	2.26	2.29	<0.009	0
3/14/2000	11:30	1.24	36.688	15.00	2.466	37.2	37.2	2.26	2.29	<0.009	0
3/14/2000	11:32	1.24	36.299	17.50	2.460	36.9	36.9	2.26	2.29	<0.009	0
3/14/2000	11:35	1.24	33.878	20.00	2.454	34.5	34.5	1.27	1.34	<0.009	0
3/14/2000	11:37	1.24	34.686	22.50	2.447	35.4	35.4	1.27	1.34	<0.009	0
3/14/2000	11:40	1.24	35.187	25.00	2.441	36.0	36.0	1.27	1.34	<0.009	0
3/14/2000	11:42	1.24	34.874	27.50	2.435	35.8	35.8	1.27	1.34	<0.009	0
3/14/2000	11:45	1.24	34.799	30.00	2.429	35.8	35.8	1.27	1.34	<0.009	0
3/14/2000	11:47	1.24	34.583	32.50	2.423	35.7	35.7	1.27	1.34	<0.009	0
3/14/2000	11:50	1.24	34.911	35.00	2.417	36.1	36.1	1.27	1.34	<0.009	0
3/14/2000	11:52	1.24	34.555	37.50	2.411	35.8	35.8	1.27	1.34	<0.009	0
3/14/2000	11:55	1.24	32.989	40.00	2.405	34.3	34.3	0.62	0.69	<0.009	0
3/14/2000	11:57	1.24	27.581	42.50	2.399	28.7	28.7	0.62	0.69	<0.009	0
3/14/2000	12:00	1.24	5.293	45.00	2.393	5.5	28.7	0.62	0.69	<0.009	0
3/14/2000	12:02	1.24	4.235	47.50	2.387	4.4	28.7	0.62	0.69	<0.009	0
3/14/2000	12:05	1.24	0	50.00	2.381	0.0	28.7	0.62	0.69	<0.009	0
3/14/2000	12:07	1.24	0.521	52.50	2.375	0.5	28.7	0.62	0.69	<0.009	0
3/14/2000	12:10	1.24	0	55.00	2.369	0.0	28.7	0.62	0.69	<0.009	0
3/14/2000	12:12	1.24	0	57.50	2.363	0.0	28.7	0.62	0.69	<0.009	0

	Hg ²⁺ ng/m ³	PHg	ng/m ³
RGM-0314-9A	2.26	P-0314-9A	-0.15
RGM-0314-9B	1.27	P-0314-9B	-0.15
RGM-0314-9C	0.62	P-0314-9C	-0.04

	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3
Sampling Method	Hg total	PHg	Hg0	Hg2+	Hg total	PHg	Hg0	Hg2+
OH	262	56	10	196	245	43	7.6	194
SOH	228	30	5.0	193				

SPDC RUN 10 with ESP, with light, no rain, with ozone, with flue gas spiking of HgCl2 and Hg0
 (ALL PARTICULATE TRAPS HAVE BLACK SOOT- THUS LOSS OF HG(0) EXPLAINED)

Date	Time	Vol l	Hg ⁰ ng/m3	Dilution Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Adjusted Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/14/2000	16:30	2.50	40.1	0.0	n/a	40.1	40.1	9.16	9.30	5.12	5.20
3/14/2000	16:32	2.50	43.0	2.5	2.494	43.1	43.1	9.16	9.30	5.12	5.20
3/14/2000	16:35	2.50	42.8	5.0	2.481	43.1	43.1	9.16	9.30	5.12	5.20
3/14/2000	16:37	2.50	43.0	7.5	2.469	43.6	43.6	9.16	9.30	5.12	5.20
3/14/2000	16:40	2.50	42.2	10.0	2.457	42.9	42.9	9.16	9.30	5.12	5.20
3/14/2000	16:42	2.50	30.4	12.5	2.444	31.1	42.2	9.16	9.30	5.12	5.20
3/14/2000	16:45	2.50	6.1	15.0	2.432	6.3	42.2	9.16	9.30	5.12	5.20
3/14/2000	16:47	2.50	5.8	17.5	2.420	6.0	42.2	9.16	9.30	5.12	5.20
3/14/2000	16:50	2.50	33.5	20.0	2.408	34.7	42.2	1.84	1.94	2.61	2.76
3/14/2000	16:52	2.50	39.7	22.5	2.396	41.5	41.5	1.84	1.94	2.61	2.76
3/14/2000	16:55	2.50	41.4	25.0	2.384	43.4	43.4	1.84	1.94	2.61	2.76
3/14/2000	16:57	2.50	41.2	27.5	2.372	43.4	43.4	1.84	1.94	2.61	2.76
3/14/2000	17:00	2.50	40.9	30.0	2.360	43.4	43.4	1.84	1.94	2.61	2.76
3/14/2000	17:02	2.50	40.0	32.5	2.349	42.6	42.6	1.84	1.94	2.61	2.76
3/14/2000	17:05	2.50	36.6	35.0	2.337	39.2	42.8	1.84	1.94	2.61	2.76
3/14/2000	17:07	2.50	28.2	37.5	2.325	30.3	42.8	1.84	1.94	2.61	2.76
3/14/2000	17:10	2.50	39.8	40.0	2.314	43.1	43.1	1.05	1.15	1.91	2.10
3/14/2000	17:12	2.50	40.7	42.5	2.302	44.2	44.2	1.05	1.15	1.91	2.10
3/14/2000	17:15	2.50	41.3	45.0	2.291	45.1	45.1	1.05	1.15	1.91	2.10
3/14/2000	17:17	2.50	38.6	47.5	2.279	42.4	42.4	1.05	1.15	1.91	2.10
3/14/2000	17:20	2.50	21.7	50.0	2.268	23.9	42.4	1.05	1.15	1.91	2.10
3/14/2000	17:22	2.50	12.5	52.5	2.256	13.8	42.4	1.05	1.15	1.91	2.10
3/14/2000	17:25	2.50	5.8	55.0	2.245	6.5	42.4	1.05	1.15	1.91	2.10
3/14/2000	17:27	2.50	2.9	57.5	2.234	3.3	42.4	1.05	1.15	1.91	2.10

Hg ²⁺	ng/m ³	PHg	ng/m ³
Hg ²⁺	ng/m ³	PHg	ng/m ³
RGM-0314-10A	9.16	P-0314-10A	5.12
RGM-0314-10B	1.84	P-0314-10B	2.61
RGM-0314-10C	1.05	P-0314-10C	1.91

EXPECTED SPDC CONCENTRATION					MEAN EXPECTED SPDC CONCENTRATION			
Sampling Method	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	181	50	10	122	160	48	7.1	105
SOH	138	46	4.6	88				

SPDC RUN 11 with Baghouse, no light, no rain, no spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m3	Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Adjusted Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/15/2000	10:32	2.5	6.7	0.0	n/a	6.71	6.71	3.50	3.56	0.22	0.22
3/15/2000	10:35	2.5	5.6	2.5	2.49376	5.62	5.62	3.50	3.56	0.22	0.22
	10:37	2.5	2.1	5.0	2.481323	2.10	5.31	3.50	3.56	0.22	0.22
3/15/2000	10:40	2.5	0.1	7.5	2.468947	0.14	5.31	3.50	3.56	0.22	0.22
3/15/2000	10:42	2.5	0.0	10.0	2.456633	0.00	5.31	3.50	3.56	0.22	0.22
3/15/2000	10:45	2.5	0.0	12.5	2.444381	0.00	5.31	3.50	3.56	0.22	0.22
3/15/2000	10:47	2.5	0.0	15.0	2.432189	0.00	5.31	3.50	3.56	0.22	0.22
3/15/2000	10:50	2.5	0.0	17.5	2.420059	0.00	5.31	3.50	3.56	0.22	0.22
3/15/2000	10:52	2.5	1.7	20.0	2.407989	1.76	5.31	0.22	0.23	0.05	0.05
3/15/2000	10:55	2.5	4.8	22.5	2.395979	5.01	5.01	0.22	0.23	0.05	0.05
3/15/2000	10:57	2.5	0.0	25.0	2.384029	0.00	3.24	0.22	0.23	0.05	0.05
3/15/2000	11:00	2.5	0.0	27.5	2.372138	0.00	3.24	0.22	0.23	0.05	0.05
3/15/2000	11:02	2.5	0.0	30.0	2.360307	0.00	3.24	0.22	0.23	0.05	0.05
3/15/2000	11:05	2.5	0.0	32.5	2.348535	0.00	3.24	0.22	0.23	0.05	0.05
3/15/2000	11:07	2.5	0.0	35.0	2.336822	0.00	3.24	0.22	0.23	0.05	0.05
3/15/2000	11:10	2.5	0.0	37.5	2.325167	0.00	3.24	0.22	0.23	0.05	0.05
3/15/2000	11:12	2.5	1.4	40.0	2.31357	1.47	1.47	0.62	0.68	<0.009	0.00
3/15/2000	11:15	2.5	0.8	42.5	2.302031	0.89	0.89	0.62	0.68	<0.009	0.00
3/15/2000	11:17	2.5	0.0	45.0	2.29055	0.00	0.89	0.62	0.68	<0.009	0.00
3/15/2000	11:20	2.5	0.0	47.5	2.279125	0.00	0.89	0.62	0.68	<0.009	0.00
3/15/2000	11:22	2.5	0.0	50.0	2.267758	0.00	0.89	0.62	0.68	<0.009	0.00
3/15/2000	11:25	2.5	0.0	52.5	2.256448	0.00	0.89	0.62	0.68	<0.009	0.00
3/15/2000	11:27	2.5	0.0	55.0	2.245194	0.00	0.89	0.62	0.68	<0.009	0.00
3/15/2000	11:30	2.5	0.0	57.5	2.233996	0.00	0.89	0.62	0.68	<0.009	0.00

Hg ²⁺ ng/m ³	PHg ng/m ³
RGM-0315-11A 3.50	P-0315-11A 0.217
RGM-0315-11B 0.22	P-0315-11B 0.047
RGM-0315-11C 0.62	P-0315-11C -0.004

Sampling Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	120	0.4	4.9	115	114	0.4	2.9	110
SOH	107	NA	0.9	106				

SPDC RUN 12 with Baghouse, with light, with rain, no spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m3	Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/15/2000	15:20	2.5	9.00	0.0	n/a	9.00	0.724	0.74	1.08	1.09
3/15/2000	15:22	2.5	9.23	2.5	2.494	9.26	0.724	0.74	1.08	1.09
3/15/2000	15:25	2.5	9.57	5.0	2.481	9.65	0.724	0.74	1.08	1.09
3/15/2000	15:27	2.5	9.90	7.5	2.469	10.02	0.724	0.74	1.08	1.09
3/15/2000	15:30	2.5	9.63	10.0	2.457	9.80	0.724	0.74	1.08	1.09
3/15/2000	15:32	2.5	9.96	12.5	2.444	10.19	0.724	0.74	1.08	1.09
3/15/2000	15:35	2.5	9.74	15.0	2.432	10.01	0.724	0.74	1.08	1.09
3/15/2000	15:37	2.5	10.19	17.5	2.420	10.52	0.724	0.74	1.08	1.09
3/15/2000	15:40	2.5	9.26	20.0	2.408	9.62	0.020	0.02	0.05	0.06
3/15/2000	15:42	2.5	9.46	22.5	2.396	9.87	0.020	0.02	0.05	0.06
3/15/2000	15:45	2.5	9.43	25.0	2.384	9.89	0.020	0.02	0.05	0.06
3/15/2000	15:47	2.5	9.40	27.5	2.372	9.90	0.020	0.02	0.05	0.06
3/15/2000	15:50	2.5	8.80	30.0	2.360	9.32	0.020	0.02	0.05	0.06
3/15/2000	15:52	2.5	9.20	32.5	2.349	9.79	0.020	0.02	0.05	0.06
3/15/2000	15:55	2.5	8.75	35.0	2.337	9.36	0.020	0.02	0.05	0.06
3/15/2000	15:57	2.5	9.29	37.5	2.325	9.99	0.020	0.02	0.05	0.06
3/15/2000	16:00	2.5	8.72	40.0	2.314	9.42	<0.007	0.00	1.98	2.18
3/15/2000	16:02	2.5	8.46	42.5	2.302	9.19	<0.007	0.00	1.98	2.18
3/15/2000	16:05	2.5	7.83	45.0	2.291	8.54	<0.007	0.00	1.98	2.18
3/15/2000	16:07	2.5	8.31	47.5	2.279	9.11	<0.007	0.00	1.98	2.18
3/15/2000	16:10	2.5	8.02	50.0	2.268	8.84	<0.007	0.00	1.98	2.18
3/15/2000	16:12	2.5	8.31	52.5	2.256	9.21	<0.007	0.00	1.98	2.18
3/15/2000	16:15	2.5	7.93	55.0	2.245	8.83	<0.007	0.00	1.98	2.18
3/15/2000	16:17	2.5	8.68	57.5	2.234	9.71	<0.007	0.00	1.98	2.18

	Hg ²⁺ ng/m ³	PHg ng/m ³
RGM-0315-12A	0.724	1.076
RGM-0315-12B	0.020	0.052
RGM-0315-12C	-0.022	1.981

	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3	ng/m3
Sampling Method	Hg total	PHg	Hg0	Hg2+	Hg total	PHg	Hg0	Hg2+
OH	117	0.1	4.0	112	108	0.1	2.8	106
SOH	100	NA	1.6	99				

SPDC RUN 13 with Baghouse, no light, no rain, with spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m3	Time min	Dilution Corr. Factor	Dilution Hg ⁰ ng/m ³	Corrected Hg ²⁺ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/16/2000	10:15	2.5	63.27	0.0	n/a	63.3	63.3	2.68	2.72	0.07	0.07
3/16/2000	10:17	2.5	64.07	2.5	2.494	64.2	64.2	2.68	2.72	0.07	0.07
3/16/2000	10:20	2.5	61.93	5.0	2.481	62.4	62.4	2.68	2.72	0.07	0.07
3/16/2000	10:22	2.5	60.20	7.5	2.469	61.0	61.0	2.68	2.72	0.07	0.07
3/16/2000	10:25	2.5	50.74	10.0	2.457	51.6	59.7	2.68	2.72	0.07	0.07
3/16/2000	10:27	2.5	51.80	12.5	2.444	53.0	59.7	2.68	2.72	0.07	0.07
3/16/2000	10:30	2.5	30.78	15.0	2.432	31.6	59.7	2.68	2.72	0.07	0.07
3/16/2000	10:32	2.5	42.39	17.5	2.420	43.8	59.7	2.68	2.72	0.07	0.07
3/16/2000	10:35	2.5	55.68	20.0	2.408	57.8	57.8	1.44	1.52	<0.009	0.00
3/16/2000	10:37	2.5	36.63	22.5	2.396	38.2	59.7	1.44	1.52	<0.009	0.00
3/16/2000	10:40	2.5	9.26	25.0	2.384	9.7	59.7	1.44	1.52	<0.009	0.00
3/16/2000	10:42	2.5	15.17	27.5	2.372	16.0	59.7	1.44	1.52	<0.009	0.00
3/16/2000	10:45	2.5	3.79	30.0	2.360	4.0	59.7	1.44	1.52	<0.009	0.00
3/16/2000	10:47	2.5	4.80	32.5	2.349	5.1	59.7	1.44	1.52	<0.009	0.00
3/16/2000	10:50	2.5	2.50	35.0	2.337	2.7	59.7	1.44	1.52	<0.009	0.00
3/16/2000	10:52	2.5	3.07	37.5	2.325	3.3	59.7	1.44	1.52	<0.009	0.00
3/16/2000	10:55	2.5	44.89	40.0	2.314	48.5	59.7	0.95	1.04	<0.009	0.00
3/16/2000	10:57	2.5	43.44	42.5	2.302	47.2	59.7	0.95	1.04	<0.009	0.00
3/16/2000	11:00	2.5	55.02	45.0	2.291	60.0	60.0	0.95	1.04	<0.009	0.00
3/16/2000	11:02	2.5	54.40	47.5	2.279	59.7	59.7	0.95	1.04	<0.009	0.00
3/16/2000	11:05	2.5	55.62	50.0	2.268	61.3	61.3	0.95	1.04	<0.009	0.00
3/16/2000	11:07	2.5	55.35	52.5	2.256	61.3	61.3	0.95	1.04	<0.009	0.00
3/16/2000	11:10	2.5	55.02	55.0	2.245	61.3	61.3	0.95	1.04	<0.009	0.00
3/16/2000	11:12	2.5	55.02	57.5	2.234	61.6	61.6	0.95	1.04	<0.009	0.00

Hg ²⁺	ng/m ³	PHg	ng/m ³
RGM-0316-13A	2.68	P-0316-13A	0.070
RGM-0316-13B	1.44	P-0316-13B	-0.086
RGM-0316-13C	0.95	P-0316-13C	-0.139

Sampling Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	226	0.1	47	178	214	0.1	45	169
SOH	203	NA	44	160				

SPDC RUN 14 with Baghouse, with light, with rain, with spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m3	Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Corrected Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/16/2000	13:25:00	2.5	61.43	0.0	n/a	61.4	0.700	0.711	<0.009	0
3/16/2000	13:27:30	2.5	64.14	2.5	2.4938	64.3	0.700	0.711	<0.009	0
3/16/2000	13:30:00	2.5	63.58	5.0	2.4813	64.1	0.700	0.711	<0.009	0
3/16/2000	13:32:30	2.5	63.46	7.5	2.4689	64.3	0.700	0.711	<0.009	0
3/16/2000	13:35:00	2.5	62.89	10.0	2.4566	64.0	0.700	0.711	<0.009	0
3/16/2000	13:37:30	2.5	63.64	12.5	2.4444	65.1	0.700	0.711	<0.009	0
3/16/2000	13:40:00	2.5	63.14	15.0	2.4322	64.9	0.700	0.711	<0.009	0
3/16/2000	13:42:30	2.5	63.57	17.5	2.4201	65.7	0.700	0.711	<0.009	0
3/16/2000	13:45:00	2.5	62.57	20.0	2.4080	65.0	0.201	0.212	<0.009	0
3/16/2000	13:47:30	2.5	62.95	22.5	2.3960	65.7	0.201	0.212	<0.009	0
3/16/2000	13:50:00	2.5	62.06	25.0	2.3840	65.1	0.201	0.212	<0.009	0
3/16/2000	13:52:30	2.5	62.23	27.5	2.3721	65.6	0.201	0.212	<0.009	0
3/16/2000	13:55:00	2.5	61.82	30.0	2.3603	65.5	0.201	0.212	<0.009	0
3/16/2000	13:57:30	2.5	61.91	32.5	2.3485	65.9	0.201	0.212	<0.009	0
3/16/2000	14:00:00	2.5	61.42	35.0	2.3368	65.7	0.201	0.212	<0.009	0
3/16/2000	14:02:30	2.5	61.59	37.5	2.3252	66.2	0.201	0.212	<0.009	0
3/16/2000	14:05:00	2.5	61.05	40.0	2.3136	66.0	0.213	0.234	0.090	0.099
3/16/2000	14:07:30	2.5	61.41	42.5	2.3020	66.7	0.213	0.234	0.090	0.099
3/16/2000	14:10:00	2.5	61.25	45.0	2.2905	66.9	0.213	0.234	0.090	0.099
3/16/2000	14:12:30	2.5	62.03	47.5	2.2791	68.0	0.213	0.234	0.090	0.099
3/16/2000	14:15:00	2.5	61.62	50.0	2.2678	67.9	0.213	0.234	0.090	0.099
3/16/2000	14:17:30	2.5	61.71	52.5	2.2564	68.4	0.213	0.234	0.090	0.099
3/16/2000	14:20:00	2.5	61.16	55.0	2.2452	68.1	0.213	0.234	0.090	0.099
3/16/2000	14:22:30	2.5	61.22	57.5	2.2340	68.5	0.213	0.234	0.090	0.099

	Hg ²⁺ ng/m ³	PHg ng/m ³
RGM-0316-14A	0.700	P-0316-14A -0.088
RGM-0316-14B	0.201	P-0316-14B -0.085
RGM-0316-14C	0.213	P-0316-14C 0.090

Sampling Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	226	0.1	51	174	219	0.1	53	166
SOH	213	NA	55	158				

SPDC RUN 15 with Baghouse, no light, with rain, with spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m3	Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Adjusted Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/16/2000	16:37:30	2.5	64.6	0.0	n/a	64.6	64.6	0.613	0.622	0.070	0.071
3/16/2000	16:40:00	2.5	65.6	2.5	2.494	65.7	65.7	0.613	0.622	0.070	0.071
3/16/2000	16:42:30	2.5	65.8	5.0	2.481	66.3	66.3	0.613	0.622	0.070	0.071
3/16/2000	16:45:00	2.5	65.7	7.5	2.469	66.5	66.5	0.613	0.622	0.070	0.071
3/16/2000	16:47:30	2.5	66.8	10.0	2.457	68.0	68.0	0.613	0.622	0.070	0.071
3/16/2000	16:50:00	2.5	65.8	12.5	2.444	67.3	67.3	0.613	0.622	0.070	0.071
3/16/2000	16:52:30	2.5	66.6	15.0	2.432	68.5	68.5	0.613	0.622	0.070	0.071
3/16/2000	16:55:00	2.5	65.3	17.5	2.420	67.5	67.5	0.613	0.622	0.070	0.071
3/16/2000	16:57:30	2.5	2.0	20.0	2.408	2.1	68.6	0.500	0.528	<0.009	0.000
3/16/2000	17:00:00	2.5	43.8	22.5	2.396	45.7	68.6	0.500	0.528	<0.009	0.000
3/16/2000	17:02:30	2.5	66.5	25.0	2.384	69.7	69.7	0.500	0.528	<0.009	0.000
3/16/2000	17:05:00	2.5	65.5	27.5	2.372	69.0	69.0	0.500	0.528	<0.009	0.000
3/16/2000	17:07:30	2.5	65.5	30.0	2.360	69.4	69.4	0.500	0.528	<0.009	0.000
3/16/2000	17:10:00	2.5	64.6	32.5	2.349	68.7	68.7	0.500	0.528	<0.009	0.000
3/16/2000	17:12:30	2.5	65.1	35.0	2.337	69.7	69.7	0.500	0.528	<0.009	0.000
3/16/2000	17:15:00	2.5	64.4	37.5	2.325	69.2	69.2	0.500	0.528	<0.009	0.000
3/16/2000	17:17:30	2.5	65.3	40.0	2.314	70.6	70.6	<0.007	0.000	0.159	0.175
3/16/2000	17:20:00	2.5	64.6	42.5	2.302	70.2	70.2	<0.007	0.000	0.159	0.175
3/16/2000	17:22:30	2.5	65.3	45.0	2.291	71.2	71.2	<0.007	0.000	0.159	0.175
3/16/2000	17:25:00	2.5	64.3	47.5	2.279	70.6	70.6	<0.007	0.000	0.159	0.175
3/16/2000	17:27:30	2.5	65.2	50.0	2.268	71.9	71.9	<0.007	0.000	0.159	0.175
3/16/2000	17:30:00	2.5	64.3	52.5	2.256	71.3	71.3	<0.007	0.000	0.159	0.175
3/16/2000	17:32:30	2.5	65.0	55.0	2.245	72.3	72.3	<0.007	0.000	0.159	0.175
3/16/2000	17:35:00	2.5	64.1	57.5	2.234	71.7	71.7	<0.007	0.000	0.159	0.175

Hg ²⁺ ng/m ³	PHg ng/m ³
RGM-0316-15A ##### P-0316-15A	0.070
RGM-0316-15B ##### P-0316-15B	-0.098
RGM-0316-15C ##### P-0316-15C	0.159

Sampling Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	217	0.2	52	165	219	0.2	51	168
SOH	220	NA	50	170				

SPDC RUN 16 with Baghouse, with light, no rain, with spiking of HgCl2 and Hg0

Date	Time	Vol l	Hg ⁰ ng/m ³	Time min	Dilution Corr. Factor	Corrected Hg ⁰ ng/m ³	Adjusted Hg ⁰ ng/m ³	Hg ²⁺ ng/m ³	Corrected Hg ²⁺ ng/m ³	PHg ng/m ³	Corrected PHg ng/m ³
3/17/2000	10:12:30	2.49	48.6	0.0	n/a	48.6	48.6	3.77	3.83	<0.009	0
3/17/2000	10:15:00	2.5	47.7	2.5	2.494	47.8	47.8	3.77	3.83	<0.009	0
3/17/2000	10:17:30	2.5	45.0	5.0	2.481	45.4	45.4	3.77	3.83	<0.009	0
3/17/2000	10:20:00	2.5	41.8	7.5	2.469	42.3	42.3	3.77	3.83	<0.009	0
3/17/2000	10:22:30	2.5	42.0	10.0	2.457	42.7	42.7	3.77	3.83	<0.009	0
3/17/2000	10:25:00	2.5	37.7	12.5	2.444	38.5	43.4	3.77	3.83	<0.009	0
3/17/2000	10:27:30	2.5	37.9	15.0	2.432	38.9	43.4	3.77	3.83	<0.009	0
3/17/2000	10:30:00	2.5	31.2	17.5	2.420	32.2	43.4	3.77	3.83	<0.009	0
3/17/2000	10:32:30	2.5	42.4	20.0	2.408	44.0	44.0	1.31	1.38	<0.009	0
3/17/2000	10:35:00	2.5	46.2	22.5	2.396	48.2	48.2	1.31	1.38	<0.009	0
3/17/2000	10:37:30	2.5	46.7	25.0	2.384	49.0	49.0	1.31	1.38	<0.009	0
3/17/2000	10:40:00	2.5	45.5	27.5	2.372	48.0	48.0	1.31	1.38	<0.009	0
3/17/2000	10:42:30	2.5	44.1	30.0	2.360	46.7	46.7	1.31	1.38	<0.009	0
3/17/2000	10:45:00	2.5	38.5	32.5	2.349	41.0	41.0	1.31	1.38	<0.009	0
3/17/2000	10:47:30	2.5	30.9	35.0	2.337	33.1	40.7	1.31	1.38	<0.009	0
3/17/2000	10:50:00	2.5	16.1	37.5	2.325	17.3	40.7	1.31	1.38	<0.009	0
3/17/2000	10:52:30	2.5	37.4	40.0	2.314	40.5	40.5	1.23	1.35	<0.009	0
3/17/2000	10:55:00	2.5	42.5	42.5	2.302	46.1	46.1	1.23	1.35	<0.009	0
3/17/2000	10:57:30	2.5	42.9	45.0	2.291	46.8	46.8	1.23	1.35	<0.009	0
3/17/2000	11:00:00	2.5	42.9	47.5	2.279	47.1	47.1	1.23	1.35	<0.009	0
3/17/2000	11:02:30	2.5	43.1	50.0	2.268	47.6	47.6	1.23	1.35	<0.009	0
3/17/2000	11:05:00	2.5	42.6	52.5	2.256	47.2	47.2	1.23	1.35	<0.009	0
3/17/2000	11:07:30	2.5	42.6	55.0	2.245	47.4	47.4	1.23	1.35	<0.009	0
3/17/2000	11:10:00	2.5	41.8	57.5	2.234	46.8	46.8	1.23	1.35	<0.009	0

Hg ²⁺	ng/m ³	PHg	ng/m ³
RGM-0317-16A	3.77	P-0317-16A	-0.004
RGM-0317-16B	1.31	P-0317-16B	0.004
RGM-0317-16C	1.23	P-0317-16C	-0.173

Sampling Method	EXPECTED SPDC CONCENTRATION				MEAN EXPECTED SPDC CONCENTRATION			
	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+	ng/m3 Hg total	ng/m3 PHg	ng/m3 Hg0	ng/m3 Hg2+
OH	261	0.1	50	211	250	0.1	50	201
SOH	240	NA	50	190				

APPENDIX B

A VALIDATION STUDY AT THE EERC OF THE FLUE GAS MERCURY SORBENT SPECIATION OR FMSS METHOD

**A Validation Study at the EERC of the
Fluegas Mercury Sorbent Speciation or
FMSS Method**

FMSS Speciation Method - Performance Based Measurement System (PBMS)

Outline of document to satisfy the PBMS requirements

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I. Abstract

An analytical study, using the guidelines of the US EPA Performance Based Measurement System (PBMS) was completed for the Fluegas Mercury Sorbent Speciation (or FMSS) Method. The FMSS method uses a semi-isokinetic sampling approach with a mini-particulate filter in the fluegas duct followed by a heated solid sorbent sample train to selectively determine particulate Hg (PHg), gaseous oxidized Hg (Hg^{+2}) and elemental Hg (Hg^0) and the sum of species, total Hg (THg). The analysis of the FMSS samples is done after appropriate strong acid digestion using cold vapor atomic fluorescence spectroscopy (CVAFS) using EPA Method 1631B (modified). The justification for this study is twofold: first the FMSS method has distinct advantages over the currently employed impinger method for use in emission and Hg control studies and secondly may be desirable for continuous emission monitor (CEM) development and then ongoing quality assurance performance evaluation. The intent of the PBMS approach to validation of a method is to define and determine under real conditions the scope, quality control requirements, method detection limit, ruggedness, accuracy, precision, interferences, matrix suitability and laboratory reproducibility. All aspects of the PBMS analytical protocol were addressed in this study. The majority of the PBMS was conducted at the University of North Dakota's Energy and Environmental Research Center Pilot Plant. Another study of the FMSS was completed at the US Department of Energy Pilot Plant, with similar results, but is not summarized here. Most importantly, all FMSS Method sample runs were collected in parallel with the ASTM approved Ontario Hydro (OH) Method. The study was conducted using various fuels, two types of particulate control devices, selective fluegas Hg species spiking and both sample flow rate and temperature variations. For all tests, at least a duplicate FMSS and OH sample were collected, and frequent quadruplicate samples were collected for the FMSS.

The FMSS method was evaluated relative to the Ontario Hydro (OH) Method at the Energy and Environment Research Center (EERC) and again at the US DOE for a variety of fuels, ash loadings, and pollution control methods. FMSS method detection limits or MDLs for coal combustion fluegas are reported (in $\mu\text{g}/\text{m}^3$) to be 0.006 for PHg, 0.027 for Hg^0 , 0.0251 for Hg^{+2} , and 0.045 for THg. The FMSS method exhibited good precision with a mean relative percent difference (RPD) of, $\pm 8\%$ for Hg^{+2} , $\pm 10\%$ for THg, $\pm 6.5\%$ for $\%\text{Hg}^{+2}$ (as the percent Hg^{+2} of the total vapor and particulate phases) $\pm 22\%$ for Hg^0 and $\pm 47\%$ for PHg. The precision values for Hg^0 and PHg were greater than the other species due to the very low concentrations measured, not inherent method or laboratory problems. Good agreement was observed between the means of the FMSS and OH method runs, with accuracy at $100 \pm 20\%$ for all species and a mean of 97% for THg and $\%\text{Hg}^{+2}$.

Based on the results of this PBMS, we conclude the FMSS Method is equivalent to the ASTM approved OH Method and a therefore a valid method for the determination of total Hg, PHg, gaseous Hg^{+2} and Hg^0 concentrations in fluegas matrices. Considering many factors, including simplicity, lack of hazardous solutions in the field, precision, sensitivity, accuracy and cost, the FMSS method has many advantages that make it a viable choice for the measurement of fluegas Hg speciation.

II. Scope and Application

The scope is to subject the Frontier Fluegas Mercury Sorbent Speciation (FMSS) Method to a full Performance Based Measurement System (PBMS) analytical study. Why is the PBMS approach being used to validate the FMSS Method? In short, the EPA Office of Solid Waste strongly supports and is committed to the agreement by the EPA's Environmental Monitoring Management Council (EMMC), with members of the regulatory community and with Congress, that changes must be made in the way monitoring requirements are specified in regulations and in permits (<http://www.epa.gov/SW-846/pbms.htm>). The PBMS approach satisfies this desired change and there is broad acceptance for its application.

The EPA defines PBMS as a set of processes wherein the data needs, mandates, or limitations of a program or project are specified, and serve as criteria for selecting appropriate methods to meet those needs in a cost-effective manner. The flexibility of PBMS extends to publication, where there is no set document type required to communicate the criteria. The primary benefit of PBMS to both regulators and the regulated community is flexibility in method selection including: 1) Expedited approval of new and emerging technologies to meet mandated monitoring requirements and 2) Development and use of cost-effective methods that meet program requirements and their associated performance criteria. As stated by the EPA, "where PBMS is implemented, the regulated community will be able to select an appropriate analytical method... including a method not found in EPA-published manuals that is cost-effective and meets the particular project criteria".

The outcome of this study is to determine if the FMSS method is an acceptable and cost-effective method for the measurement of total and speciated mercury (particulate Hg, gaseous Hg(II) and elemental Hg) in coal fluegas. It is expected that the FMSS method will be applicable to other fluegas matrices, such as from municipal waste incinerators, sewage sludge incinerators, smelting and other high temperature or combustion processes. However, the limitations of this study are for coal fluegas only.

The principle of the FMSS method has been described previously in Prestbo and Bloom (1995) and Prestbo and Tokos (1997). The FMSS method uses a semi-isokinetic sampling approach with a mini-particulate filter in the fluegas duct followed by a heated solid sorbent sample train to selectively determine particulate Hg (PHg), gaseous oxidized Hg (Hg^{2+}) and elemental Hg (Hg^0) (Figure 1). The FMSS method uses dual dry sorbent traps for the gas phase Hg species capture with the first trap consisting of KCl coated quartz chips and the second trap containing tri-iodine impregnated activated carbon. The sampled fluegas pulled by vacuum at 0.5 liters/minute through the sample train separates in order of the traps, particulate Hg (PHg), gaseous Hg^{2+} physi-sorbed on the KCl/quartz trap and finally chemi-sorption of Hg^0 on the iodinated carbon trap. An inlet nozzle is used and its inner diameter is chosen so that the sample is collected isokinetically by matching the nozzle velocity with the fluegas velocity in the duct. The mini-particulate filter consisting of a small quartz-fiber filter disk is inserted into a quartz tube on a pure nickel support screen to collect flyash for particulate Hg (PHg) determination (Lu et al., 1998). After sample collection, the entire sample train trap is sent to a laboratory for analysis. Analysis of the sorbent traps is by CVAFS following strong acid digestion, BrCl oxidation, aqueous SnCl_2 reduction, and dual gold amalgamation (EPA Method 1631B modified). The analysis of the PHg on the flyash is by thermal desorption at 800° C, passing through a heated MnO converter, gold preconcentration and CVAFS detection. In the event of anticipated high flyash loadings, it is recommended for PHg quantification a 47-mm quartz fiber filter should be used for sampling, followed by strong acid digestion and EPA Method 1631 (modified) CVAFS detection.

Figure 1. Schematic of the FMSS Solid Sorbent Sample Train

The intent of the PBMS approach for the FMSS method evaluation is to define and determine under real conditions the quality control requirements, matrix suitability and laboratory reproducibility, method detection limit, ruggedness, accuracy, precision, bias, and interference,. All aspects of the PBMS analytical protocol were addressed in this study. The majority of the PBMS study was conducted at the Energy and Environmental Research Center (EERC) Pilot Plant. One important aspect of the PBMS is to collect samples in different fluegas systems, coal type, sampling technicians and analysis technicians. This was accomplished in a similar intercomparison study of the FMSS and OH Method at the US DOE National Energy Technology Laboratory (DOE/NETL-2001/1147 "Comparison of Sampling Methods to Determine Total and Speciated Mercury in Flue Gas," CRADA 00-F038 Final Report). Most importantly, for this study, all FMSS Method sample runs were collected in parallel with the ASTM approved Ontario Hydro (OH) Method. The study was conducted using various fuels, two types of particulate control devices, selective fluegas Hg species spiking and both sample flow rate and temperature variations. For all tests, at least a duplicate FMSS and OH sample were collected, and frequent quadruplicate samples were collected for the FMSS.

The full matrix of 16 tests at the EERC facility are listed below in Table II-1.

Table II-1: Summary of FMS Tests at EERC

Sample					OH	FMS	
Date	Run	Fuel	PCD	Hg Spiked	Samples	Samples	Notes
3/6/00	0	Nat'l Gas	Baghouse	none	none	4	MDL-precision at low level [Hg]
3/7/00	0	Nat'l Gas	Baghouse	none	none	5	MDL-precision at low level
3/7/00	1	Nat'l Gas	Baghouse	Hg ⁰	2	3 + FB	Hg ²⁺ Bias Check – clean matrix
3/7/00	2	Nat'l Gas	Baghouse	HgCl ₂	2	3 + FB	Hg ⁰ Bias Check – clean matrix
3/8/00	3	Bit. Coal	ESP	none	2	4	Flow rate variation
3/8/00	4	Bit. Coal	ESP	none	2	8	MDL – precision at mid level [Hg]
3/10/00	5	Bit. Coal	ESP	none	2	2 + FB	Replicates at mid level [Hg]
3/13/00	6	Bit. Coal	ESP	HgCl ₂ + Hg ⁰	2	2	Spike recovery replicates
3/13/00	7	Bit. Coal	ESP	HgCl ₂ + Hg ⁰	2	2 + FB	Spike recovery replicates
3/13/00	8	Bit. Coal	ESP	HgCl ₂ + Hg ⁰	2	2	Spike recovery replicates
3/14/00	9	Bit. Coal	ESP	HgCl ₂ + Hg ⁰	2	3 + FB	Spike recovery replicates
3/14/00	10	Bit. Coal	ESP	HgCl ₂ + Hg ⁰	2	3 + FB	Spike recovery replicates
3/15/00	11	Bit. Coal	Baghouse	none	2	3 + FB	Replicates at mid level [Hg]
3/15/00	12	Bit. Coal	Baghouse	none	2	4	Sorbent temperature variation
3/16/00	13	Bit. Coal	Baghouse	HgCl ₂ + Hg ⁰	2	4	Sorbent temperature variation
3/16/00	14	Bit. Coal	Baghouse	HgCl ₂ + Hg ⁰	2	3 + FB	Spike recovery replicates
3/16/00	15	Bit. Coal	Baghouse	HgCl ₂ + Hg ⁰	2	4	Flow rate variation
3/17/00	16	Bit. Coal	Baghouse	HgCl ₂ + Hg ⁰	2	7	MDL – precision at high level [Hg]
Total					32	66 + 8FB	

II.A Justification

Hg speciation is important both as an aid to engineering efforts focused on Hg control; and as a tool for evaluating the fate and transport of Hg, since fuel combustion is a significant input of anthropogenic Hg to the atmosphere. As each species has dramatically different chemical and biological properties, understanding its speciation directly effects our ability to model its effects (Bullock et al, 1997, Pai et al.1996).

II.B Historical Studies of Interest

Bloom (1993) identified a need to develop and verify methods to accurately quantify the mercury speciation in combustion fluegases. Prestbo and Bloom (1994, 1995) introduced the MESA method, a much simpler and more economical sampling collection method than the complex bubbling systems that were currently employed in stack-gas monitoring. The MESA sampling system for gas phase Hg employed a series of heated, solid phase adsorbent traps. Flue-gas oxidized Hg species (Hg^{+2}) are adsorbed by a potassium chloride (KCl) impregnated soda lime sorbent. Elemental Hg (Hg^0) is collected by an iodated-carbon sorbent after passing through the KCl/lime. The MESA method had several distinct advantages over the impinger methods, namely: lower detection limits, simplified sample collection, and the added value provided by the species information. Although the MESA method was able to measure THg accurately to within $\pm 10\%$ of accepted methods (Nott et al., 1994, Laudal et al., 1997, Prestbo and Bloom, 1995), the major disadvantage of the MESA method was non-quantitative collection of particulate material. (Prestbo and Bloom, 1995) and possible over-estimation of the oxidized fraction at high SO_2 (Laudal, 1997, Chu and Porcella, 1995) compared to the liquid impinger based methods. The major disadvantages of the impinger methods have been discussed in detail elsewhere (Meij, 1991) but are namely the cost, hazardous chemical transport, large sample volume needed to overcome the high Hg blanks, SO_2 interference, and wall losses.

Because the MESA method was mechanically much easier, Frontier Geosciences set out to improve the method by modifying the sorbents. They initiated some internally funded bench scale experiments, and eventually showed that the positive bias of the MESA method Hg^{+2} was not found when sulfur levels were low ($< 500\text{ppm}$) (Prestbo and Tokos, 1997). Also replaced was the first dry sorbent, KCl/lime, with KCl/quartz. This new method has since been referred to as the Fluegas Mercury Sorbent Speciation method or FMSS.

Ongoing tests in the past year of the FMSS method in numerous studies have shown that the potential for bias has been eliminated by this change in solid sorbent chemistry. What follows are experimental results from a rigorous evaluation of the FMSS method, including technical information about the method. By directly comparing the FMSS method to the ASTM standard Ontario Hydro (OH) method, validation of the FMSS method will result with acceptable accuracy, precision for total and speciated mercury.

II.C Objectives

The objectives of this study were twofold. First, was to collect good quality data based on the 'test matrix' presented in Table II-1 and to evaluate the FMSS method under the EPA's new PBMS relative the EPA standard OH method. And second, the object was to define and determine under real conditions the scope, quality control requirements, method detection limit, ruggedness, accuracy, precision, interferences, matrix suitability and laboratory reproducibility.

II.D Target Analytes

In combustion fluegas, mercury is present as elemental (Hg^0), oxidized (or Hg^{+2}) and particulate (PHg) forms. Total Hg (THg) is the sum of the gas phase Hg^0 and Hg^{+2} and PHg. The $\%\text{Hg}^{+2}$ is the fraction of the Hg that exists as Hg^{+2} . The symbols that will be referred to through out the document are listed in Table II-2.

Table II-2

Nomenclature of the Target Analytes

Particulate Hg	PHg
Elemental Hg	Hg^0
Oxidized Hg	Hg^{+2}
Total Hg	$\text{THg} = \text{Hg}^0 + \text{Hg}^{+2} + \text{PHg}$
Percent Oxidized Hg	$\%\text{Hg}^{+2} = \text{Hg}^{+2} / \text{THg}$

II.E Applicable Matrices

The applicable matrices for the FMSS method include fluegas from the combustion of natural gas and/or coal in the presence of SO_2 , NO_x , HCl, and flyash. It is expected that the FMSS method will be applicable to other fluegas matrices, such as from municipal waste incinerators, sewage sludge incinerators, smelting and other high temperature or combustion processes. However, the limitations of this study are for coal fluegas only.

II.F Availability of Equipment

Fluegas is a challenging sampling environment. However, the equipment required to collect the FMSS method samples are standard air sampling equipment, such as in-stack probes, mass flow meters, sampling media or solid sorbents, and temperature controllers (Table II-3). The handling procedures are technical, but easy to learn for an experience stack sampler. The FMSS method sorbents are made routinely at Frontier Geosciences with careful screening to make sure blanks are low and consistent (<1 ng/trap). Hg analysis requires setting up a low-level cold vapor atomic fluorescence spectrometer (CVAFS) system as described in EPA Method 1631 and EPA document "Guidance on Establishing Trace Metal Clean Rooms in Existing Facilities." For shipping, the FMSS traps do not need hazardous or controlled substance designations.

Table II-3
Availability of Equipment

	Requirement	Availability of Equipment
CLEAN HANDLING TECHNIQUES	Standard Clean Handling Field Techniques less than 2 hours of training	Standard Stack Sampling Capabilities
GAS SAMPLING TRAPS AND MEDIA	Mini-particulate filter (Quartz-fiber filter in quartz tube on nickel support screen) for PHg	Standard Procedure at FGS
	KCl/quartz traps for gaseous Hg ⁺²	Standard Procedure at FGS
	Iodinated carbon trap for Hg ⁰	Standard Procedure at FGS
TEMPERATURE CONTROL	Temperature control requirements of 90+/-5 deg C using heated probes and temperature controllers	Standard Procedure at FGS
FLOWRATE CONTROL	Flow Rate control requirements of 0.4 +/- 0.1 slpm with fluegas pulled by vacuum to mass flow meters	Standard Procedure at FGS
ANALYTICAL LABORATORY CAPABILITIES	Mercury determinations by CVAFS	Standard CVAFS Analytical Capabilities
	Sorbent traps analyzed by strong acid digestion, BrCl oxidation, aqueous SnCl ₂ reduction, and dual gold amalgamation.	EPA Method 1631B Modified
	Analysis of PHg by thermal desorption at 800 deg C, with MnO converter, gold preconcentration and CVAFS detection	EPA Method 1631B Modified

II.G Cost of Hg Speciation Methods

The costs to perform the FMSS method should be most comparable to the Ontario Hydro method based on most similar inlet configurations and principles of operation. Continuous emission measurement or CEM methods although operating are included here for comparison of costs basis, but the meaning is less valuable, since it operates on different inlets and operating principles. The costs to perform Hg Speciation Methods are outlined in Table II-4. Because it is simple to operate, it requires only a few man-hours to learn.

Table II-4

Cost of Hg Speciation Methods

Hg Speciation Method	Sampling Method	Analytical Method	Approximate Cost for a 3-Day Field Sampling Campaign and Laboratory Analysis
FMSS	Non-continuous	CVAFS	\$10,000
Ontario Hydro	Non-continuous	CVAA	\$30,000
Continuous Emission Monitor	Continuous	CVAFS or CVAA	\$30,000

III. QUALITY CONTROL REQUIREMENTS (Method Quality Objectives)

Presented here is the quality control defined and determined under real conditions for experiments at the EERC. This provided the ongoing quality assurance performance evaluations required by the PBMS, and defines ongoing future evaluations. The numerous results include replicate analysis, laboratory and field blanks, recoveries of standard reference materials, recoveries of matrix spikes, temperature and volume control requirements. Based on the performance of the FMSS method under real conditions during these tests, method quality objectives (MQO) were set. Although the specific results of the study are better than the following MQOs, the MQOs are set higher so that future applications of the method by new users could reasonably be achieved.

III.A Replicate Analysis

This section discusses the replicate analysis in the laboratory and provided the means to measure the precision of the CVAFS analytical method. In most cases, laboratory replicate analysis occurred on the same analysis day, using the same instrument. However, in some instances, replicates sample analysis occurred on different days and in some cases using different instruments. For samples analyzed in duplicate (n=2), a relative percent difference (RPD) was calculated as a percentage using the formula:

$$RPD = \frac{ABS[A-B]}{AVG(A+B)}$$

Laboratory replicate results are presented as analytical duplicates in Table III-1.

Table III-1
FMSS Replicate Analysis

A. ANALYTICAL DUPLICATES for PHg				
Particulate Hg	Rep 1	Rep 2	mean	RPD or RSD
PHG-6-031400	139.9	146.8	143.3	4.8%
PHG-6-031300	23.1	22.79	23.0	1.6%
PHG-5-031400	129.7	147.8	138.8	13.0%
PHG-4-030800	11.5	13.8	12.6	18.5%
PHG-3-031400	91.0	100.9	95.9	10.3%
PHG-2-031400	61.9	64.0	63.0	3.3%
PHG-2-030800	11.5	11.0	11.3	4.5%
PHG-1-031300	15.3	14.82	15.1	3.5%
PHG-2031400	60.6	63.3	61.9	4.5%
			MEAN	7.1%
			STDEV	5.6%
B. ANALYTICAL DUPLICATES (KCLA and KCLB) for Hg ⁺²				
	Rep 1	Rep 2	mean	RPD or RSD
KCLA-9-030800	161.96	181.3	171.6	11.2%
KCLA-8-031500	241.8	258.3	250.1	6.6%
KCLA-7-031700	838.9	824.1	831.5	1.8%
KCLA-7-031600	577.3	621.74	599.5	7.4%
KCLA-7-031500	221.5	209.0	215.3	5.8%
KCLA-7-031400	191.9	352.9	272.4	59.1%
KCLA-7-031300	522.11	522.10	522.1	0.00%
KCLA-6-031700	1003.1	840.6	921.9	17.6%
KCLA-6-031500	246.5	240.7	243.6	2.4%
KCLA-6-031400	128.2	208.2	168.2	47.5%
KCLA-6-031300	692.5	619.3	655.9	11.2%
KCLA-5-031400	406.8	434.5	420.7	6.6%
KCLA-4-031600	621.7	678.6	650.1	8.8%
KCLA-4-031300	766.9	644.54	705.7	17.3%
KCLA-4-030800	166.5	199.2	182.8	17.9%
KCLA-3-031700	882.7	735.7	809.2	18.2%
KCLA-3-031600	812.6	651.8	732.2	22.0%
KCLA-3-031400	628.4	565.0	596.7	10.6%
KCLA-3-031300	589.0	670.56	629.8	13.0%
KCLA-3-030800	234.2	203.8	219.0	13.9%
KCLA-2-031700	869.6	730.9	800.2	17.3%
KCLA-2-031600	761.6	711.1	736.4	6.9%
KCLA-2-031600	660.6	761.6	711.1	14.2%
KCLA-2-031400	556.5	451.0	503.8	20.9%
KCLA-1-031700	905.70	736.0	820.9	20.7%
KCLA-1-031600	742.3	895.7	819.0	18.7%
KCLA-1-031600	895.7	742.3	819.0	18.7%
KCLA-1-031400	482.9	414.9	448.9	15.1%
KCLA-10-030800	133.86	152.1	143.00	12.8%
KCLB-7-031300	150.3	153.4	151.8	2.0%
KCLA-10-030700	378.9	384.6	381.7	1.5%
KCLA-2-030800	177.0	180.7	178.9	2.1%
			MEAN	14.1%
			STDEV	12.3%

Table III-1 (continued)

C. ANALYTICAL DUPLICATES (ICA) for Hg ⁰				
	Rep 1	Rep 2	mean	RPD or RSD
ICA-9-030800	7.46	7.74	7.60	3.7%
ICA-7-031700	196.8	186.3	191.6	5.5%
ICA-7-031600	238.6	215.67	227.2	10.1%
ICA-7-031400	18.8	14.2	16.5	27.8%
ICA-7-031300	191.1	171.71	181.4	10.7%
ICA-6-031700	249.1	220.0	234.6	12.4%
ICA-6-031400	10.4	7.28	8.9	35.6%
ICA-5-031400	22.4	18.81	20.6	17.3%
ICA-4-031600	231.2	202.3	216.7	13.3%
ICA-4-030800	16.3	19.61	17.9	18.8%
ICA-3-031700	234.2	194.5	214.3	18.5%
ICA-3-031600	171.5	169.8	170.6	1.0%
ICA-3-031400	15.4	17.01	16.2	9.7%
ICA-3-030800	17.4	15.75	16.6	9.8%
ICA-2-031700	253.6	220.4	237.0	14.0%
ICA-2-031600	214.7	220.0	217.3	2.4%
ICA-2-031600	225.2	214.7	220.0	4.8%
ICA-2-031400	14.4	11.28	12.8	24.3%
ICA-2-031000	8.3	6.79	7.57	20.6%
ICA-2-030800	13.2	10.9	12.0	19.2%
ICA-1-031700	256.54	233.4	245.0	9.5%
ICA-1-031600	194.0	237.1	215.6	20.0%
ICA-1-031600	237.1	194.0	215.6	20.0%
ICA-1-031400	14.1	11.3	12.7	21.8%
ICA-1-031000	17.4	4.63	11.00	115.8%
ICA-10-030800	4.69	5.2	4.92	9.4%
ICA-7-030700	389.2	395.0	392.1	1.5%
ICA-11-030800	47.8	48.4	48.1	1.4%
			MEAN	17.1%
			STDEV	21.2%

The mean precision of the laboratory replicates was 7.1% for PHg, 17.1% for Hg⁰, and 14.1% for Hg⁺². Based on this result, the MQO for future studies was estimated to be 25% for laboratory replicates.

III.B Field Blanks

Field blanks provided the overall blank, including sample handling in the field, transportation, storage and handling, analytical and matrix considerations. Field blanks were identical to field samples except that the field blanks were downloaded in the field without sampling. Eight field blanks were collected for each type of the sample traps. The means and standard deviations of the means for the field blanks are shown in Table III-2.

Table III-2
FMSS Field Blank Summary

FIELD BLANKS						Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result			
RESULT	PHg	KCLA	KCLB	ICA	T-Vol	[pHg]	[Hg ⁰]	[Hg ⁺²]	[THg]
FMSS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter	ug/m ³	ug/m ³	ug/m ³	ug/m ³
FMS-9-030700 FB	nc	0.1473	0.0859	0.145	30.0	na	0.0048	0.0078	0.0126
FMS-13-030700 FB	nc	2.78	0.0205	0.015	30.0	na	0.0154	0.0029	0.0183
FMS-3-031000 FB	0.0419	0.0395	0.0000	0.490	30.0	0.0014	0.0163	0.0013	0.0177
FMS-5-031300 FB	na	0.3990	0.0000	0.377	30.0	na	0.0126	0.0000	0.0126
FMS-4-031400 FB	0.0239	0.0000	0.0000	0.416	30.0	0.0008	0.0139	0.0000	0.0139
FMS-8-031400 FB	0.132	0.0356	0.0823	0.371	30.0	0.0044	0.0124	0.0039	0.0163
FMS-4-031500 FB	nc	0.0000	0.0000	0.640	30.0	na	0.0213	0.0000	0.0213
FMS-8-031600 FB	nc	0.2026	0.0000	0.789	30.0	na	0.0263	0.0068	0.0330
2.78 t-test									
n=8						FMSS mean	0.0022	0.0154	0.0028
						STDEV	0.0019	0.0064	0.0031
One KCLA value was removed by t-test and replaced by mean.						MDL-A	0.0058	0.0192	0.0093
							0.0202		

Hg trap concentrations (on the left) were used to determine fluegas concentrations (on the right) based on the mean sample volume of 30.0 standard liters. Hg⁺² was determined by KCLA + KCLB; Hg⁰ was determined by ICA ng/trap values. In one case, the KCLA value was considered bad and the value was thrown out based on the result of a t-test.

Laboratory blanks were analyzed along with the calibration curve and then at a rate of 1-in-10 samples thereafter. Based on standard operating procedures of the analytical laboratory laboratory blanks were always less than 1 ng and are available if requested for review.

The method quality objectives or MQOs for field blanks in future studies were estimated based on the results at the EERC and are shown in Table III-3

Table III-3
Field Blank Method Quality Objectives

Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result				
MQO FOR FIELD BLANKS	PHg ug/m ³	Hg ⁰ ug/m ³	Hg ⁺² ug/m ³	THg ug/m ³
LESS THAN	0.02	0.05	0.05	0.05
(STDEV) +/-	0.01	0.02	0.02	0.02

III.C Standard Reference Materials

Standard Reference Material (SRM) spikes and recoveries were used to provide ongoing evaluation of the accuracy of the overall analytical method. The SRM was spiked to a CVAFS analytical bubbler and the recovery of the SRM was calculated in percent as the measured divided by the expected concentration. The SRM used during this study was NIST 1641d, which was standardized by the National Institute of Science & Technology for total Hg. The SRM was analyzed a minimum of once per analysis day and typically several times throughout the day. SRM recovery data are reported in Table III-4.

Table III-4
SRM Recoveries

SRM Recovery of NIST 1641d		
Analysis Date	ng/aliquot	% Recovery
3/16/00	1.583	99.6%
3/16/00	1.336	84.0%
4/27/00	1.731	111.1%
4/27/00	1.661	106.6%
4/28/00	1.618	103.8%
4/28/00	1.527	98.0%
5/1/00	1.456	93.4%
5/1/00	1.491	95.6%
	MEAN	99.0%
	STDEV	8.4%

SRM recoveries averaged $99.0 \pm 8.4\%$ and were considered good. The MQOs for future studies were estimated based on this result. The MQO for SRM Recoveries of $100 \pm 20\%$ would be easily obtainable in future studies.

III.D Matrix Spike Recoveries

Matrix spike recoveries provided an ongoing evaluation of the accuracy of the analytical method for Hg^0 and Hg^{+2} in the presence of the sampling matrix. Matrix spike recoveries were calculated for 1.00ng additions to laboratory analytical bubblers, which contained a sample aliquot.

Recoveries of the 1.00ng matrix spikes are shown in Table III-5. The percent recovery was calculated as:

$$\% \text{ Recovery} = (\text{ng/aliquot of sample with spike}) / (\text{ng/aliquot of sample} + 1.00 \text{ ng})$$

Table III-5
Matrix Spike Recoveries

1.00 ng Analytical Matrix Spikes	
Sample ID	% Recovery
ICA-SOH-7-0307 AS + 1.0 ng	98.0%
KCLA-SOH-10-0307 AS + 1.0 ng	92.8%
KCLA-2030800 AS + 1ng	106.4%
IC-A-11030800 AS + 1ng	107.7%
KCLB-7031300 AS + 1ng	105.0%
PHG-2031400 AS + 1ng	95.5%
KCLA-5031500 AS + 1.0 ng	100.7%
MEAN	100.9%
STDEV	5.72%

Matrix spikes averaged $100.9\% \pm 5.7\%$ and were similar for the different types of samples. The MQO for matrix spikes for future studies was estimated based on these results for Lab Analysis Spike Recoveries to be $100 \pm 20\%$.

III.E Temperature Control

Probe temperatures during normal application of the FMSS method at the EERC were held constant at $90 \pm 5^\circ\text{C}$ using heated-probes and Omega temperature controllers. This is the ideal working temperature range and indicates a relative percent difference of 6%. Proper temperature control during normal application at the EERC was almost always successful, except in a few cases in when condensation was noted on the sample traps upon downloading, which indicated a problem or failure of temperature control. In these cases the sample was compromised and it was noted in the sampling logs.

The importance of the sampling probe temperature to Hg determination was investigated during two quadruplicate temperature tests (Runs 12 and 13). During these runs, four parallel samples were collected that were identical except that probe temperature was varied from 75 to 105°C . This represented a range in temperatures that is one to three times the ideal FMSS range. Temperature control and data for the temperature test results are summarized in Table III-6.

Several points will help in the interpretation of Table III-6. The precision of the temperature control is given as an RSD at the top of the table (A). The analytical data during ruggedness tests is presented at the bottom of the table (B). T-Vol (column 3) is the temperature corrected dry gas volume. Breakthrough is calculated as KCLB/KCLA (column 6). In ideal situations, the breakthrough to the KCLB trap is low ($<2\%$), and even this is taken into consideration by the method as the Hg concentrations on both traps are used to determine Hg^{+2} concentrations. For these tests breakthrough was used to indicate the collection efficiency of the KCl traps for Hg^{+2} during ruggedness tests.

Table III-6

A. Temperature Control at the EERC

OVERALL TEMPERATURE CONTROL AT EERC	
	PROBE TEMP %RSD
Ruggedness , FR Tests	< 6%
Ruggedness , Temp Tests	14.2%
ESP malfunction, additional ruggedness	< 6%
Natural Gas No Tests	< 6%
Bitcoal No Tests	< 6%

B. Temperature and Breakthrough during ruggedness tests

RUN 12 ANALYTICAL TRAP DATA

	Temp	KCLA	KCLB	ICA	T-Vol	$KCLB/KCLA$
	C	ng/trap	ng/trap	ng/trap	corr., liter	%
FMS-5-031500	75.7	217.7	0.09	3.03	26.0	0.04%
FMS-6-031500	88.4	243.6	0.04	5.88	26.2	0.01%
FMS-7-031500	96.4	215.3	0.19	3.09	26.3	0.09%
FMS-8-031500	105.7	250.1	0.35	4.54	26.4	0.14%
mean	91.6					
T, RSD	13.91%					

RUN 13 ANALYTICAL TRAP DATA

Run 15	Temp	KCLA	KCLB	ICA	T-Vol	$KCLB/KCLA$
	C	ng/trap	ng/trap	ng/trap	corr., liter	%
FMS-1-031600	75.3	819.0	15.3	215.6	26.5	1.87%
FMS-2-031600	86.7	711.1	14.9	220.0	26.4	2.10%
FMS-3-031600	96.6	732.2	6.9	170.6	26.3	0.94%
FMS-4-031600	105.9	650.1	61.3	216.7	26.4	9.43%
mean	91.1					
T, RSD	14.45%					

The results of the ruggedness temperature tests are presented elsewhere (see section IV). However, no significant trends were observed between Hg concentration and probe temperature, even at the highest temperature when the breakthrough from the KCLA trap to KCLB trap was significantly higher than the other traps (see $KCLB/KCLA$ in Table III-6). However, the highest temperature (~106°C) was almost four times above the control range and despite the breakthrough, the precision and accuracy were still good.

III.F Flow Rate Control

The ideal flowrate is nominally 0.4 ± 0.1 slpm so that the nozzle diameter must be chosen based on flue gas flow rates to maintain isokinetic sampling conditions within $\pm 25\%$ (see section III-H). At EERC, the ideal flowrate range was 0.4 ± 0.1 slpm. Flow rate control was obtained using flow control valves and mass flow meters (Sierra). Flow rates were recorded manually at the beginning and end of the sample, and most often at quarter points in between. Flow rates were used to determine sample volume based on sample collection times, and a correction to dry gas volume was made based on temperature. Volumes are reported at standard temperature (20°C) and pressure (1 atmosphere) in standard liters per minute (slpm). At the EERC all samples were in the ideal flowrate range (unless indicated in the sample logs) and generally flowrate varied by less than 3% (See RSD, Table III-5). Normal flowrate control was not maintained during Runs 9 and 10 when the ESP was malfunctioning and PHg loading was extremely high, which provided an unexpected ruggedness test.

The importance of flowrate to Hg determinations was evaluated during two quadruplicate flowrate tests during Runs 3 and 15. During these runs four parallel samples were collected that were identical except the collection flow rates was varied from 0.25 to 0.85 slpm. This represented flow rates that were two to four times the ideal FMSS range. Flowrate control and the analytical data for the flowrate ruggedness tests are summarized in Table III-7.

Several points will help in the interpretation of Table III-7. The precision of the flowrate control at the EERC is given as RSD at the top (section A). The analytical data during ruggedness tests is presented at the bottom (section B). T-Vol (column 3) is the temperature corrected dry gas volume and breakthrough is calculated as KCLB/KCLA (column 6).

Table III-7

A. Flowrate Control at the EERC

OVERALL FLOWRATE CONTROL AT EERC	
	COLLECTION FLOW RATE
	%RSD
Ruggedness , FR Tests	41.3%
Ruggedness , Temp Tests	0.6%
ESP malfunction, additional ruggedness	26.1%
Natural Gas No Tests	9.3%
Bitcoal No Tests	2.8%

Table III-7 (continued)
B. Flowrate and breakthrough during ruggedness tests

RUN 3 ANALYTICAL TRAP DATA

Run 3*	Flow Rate	PHg	KCLA	KCLB	ICA	T-Vol	$KCLB/KCLA$
	slpm	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter	%
FMSS-1-030800**	0.22	na	88.81	0.35	5.79	9.1	0.4%
FMSS-2-030800	0.44	11.15	178.9	1.366	12.04	23.6	0.8%
FMSS-4-030800***	0.44	na	234.2	0.776	16.56	35.1	0.3%
FMSS-3-030800	0.57	13.81	199.2	0.347	19.61	24.4	0.2%
mean	0.42	* heavy particulate noted all samples					
FR, %RSD	33.9%	** condensation noted					
		***failure to maintain 0.8 slpm, rapid drop in flowrate					

RUN 15 ANALYTICAL TRAP DATA

Run 15	Flow Rate	PHg	KCLA	KCLB	ICA	T-Vol	$KCLB/KCLA$
	slpm	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter	%
FMSS-9-031600	0.24	nc	467.1	7.10	123.7	14.6	1.5%
FMSS-10-031600	0.47	nc	836.7	7.67	193.9	26.3	0.9%
FMSS-11-031600	0.60	nc	1060	12.5	362.5	38.0	1.2%
FMSS-12-031600	0.88	nc	1353	132.7	534.8	50.8	9.8%
mean	0.55						
FR, %RSD	48.8%						

The results of the quad flowrate tests are reported elsewhere (see section VI). However, no significant trends were observed between Hg concentration and flowrate, even at the highest flowrate where the breakthrough from the KCLA trap to KCLB trap was greatest (See KCLB/KCLA in Table III-7). However, this flowrate is almost five times above the control range and despite the breakthrough, the precision and accuracy was still good.

III.G Isokinetic sampling tolerance limits

In order to keep it simple, the FMSS method is designed to be only semi-isokinetic. Thus, the inlet nozzle diameter is chosen to match the flue gas flow rate and the FMSS method flow rate range of 0.4 ± 0.1 slpm. During sampling, there is no adjustment for changing flue gas flow. This approach works because most coal flue gas has less than 5% PHg and the Hg is associated with sub-micron particles that will sample like a gas. Thus the MQO for semi-isokinetic sampling is $\pm 50\%$. If however there is reason to sample within a $\pm 10\%$ tolerance for isokinetics, this is certainly possible using standard EPA isokinetic stack sampling methods.

III.H Summary of Method Quality Objectives

Method quality objectives or MQOs were defined based on the performance of the FMSS method during the quality control experiments for the EERC and are summarized here.

Table III-8

A. MQOs for the quality control of future studies

	IDEAL FMSS Method	FMSS Method Result EERC	FMSS Method Future Studies MQO
Laboratory Replicate	$\pm 25\% \text{RPD}$	$< 20\% \text{RPD}$	$\pm 25\%$
SRM Spike Recoveries	$100 \pm 20\%$	$99 \pm 8\%$	$100 \pm 20\%$
Matrix Spike Recoveries	$100 \pm 20\%$	$100.9 \pm 5.7\%$	$100 \pm 20\%$
Temperature Control	$90 \pm 5^\circ \text{C}$ or $6\% \text{RPD}$	$90 \pm 5^\circ \text{C}$ or $6\% \text{RPD}$	$90 \pm 5^\circ \text{C}$ or $6\% \text{RPD}$
Flow Rate Control	$0.4 \pm 0.1 \text{slpm}$ or $25\% \text{RPD}$	$0.4 \pm 0.05 \text{slpm}$ or $2.1\% \text{RPD}$	$0.4 \pm 0.1 \text{slpm}$ or $25\% \text{RPD}$

Several points may help in the interpretation of Table III-8. The numerous quality control experiments are listed in column 1. The Ideal Result (column 2) is the performance level required to satisfy the current needs of the Hg fluegas sampling community. This need was estimated as 5 ug/m^3 levels of Hg in fluegas with a precision or RSD of $\pm 20\%$ (or $0.5 \pm 0.1 \text{ ug/m}^3$ at $n=3$) and of better than 90% for THg and $\% \text{Hg}^{+2}$. The FMSS Method performance result at the EERC is listed in column 3 and the MQO for future studies as it applies to Hg speciation in combustion fluegas matrices is listed in column 4.

IV. FMSS Method Detection Limits

Method detection limits or MDLs were estimated in three ways and were based on the analysis of A) Eight field blanks; B) Eight samples collected at near blank levels (Run 0) and C) Eight Low-level, Mid-level, and High level samples.

MDL estimates by each method are discussed below and were based on the data in Table IV-1.

Table IV-1

Data for FMSS MDL estimates

A. MDL DATA AT THE EERC							
Analysis of Eight Each of	MDL method			Mean PHg ug/m ³	Mean Hg ⁰ ug/m ³	Mean Hg ⁺² ug/m ³	Mean THg ug/m ³
MEAN							
EERC Field Blanks	A		mean	0.0022	0.0154	0.0028	0.0182
EERC Run 0	B,C	Low level	mean	NA	0.0215	0.0063	0.0277
EERC Run 4	C	mid level	mean	0.0070	0.303	8.36	8.66
EERC Run 16	C	high level	mean	NA	8.74	33.1	41.8
STDEV							
EERC Field Blanks	A	n=8	stdev	0.0019	0.006	0.003	0.007
EERC Run 0	B,C	n=8	stdev	NA	0.011	0.009	0.017
EERC Run 4	C	n=6	stdev	0.005	0.087	0.67	0.68
EERC Run 16	C	n=8	stdev	NA	1.02	1.86	2.69
B. MDL C - GRAPH DATA							
MDL-C CALCULATION				MDL PHg ug/m ³	MDL Hg ⁰ ug/m ³	MDL Hg ⁺² ug/m ³	MDL THg ug/m ³
FMSS GRAPH DATA:							
Mean(x-axis) vs Stdev(y-axis)		C	slope	NA	0.1138	0.0541	0.0361
Mean(x-axis) vs Stdev(y-axis)		C	y-int	NA	0.0301	0.0969	0.0688
Mean(x-axis) vs Stdev(y-axis)		C	R ²	NA	0.9984	0.9871	0.9980

Method A MDLs were generated based on 3σ of the field blanks (see Table III-2).

Method B MDLs were generated based on 3σ of Run 0. Run 0 was burning natural gas with SO₂ and HCl without Hg spiking.

Method C MDLs were generated based on the MDL plots of mean Hg concentrations (x-axis) verses standard deviation of the means (y-axis) for the low level, mid-level and high-level replicate of Run 0, 4 and 16. The MDL plots for method C are shown in Figure IV-1.

Figure IV-1

Hg concentration versus standard deviation for Runs 0, 4 and 16 at the EERC

MDL estimates are summarized in Table IV-2.

Table IV-2
Summary of the FMSS MDL estimates at the EERC

FMSS MDL ESTIMATES RESULT SUMMARY AT THE EERC	FMSS MDL PHg ug/m ³	FMSS MDL Hg ⁰ ug/m ³	FMSS MDL Hg ⁺² ug/m ³	FMSS MDL THg ug/m ³
MDL-A	0.0058	0.019	0.009	0.020
MDL-B	NA	0.032	0.027	0.051
MDL-C	NA	0.090	0.291	0.206
lowest estimate	0.006	0.019	0.009	0.020
highest estimate	0.006	0.090	0.291	0.206
FMSS MDL ESTIMATE*	0.006	0.026	0.018	0.035

* The best MDL estimate was based on the MDL-A and MDL-B result

There was good agreement between the MDL estimated by method A and B. However, MDL estimates by method C were three to ten times higher¹. Based on the higher uncertainties associated with this method, the best estimate for the FMSS MDL was determined by the mean of method A and method B (see Table IV-2).

In conclusion, FMSS MDL estimates at the EERC were (in ug/m³) 0.006 for PHg, 0.026 for Hg⁰, 0.018 for Hg⁺², and 0.035 for THg. Based on this result, the MQO of future studies (in ug/m³) was defined as 0.01 for PHg, 0.03 for Hg⁰, 0.02 for Hg⁺² and 0.04 for THg.

¹ It should be noted that even the higher MDL estimates of method C (See Table IV-1) are quite good and adequate for understanding the apportionment of Hg in fluegas.

V. Precision, Accuracy, and Bias

The precision accuracy and bias of the FMSS method for the full matrix of 16 tests at the EERC facility are presented in this section. The combustion flue gases measured during these runs were burning various fuels, had two types of particulate control devices, and were selectively spiked with Hg. The precision, accuracy and bias of the replicates with no ruggedness testing are discussed in detail here. The result from the ruggedness tests is included in the tables and the figures presented here, but are discussed later (see ruggedness tests, see Section VI).

V.A Precision

Replicate samples at the EERC provided the means to measure the precision of the FMSS method under real conditions. FMSS and OH precision was calculated for each run using the following formulas. For duplicate samples (n=2), a relative percent difference (RPD) was calculated from the absolute difference of the means (as a percentage) from:

$$RPD = \left| \text{Run 1}_{\text{FMSS}} - \text{Run 2}_{\text{FMSS}} \right| / \text{average run 1 \& 2}_{\text{FMSS}}$$

For replicate samples where n>2 then a relative standard deviation (RSD) was calculated from the mean and standard deviation of the mean as a percentage as:

$$RSD = \text{STDEV} / \text{MEAN}$$

The accuracy of the FMSS result is reported relative to the OH method and expressed as a percent recovery of the means of the FMSS and OH methods as

$$\% \text{Recovery} = \text{mean}_{\text{FMSS}} / \text{mean}_{\text{OH}}$$

The mean Hg concentrations and precision of the replicates are presented in Table V-1.

A key for the fluegas conditions or ruggedness test and number of replicates (n) averaged for each run is shown below.

Several points may help in the interpretation of Tables V-1 and V-2. Hg concentrations, precision and accuracy are listed by species in Table V-1. FMSS and OH precision summaries are listed by the EERC run # in Table V-2. Hg concentrations were the means of a number of replicates. FMSS replicates were at least duplicates, and frequently quadruplicates, while OH method replicates were always duplicates except during Runs 1 and 2, when n was one and no precision or accuracy is reported for these runs.

Table V-1
CONDITIONS AT THE EERC

CONDITIONS AT THE EERC			TESTS	number of replicates	fraction as vapor
Date-Starttime					
MDL CALCUALTIONS					
EERC Field Blanks		Field Blanks		n=8	
EERC Run 0	3/6/00-3/7/00	Natural Gas no spikes		n=8	
EERC Run 4	3/8/2000-18:37	Bitcoal ESP with spikes	Quads No Test	n=6	0.98
EERC Run 16	3/17/2000-10:06	Bitcoal BH with spikes	Quads No Test	n=8	1.00
				mean	0.99
				stdev	0.01
QUAD RUGGEDNESS TESTS					
EERC Run 3*	3/8/2000-11:37	Bit-coal ESP no spikes	Flowrate Test	n=4	0.89
EERC Run 12*	3/15/2000-13:55	Bitcoal BH no spikes	Temperature Test	n=4	1.00
EERC Run 13*	3/16/2000-9:23	Bitcoal BH with spikes	Temperature Test	n=4	1.00
EERC Run 15*	3/16/2000-16:13	Bitcoal - BH with spikes	Flowrate Test	n=4	1.00
				mean	0.97
				stdev	0.06
REPLICATES NATURAL GAS					
EERC Run 1	3/7/2000-13:35	Natural Gas	Hg ⁰ spikes	n=3	1.00
EERC Run 2	3/7/2000-16:40	Natural Gas	Hg ⁺² spikes	n=3	0.86
				mean	0.93
				stdev	0.09
REPLICATES COAL FLUEGAS MATRICES					
EERC Run 5	3/10/2000-16:24	Bitcoal ESP no spikes		n=2	0.97
EERC Run 6	3/13/2000-9:20	Bitcoal ESP with spikes		n=2	0.98
EERC Run 7	3/13/2000-14:50	Bitcoal ESP with spikes		n=2	0.99
EERC Run 8	3/13/2000-18:53	Bitcoal ESP with spikes		n=2	0.98
EERC Run 9**	3/14/2000-10:30	Bitcoal ESP with spikes	Heavy Particulate	n=3	0.93
EERC Run 10**	3/14/2000-16:00	Bitcoal ESP with spikes	Heavy Particulate	n=3	0.70
EERC Run 11	3/15/2000-9:40	Bitcoal BH no spikes		n=3	1.00
EERC Run 14	3/16/2000-12:31	Bitcoal BH with spikes		n=3	1.00
				mean	0.87
			*Ruggedness	stdev	0.26
**ESP malfunction, additional ruggedness					

Table V-1

RESULTS BY SPECIES

A. Particulate Hg at EERC

PHg RESULT	number (n)	FMSS Mean PHg ug/m ³	FMSS Precision PHg RSD or RPD	Accuracy PHg %Rec	OH Mean PHg ug/m ³	OH Precision PHg RPD
ID	replicates					
R4	n=6	0.007	71.3%	NA	0.255	35.29%
R16	n=8	NA	NA	NA	0.025	40.00%
R3*	n=4	0.516	12.8%	30.4%	1.700	NA
R12*	n=4	NA	NA	NA	0.010	0.00%
R13*	n=4	NA	NA	NA	0.020	0.00%
R15*	n=4	NA	NA	NA	0.040	50.00%
R1	n=3	NA	NA	NA	0.020	NA
R2	n=3	NA	NA	NA	1.940	NA
R5	n=2	0.079	53.5%	20.6%	0.385	7.79%
R6	n=2	0.599	NA	105.1%	0.570	31.6%
R7	n=2	0.104	41.0%	34.6%	0.300	6.67%
R8	n=2	0.914	NA	145.0%	0.630	9.52%
R9*	n=3	3.721	31.6%	91.9%	4.05	140%
R10*	n=3	8.140	38.4%	91.6%	8.89	30.6%
R11	n=3	NA	NA	NA	0.040	100%
R14	n=3	NA	NA	NA	0.020	NA

B. Elemental Hg at the EERC

Hg ⁰ RESULT	number (n)	FMSS Mean Hg ⁰ ug/m ³	FMSS Precision Hg ⁰ RSD or RPD	Accuracy Hg ⁰ %Rec	OH Mean Hg ⁰ ug/m ³	OH Precision Hg ⁰ RPD
ID	replicates					
R4	n=6	0.303	28.8%	65.2%	0.465	6.45%
R16	n=8	8.74	11.7%	98.1%	8.910	28.5%
R3*	n=4	0.579	31.3%	108.3%	0.535	9.35%
R12*	n=4	0.142	36.3%	39.6%	0.360	5.56%
R13*	n=4	7.78	11.2%	92.0%	8.45	5.21%
R15*	n=4	8.96	15.2%	95.5%	9.38	22.0%
R1	n=3	12.82	3.4%	NA	10.1	NA
R2	n=3	2.10	14.2%	NA	2.11	NA
R5	n=2	0.347	36.4%	75.4%	0.460	0.00%
R6	n=2	4.14	31.6%	70.8%	5.85	40.2%
R7	n=2	7.15	14.3%	105.0%	6.81	4.85%
R8	n=2	5.32	NA	69.9%	7.61	1.31%
R9*	n=3	0.618	18.5%	56.9%	1.09	32.3%
R10*	n=3	0.813	17.5%	47.4%	1.72	15.7%
R11	n=3	0.080	17.1%	18.3%	0.435	16.1%
R14	n=3	9.81	10.5%	107.6%	9.120	12.7%

A. Hg⁺² at the EERC

Hg ⁺² RESULT		FMSS Mean Hg ⁺² ug/m ³	FMSS Precision Hg ⁺² RSD or RPD	Accuracy Hg ⁺² %Rec	OH Mean Hg ⁺² ug/m ³	OH Precision Hg ⁺² RPD
ID	number (n) replicates					
R4	n=6	8.36	7.98%	106.0%	7.88	9.90%
R16	n=8	33.1	5.61%	87.7%	37.7	5.94%
R3*	n=4	7.49	9.93%	86.4%	8.68	7.03%
R12*	n=4	8.84	7.35%	87.8%	10.1	8.74%
R13*	n=4	28.5	7.02%	89.5%	31.85	11.7%
R15*	n=4	30.5	6.86%	103%	29.56	14.1%
R1	n=3	0.023	70.15%	NA	0.230	NA
R2	n=3	14.14	2.65%	NA	6.380	NA
R5	n=2	7.86	9.09%	115.4%	6.81	5.29%
R6	n=2	32.5	1.27%	116.1%	28.0	17.5%
R7	n=2	26.3	11.32%	91.5%	28.7	27.1%
R8	n=2	26.7	2.00%	98.1%	27.2	24.7%
R9*	n=3	23.8	12.01%	104.0%	22.8	11.0%
R10*	n=3	15.6	26.18%	72.1%	21.6	18.0%
R11	n=3	9.45	8.12%	92.3%	10.2	8.80%
R14	n=3	28.3	16.32%	90.8%	31.1	8.16%

B. THg at the EERC

THg RESULT		FMSS Mean THg ug/m ³	FMSS Precision THg RSD or RPD	Accuracy THg %Rec	OH Mean THg ug/m ³	OH Precision THg RPD
ID	number (n) replicates					
R4	n=6	8.66	7.88%	103.8%	8.35	8.99%
R16	n=8	41.8	6.44%	89.7%	46.6	10.3%
R3*	n=4	8.07	11.2%	87.6%	9.21	6.08%
R12*	n=4	8.99	7.73%	86.1%	10.4	8.63%
R13*	n=4	36.3	6.15%	90.0%	40.3	10.37%
R15*	n=4	39.5	3.27%	101.4%	38.9	16.0%
R1	n=3	12.8	3.34%	NA	10.3	NA
R2	n=3	16.2	0.48%	NA	8.49	NA
R5	n=2	8.20	7.16%	112.9%	7.27	4.95%
R6	n=2	36.6	4.70%	108.3%	33.8	7.48%
R7	n=2	33.4	12.0%	94.0%	35.5	21.0%
R8	n=2	32.0	13.3%	91.9%	34.8	19.6%
R9*	n=3	24.4	12.2%	87.1%	28.0	30.6%
R10*	n=3	16.4	25.7%	70.3%	23.4	17.8%
R11	n=3	9.53	8.20%	89.3%	10.7	9.10%
R14	n=3	38.1	14.8%	94.6%	40.3	9.19%

C. Speciation at the EERC

%Hg ⁺² Result		FMSS Mean %Hg ⁺²	FMSS Precision %Hg ⁺² RSD or RPD	Accuracy %Hg ⁺² %rec	OH Mean %Hg ⁺²	OH Precision %Hg ⁺² RPD
ID	number (n) replicates					
R4	n=6	96.5%	1.1%	102.2%	94.4%	0.91%
R16	n=8	79.3%	1.9%	97.9%	81.0%	4.3%
R3*	n=4	93.2%	1.4%	99.0%	94.2%	1.0%
R12*	n=4	98.4%	0.5%	102.0%	96.5%	0.1%
R13*	n=4	78.6%	2.84%	99.4%	79.0%	1.4%
R15*	n=4	77.3%	4.8%	101.7%	76.0%	1.9%
R1**	n=3	0.184%	0.14%	NA	2.2%	NA
R2	n=3	87.0%	2.18%	NA	75.1%	NA
R5	n=2	95.7%	1.93%	102.2%	93.7%	0.33%
R6	n=2	88.7%	3.43%	107.5%	82.6%	10.0%
R7	n=2	78.6%	0.65%	97.6%	80.6%	6.2%
R8	n=2	83.7%	11.3%	107.4%	77.9%	5.2%
R9*	n=3	97.5%	0.16%	117.6%	82.9%	19.7%
R10*	n=3	95.0%	0.55%	102.5%	92.7%	0.2%
R11	n=3	99.2%	0.08%	103.4%	95.9%	0.30%
R14	n=3	74.1%	1.75%	95.8%	77.4%	1.04%

**Run 1 had Hg⁰ spikes only and had significant amounts of Hg⁰ (99.816%).
and the precision for Run 1 is as %Hgo.

Table V-2

FMSS AND OH PRECISION RESULTS BY RUN

A. Hg Species Concentration Result

FMSS MEAN	FMSS Mean	FMSS Mean	FMSS Mean	FMSS Mean	OH MEAN	OH Mean	OH Mean	OH Mean	OH Mean
ID	PHg ug/m3	Hgo ug/m3	Hg+2 ug/m3	THg ug/m3	ID	PHg ug/m3	Hgo ug/m3	Hg+2 ug/m3	THg ug/m3
R4	0.0070	0.303	8.36	8.66	R4	0.255	0.465	7.88	8.35
R16	NA	8.74	33.1	41.8	R16	0.025	8.91	37.7	46.6
R3*	0.516	0.58	7.49	8.07	R3*	1.70	0.535	8.68	9.21
R12*	NA	0.14	8.84	8.99	R12*	0.010	0.360	10.1	10.4
R13*	NA	7.78	28.5	36.3	R13*	0.02	8.45	31.9	40.3
R15*	NA	8.96	30.5	39.5	R15*	0.04	9.38	29.6	38.9
R1	NA	12.8	0.0233	12.8	R1	0.02	10.05	0.23	10.3
R2	NA	2.10	14.1	16.2	R2	1.94	2.11	6.38	8.49
R5	0.079	0.347	7.86	8.20	R5	0.385	0.46	6.81	7.27
R6	0.599	4.14	32.5	36.6	R6	0.570	5.85	28.0	33.8
R7	0.104	7.15	26.3	33.4	R7	0.300	6.81	28.7	35.5
R8	0.91	5.32	26.7	32.0	R8	0.63	7.61	27.2	34.8
R9*	3.72	0.618	23.8	24.4	R9*	4.05	1.09	22.8	28.0
R10*	8.14	0.81	15.6	16.4	R10*	8.89	1.72	21.6	23.4
R11	NA	0.080	9.45	9.53	R11	0.040	0.435	10.2	10.7
R14	NA	9.8	28.3	38.1	R14	0.02	9.12	31.1	40.3

B. Mean Precision Result

PRECISION RESULT	FMSS	FMSS	FMSS	FMSS	PRECISION RESULT	OH	OH	OH	OH
ID	PHg RSD or RPD	Hgo RSD or RPD	Hg+2 RSD or RPD	THg RSD or RPD	ID	[pHg] RPD	[Hgo] RPD	[Hg+2] RPD	THg RPD
R4	71.3%	28.8%	7.98%	7.88%	R4	35.3%	6.45%	9.90%	8.99%
R16	NA	11.7%	5.61%	6.44%	R16	40.0%	28.5%	5.94%	10.3%
R3*	12.8%	31.3%	9.93%	11.2%	R3*	NA	9.35%	7.03%	6.08%
R12*	NA	36.3%	7.35%	7.73%	R12*	0.0%	5.56%	8.74%	8.63%
R13*	NA	11.2%	7.02%	6.15%	R13*	0.0%	5.21%	11.7%	10.4%
R15*	NA	15.2%	6.86%	3.27%	R15*	50.0%	22.0%	14.1%	16.0%
R1	NA	3.45%	70.2%	3.34%	R1	NA	NA	NA	NA
R2	NA	14.2%	2.65%	0.48%	R2	NA	NA	NA	NA
R5	53.5%	36.4%	9.09%	7.16%	R5	7.8%	0.00%	5.29%	5.0%
R6	NA	31.6%	1.27%	4.70%	R6	31.6%	40.2%	17.5%	7.5%
R7	41.0%	14.3%	11.3%	12.0%	R7	6.7%	4.85%	27.1%	21.0%
R8	NA	NA	2.0%	13.3%	R8	9.5%	1.3%	24.7%	19.6%
R9*	31.6%	18.5%	12.0%	12.2%	R9*	140.2%	32.3%	11.0%	30.6%
R10*	38.4%	17.5%	26.2%	25.7%	R10*	30.6%	15.7%	18.0%	17.8%
R11	NA	17.1%	8.12%	8.20%	R11	100.0%	16.1%	8.80%	9.10%
R14	NA	10.5%	16.3%	14.8%	R14	NA	12.7%	8.16%	9.19%

The first significant result from the tables is that the precision of either method did not vary much over the course of the 16 runs. The precision was generally similar for both the FMSS and simultaneous OH methods. The precision was worst at the lowest concentrations especially near method detection limits (For FMSS method MDLs, see section IV). Hg^0 concentrations during many of the runs were generally low relative to much larger concentrations of Hg^{+2} and in a few cases near the MDL (Runs 3, 10, 11, 12). Under these conditions, field blank and MDL considerations for both the FMSS and OH methods are more significant. In general PHg levels were also low, and in a few cases near MDL. However, in the few instances when the Hg on particulate flyash was significant, the precision of the PHg and other species concentrations remained quite good. This can be seen by the precision result for Run 9 and Run 10 (see Table V-2) during which time the ESP pollution control device was possibly malfunctioning.

The mean precision of the FMSS and OH replicates with and without testing by either method is summarized in Table V-3.

Table V-3
Precision Summary at the EERC

	PRECISION					PRECISION				
	RSD or RPD	FMSS PHg	FMSS Hg^0	FMSS Hg^{+2}	FMSS THg	RPD	OH [pHg]	OH [Hg^0]	OH [Hg^{+2}]	OH THg
Quads No Tests	mean	71.3%	20.2%	6.80%	0.072	mean	37.6%	17.5%	7.92%	9.62%
	stdev	NA	12.1%	1.7%	0.010	stdev	3.3%	15.6%	2.8%	0.90%
Quad Ruggedness Tests*	mean	12.8%	23.5%	7.79%	0.071	mean	16.7%	10.5%	10.4%	10.3%
	stdev	NA	12.2%	1.4%	0.033	stdev	28.9%	7.9%	3.1%	4.2%
Replicates Natural Gas	mean	NA	8.81%	36.4%	1.91%	mean	NA	NA	NA	NA
	stdev	NA	0.076	0.5	0.020	stdev	NA	NA	NA	NA
Additional Ruggedness **High PHg	mean	35.0%	18.0%	19.1%	18.9%	mean	85.4%	24.0%	14.5%	24.2%
	stdev	4.8%	0.7%	10.0%	9.6%	stdev	77.5%	11.7%	4.9%	9.0%
Replicates No Ruggedness	mean	47.3%	22.0%	8.02%	10.0%	mean	31.1%	12.5%	15.3%	11.9%
Duplicates and Triplicates	stdev	8.9%	11.3%	5.7%	3.9%	stdev	39.9%	15.0%	9.2%	6.7%

A significant result is that the precision of the duplicates by either the FMSS method or the OH method was fairly similar, except during the ruggedness tests when the precision of the Hg^0 was worse. Again, however, the Hg^0 levels were often quite low during these runs making field blank considerations more significant.

Although it is difficult to draw significant conclusions by comparing precision RSD ($n > 2$) and RPD ($n = 2$), it is possible to make general comparisons. One result is that in general the precision of the FMSS duplicates, triplicates, quadruplicates were quite similar. Also, the precision of the quadruplicates with no testing was similar to the precision during the FMSS ruggedness tests. Based on the replicates with no additional ruggedness at the EERC (Table V-3), it is concluded that the mean precision of the FMSS method as determined under real conditions for duplicates and triplicates was $\pm 47\%$ for PHg, $\pm 22\%$ for Hg^0 , $\pm 8\%$ for Hg^{+2} , and $\pm 10\%$ for THg. The mean precision of the FMSS method was $\pm 6.5\%$ for Hg^{+2} or the percent oxidized Hg of the total (THg+PHg).

The FMSS precision can best be seen from the graphs Figure V-1. These graphs include A) FMSS Precision versus Hg concentration and the precision of both the FMSS and OH methods for B) THg and C) %Hg⁺² for each run at the EERC.

Figure V-1 A.

FMSS Precision versus Hg concentration

Figure V-1 B.

Precision of THg verses EERC run for different fluegas conditions.

Figure V-1 C.

Precision of Hg⁺² verses EERC run for different fluegas conditions

The significant result as evidenced in precision graphs is that FMSS precision was almost always better than 20%. This was true despite the ruggedness testing and concentrations near MDL. The worst precision of 25% was during Runs 9 and 19 when the ESP was malfunctioning and particulate loads were abnormally high. However, during these same runs the OH precision was also worse.

V.B Accuracy – Comparison to established OH method.

The accuracy of the means of the FMSS and OH methods can best be seen from Figure V-2.

Figure V-2.

FMSS and OH comparison of the means

The mean PHg, Hg⁰ and Hg⁺² species concentrations are combined in a single bar for both the FMSS and OH method replicates. The bar on the left represents the FMSS method result; the bar on the right represents a separate determination by the OH method. The height of the bar represents the total Hg in the sample and is the sum of the total vapor phase Hg⁰ and Hg⁺² and any Hg collecting in the particulate phase (or THg+PHg). That the THg+PHg result by the FMSS and OH method was sometimes higher and sometimes lower, this indicated that slight differences between the methods exist, but that there was generally no significant biases. For a complete discussion of bias see section V-C.

Good agreement of the means was observed almost all cases. The accuracy was worst during Runs 9 and 10 which had abnormally high particulate (3-10ug/m³) and for Hg⁰ concentrations whose concentrations were generally quite low, and made in the presence of much larger concentrations of Hg⁺². Under these conditions, field blank and MDL considerations for both the FMSS and OH methods are more significant.

The accuracy of the FMSS result relative to the OH method was presented for each species (PHg, Hg⁰, Hg⁺²) concentration for THg and %Hg⁺² in Table V-1. Presented here is the accuracy expressed for comparison purposes as the means of the accuracy of the replicates, with and without additional ruggedness. This result is shown in Table V-4.

Table V-4
Accuracy Summary at the EERC

ACCURACY SUMMARY		PHg % rec	[Hg ⁰] % rec	[Hg ⁺²] % rec	THg % rec	% Hg ⁺² % rec
Quads No Tests	mean	NA	81.6%	96.9%	96.7%	100.1%
	stdev	NA	23.3%	12.9%	9.9%	3.0%
Quad Ruggedness Tests*	mean	30.4%	83.9%	91.7%	91.3%	100.5%
	stdev	NA	30.3%	7.8%	6.9%	1.54%
Replicates Natural Gas	mean	NA	NA	NA	NA	NA
	stdev	NA	NA	NA	NA	NA
Additional Ruggedness **High PHg	mean	91.7%	52.2%	88.0%	78.7%	110.1%
	stdev	0.2%	6.7%	22.6%	11.9%	10.7%
Replicates No Ruggedness	mean	76.3%	74.5%	100.7%	98.5%	102.3%
	stdev	58.9%	32.3%	11.9%	9.6%	4.9%

On average, the accuracy (as %Rec= mean_{FMSS} / mean_{OH}) of the FMSS with the OH method was 100±3% for Hg⁺², THg, and %Hg⁺²; and 100±25% for Hg⁰ and PHg. However, the result for Hg⁰ and PHg included many cases when the concentration was quite low.

The accuracy of the FMSS relative to the OH method can best be seen in Figure V-3. Figure V-3 shows the accuracy (y-axis) relative to Hg concentrations (x-axis) and includes data from all 16 runs at the EERC, including the ruggedness tests.

Figure V-3

FMSS-OH % recovery verses mean Hg concentration at the EERC.

When values near the MDL are not considered, the accuracy of the FMSS was better than $\pm 20\%$ for all species for the range of conditions in Table V-1. This was true even for the ruggedness tests, including those with abnormally high particulate levels.

V. Bias – Comparison of all FMSS and OH results.

The bias between the FMSS and OH methods for the measurement of Hg species concentrations is summarized in Table V-5. This bias was determined under real conditions at the EERC for the whole range of fluegas conditions (see condition, Table V-1).

Table V-5

FMSS –OH Bias Summary at the EERC

BIAS Graph Data	PHg	Hg ⁰	Hg ⁺²	THg	THg+PHg	%Hg ⁺²
Slope or Bias	1.075	0.839	1.070	1.120	1.118	84.4%
y-int (ug/m ³)	0.07	0.9177	-1.61	-3.127	-2.979	51.0%
R ²	0.9953	0.9143	0.93	0.9602	0.9582	85.1%
FMSS Error Bar	47.3%	22.0%	8.02%	10.0%	10.0%	4.0%
OH Error Bar	31.1%	12.5%	15.3%	11.9%	11.9%	3.5%

The bias was calculated from the slopes of the plots of the mean Hg species concentration for the FMSS method (X-axis) with the simultaneous result by the OH method (Y-axis). The plots for %Hg⁺², THg, Hg⁺² and Hg⁰ can be seen in Figure V-4. The error bars in the figure (shown in Table V-5) are the precision of the means for the FMSS and OH methods for replicates with no ruggedness. A slope of 1.00 indicates perfect agreement between the methods and no bias. The figure includes data for the entire 16 Runs at the EERC, including several results near the method detection limits and results from runs that underwent additional ruggedness testing.

Figure V-4 A

Figure V-4 B

Figure V-4 C

Figure V-4 D

Figure V-4 E

VI. Ruggedness Testing at the EERC

Tests for FMSS ruggedness were an important part of the PBMS. A detailed discussion of each ruggedness test is provided in the following section.

VI. A Quadruplicate Temperature Tests

The importance of the sampling probe temperature to Hg determination was investigated during two quadruplicate temperature tests (Runs 12 and 13). During these runs, four parallel samples were collected that were identical except that probe temperature was varied from 75 to 105°C. This represented ruggedness that is one to three times the ideal FMSS range.

The precision and accuracy results of the temperature tests are shown in Table VI-1.

Table VI-1

Precision and Accuracy during temperature tests

RUN 12 TEMPERATURE TEST						
FMSS	Temp	PHg	Hg ⁰	Hg ⁺²	THg	%Hg ⁺²
Sample ID	C	ug/m ³	ug/m ³	ug/m ³	ug/m ³	
FMS-5-031500	75.67	nc	0.101	8.39	8.49	98.8%
FMS-6-031500	88.44	nc	0.209	9.31	9.52	97.8%
FMS-7-031500	96.41	nc	0.102	8.19	8.29	98.8%
FMS-8-031500	105.7	nc	0.157	9.49	9.65	98.4%
FMSS MEAN	91.6	NA	0.158	8.85	9.00	98.3%
STDEV	12.7	NA	0.052	0.65	0.69	0.46%
% RSD	13.9%	NA	32.7%	7.3%	7.7%	0.46%
OH MEAN		0.01	0.36	10.1	10.4	96.4%
OH %RPD		0.0%	5.6%	8.74%	8.71%	0.03%
ACCURACY FMSS-OH (%REC)		NA	43.8%	88%	86%	101.9%
RUN 13 TEMPERATURE TEST						
FMSS	Temp	PHg	Hg ⁰	Hg ⁺²	THg	%Hg ⁺²
Sample ID	C	ug/m ³	ug/m ³	ug/m ³	ug/m ³	
FMS-1-031600	75.28	nc	8.10	31.4	39.5	79.5%
FMS-2-031600	86.67	nc	8.33	27.5	35.9	76.8%
FMS-3-031600	96.56	nc	6.47	28.1	34.6	81.3%
FMS-4-031600	105.9	nc	8.20	27.0	35.2	76.7%
FMSS MEAN	91.11	NA	7.78	28.50	36.28	78.6%
STDEV	13.17	NA	0.87	2.00	2.23	2.23%
% RSD	14.5%	NA	11.2%	7.0%	6.1%	2.8%
OH MEAN		0.02	8.45	31.9	40.3	79.0%
OH %RPD		0.0%	5.2%	11.7%	10.4%	1.38%
ACCURACY FMSS-OH (%REC)		NA	92.0%	89%	90%	99.5%

The results of the temperature tests can perhaps best be seen in Figures VI-1.

Figure VI-1 A

Quad Temperature Test Run 12

Temperature during Run 12 samples represented twice to three times the ideal range (top figure). Despite the additional ruggedness, the precision of the %Hg⁺² FMSS method (error bar on point) was similar to that of the OH method (error bars on line) and the accuracy of the %Hg⁺² was 102%. The worst precision and accuracy was for Hg⁰ (see RSD, Table VI-1) when Hg was at near blank levels.

Figure V-1 B.

Quad Temperature Test Run 13

Temperatures during Run 13 ranged from 75 to 105 °C, about three times above the control range. However, despite the additional ruggedness, the precision of the FMSS method (error bar on point) was similar to that of the OH method (error bar on line) and the accuracy of the %Hg⁺² was 99.4%.

VI. Quadruplicate Flow Rate Tests

The importance of flowrate to Hg determinations was evaluated during two quadruplicate flowrate ruggedness tests during Runs 3 and 15. During these runs four parallel samples were collected that were identical except the collection flow rates was varied from 0.25 to 0.85 slpm.

The precision and accuracy results of the flowrate tests are shown in Table VI-2.

Table VI-2

Precision and Accuracy during flowrate tests

RUN 3 FLOWRATE TEST						
FMSS	Flow Rate	PHg	Hg ⁰	Hg ⁺²	THg	%Hg ⁺²
Sample ID	slpm	ug/m ³	ug/m ³	ug/m ³	ug/m ³	
FMSS-1-030800**	0.22	na	0.622	9.82	10.44	94.0%
FMSS-2-030800	0.44	0.47	0.494	7.62	8.12	93.9%
FMSS-4-030800**	0.44	na	0.457	6.69	7.15	93.6%
FMSS-3-030800	0.57	0.56	0.787	8.16	8.95	91.2%
MEAN (including **)	0.417	0.516	0.590	8.075	8.665	93.2%
(n=4) STDEV	0.141	0.066	0.149	1.313	1.396	1.3%
% RSD	33.9%	12.8%	25.3%	16.3%	16.1%	1.4%
OH MEAN						
		1.7	0.535	8.7	10.1	86.7%
%RPD						
		NA	9.3%	7.03%	11.3%	18.3%
ACCURACY FMSS-OH (%REC)*						
		30.4%	110%	93.1%	86.1%	107.5%
RUN 15 FLOWRATE TEST						
FMSS	Flow Rate	PHg	Hg ⁰	Hg ⁺²	THg	%Hg ⁺²
Sample ID	slpm	ug/m ³	ug/m ³	ug/m ³	ug/m ³	
FMSS-9-031600	0.24	na	8.44	32.41	40.85	79.3%
FMSS-10-031600	0.47	na	7.36	32.14	39.51	81.4%
FMSS-11-031600	0.60	na	9.52	28.21	37.73	74.8%
FMSS-12-031600	0.88	na	10.5	29.24	39.76	73.6%
FMSS MEAN	0.548	NA	8.960	30.501	39.462	0.773
STDEV	0.267	NA	1.360	2.093	1.291	0.037
% RSD	48.8%	NA	15.2%	6.9%	3.3%	4.8%
OH MEAN						
		0.04	9.38	29.6	39.0	75.9%
OH %RPD						
		50.0%	22%	14.1%	15.9%	1.83%
ACCURACY FMSS-OH (%REC)						
		NA	96%	103%	101%	101.8%

The results of the flowrate tests can perhaps best be seen in Figures VI-2.

Figure VI-2 A

Quad Flowrate Run 3

Flowrates for Run 3 ranged from 0.2 to 0.6 slpm and represented twice the ideal range. Run 3 had additional ruggedness. Heavy particulate noted on all samples, condensation was noted on the sample with the lowest flowrate, indicating possible failure to maintain temperature control, and attempts to maintain the 0.8 slpm flowrate failed thus spanning a whole range of flowrates in a single sample. FMSS precision (error bar) was similar to OH precision (error bars of the line). The accuracy relative to the OH result was worse than normal but still better than 10% with a percent recovery (relative to OH) for %Hg⁺² of 107%.

Figure VI-2 B

Quad Flowrate Test Run 15

Flowrates during Run 15 ranged from 0.8 to 0.2 slpm and represented four times the ideal range. Despite the ruggedness, the precision of the FMSS method was similar to the OH method, and there was good agreement between the two methods. The accuracy of $\%Hg^{+2}$ was 102%.

The actual control used at the EERC (in RSD) during normal operation of the FMSS method was defined earlier and was $\pm 6\%$ for temperature and $\pm 3\%$ for flowrate. During ruggedness tests variations in temperature were $\sim 14\%$ and in flowrate were $\sim 30\text{-}50\%$ (See Table VI-1 and VI-2). This is roughly two to four times the normal control range.

Despite the additional ruggedness, the precision and accuracy of the FMSS remained quite good and was similar to that of the OH methods duplicates. And no significant trends were observed between the measured Hg concentrations and flowrate or temperature.

In conclusion, based on the good result at the EERC for FMSS precision and accuracy during the ruggedness tests, the FMSS method is expected to be very reliable method for measuring total and speciated mercury in coal fluegas matrices.

VII. Interferences

The samples collected at both the EERC and the DOE reflect matrices of natural gas and coal fluegas matrices with SO_2 and HCl. As such, the results of this study include interferences from SO_2 , NO_x , HCl, Flyash and other components of fluegas and were an integral part of the experiments.

VIII. Matrix Suitability

The application of the FMSS to measurements of total mercury has in the past (see historical studies, section II) shown more than adequate performance. This study was applied at the EERC, mainly for coal matrices, for the range of conditions presented in Table VIII-1.

Table VIII-1

Range of applicable matrices tested under real conditions at the EERC

HG CONCENTRATIONS STUDIED AT EERC							
Overall range all runs*	PHg ug/m ³	Hg ⁰ ug/m ³	Hg ⁺² ug/m ³	THg (v) ug/m ³	THg+PHg ug/m ⁴	fraction as vapor	%Hg ⁺² ***
MDL**	0.006	0.026	0.018	0.035			
lowest measured [Hg]	0.010	0.251	0.127	7.737	7.969	0.70	1.21%
highest	8.5	11.4	35.4	44.2	44.2	1.00	97.5%
mean of range	1.03	3.98	16.9	21.0	24.6	0.96	81.4%
stdev of range (mean +/-1 stdev)	2.17	4.11	12.2	15.0	13.7	0.08	22.8%
lower limit of applicable range	0.01	0.03	4.73	6.01	10.96	0.88	58.6%
upper limit of applicable range	3.21	8.08	29.16	36.06	38.30	1.04	104.2%

* The ranges of Hg concentrations covered a factor >6

**MDL was based on MDL-A and MDL-B Result

***%Hg+2 is the percent of the THg+PHg

Based on the results of this study, it is argued here that the FMSS method is suitable for future applications under real conditions in similar matrices. It is expected that the FMSS method will be applicable to other fluegas matrices, such as from municipal waste incinerators, sewage sludge incinerators, smelting and other high temperature or combustion processes. However, the limitations of this study are for coal fluegas only.

IX. Laboratory Reproducibility

One important aspect of the PBMS is to collect samples in different fluegas systems, coal type, sampling technicians and analysis technicians. Therefore, to address the question of: "Can multiple operators and multiple laboratories obtain comparable data?" The answer is yes based on a brief inter-comparison of the FMSS and OH methods that was applied at the DOE. Results of this inter-comparison can be seen in Figure IX-1 in which the Hg concentration in $\mu\text{g}/\text{m}^3$ (y-axis) is shown for the FMSS method (left bar) and for the OH method (right bar) for each run at the DOE.

Figure IX-1 Intercomparison at another site (DOE)

Note: FMSS Method Result is indicated as SOH for the "sorbent" method that is similar to OH

FMSS precision during this brief inter-comparison ranged from 0.8% to 8.4% (RPD) which was excellent considering that the data was collected by people trained to operate (albeit a simple system) for essentially a 2 hour session. This again demonstrates the robustness of the technique and shows that field operators can be trained to perform clean techniques easily in the field.

X. Conclusions

The FMSS method was evaluated relative to the Ontario Hydro (OH) Method at the Energy and Environment Research Center (EERC) and again briefly at the US DOE for a variety of fuels, ash loadings, and pollution control methods. The evaluation included numerous ruggedness tests using quadruplicate sampling devices, both with and without species specific (Hg^0 and Hg^{+2}) spiking.

The FMSS method exhibited very good precision with a mean relative percent difference (RPD) of $\pm 22\%$ for Hg^0 , $\pm 8\%$ for Hg^{+2} , $\pm 10\%$ for THg, and $\pm 6.5\%$ for $\% \text{Hg}^{+2}$ or the percent oxidized Hg of the total (THg+PHg). The FMSS exhibited good agreement with OH methods and accuracy was better than $\pm 20\%$ for all species for the range of conditions in this study, including during ruggedness tests. The mean accuracy of the duplicates and the triplicates for the FMSS method was better than 97% for Hg^{+2} , THg, and $\% \text{Hg}^{+2}$. Based on the results presented here, the FMSS method is expected to be very reliable, even in the presence of SO_2 , NO_x , HCl, and flyash.

Based on the results of this PBMS, we conclude the FMSS Method is equivalent to the ASTM approved OH Method and a therefore a valid method for the determination of total Hg, PHg, gaseous Hg^{+2} and Hg^0 concentrations in a flue gas matrix. Considering many factors, including simplicity, lack of hazardous solutions in the field, precision, sensitivity, accuracy and cost, the FMSS method has many advantages that make it a viable choice for the measurement total and speciated mercury in coal fluegas.

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Appendix A

SUMMARY OF ALL THE FMSS AND OH RESULTS AT THE EERC

FIELD BLANKS		Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result									
RESULT		PHg	KCLA	KCLB	ICA	T-Vol		[pHg]	[Hg ⁰]	[Hg ⁺²]	[THg]
FMSS SAMPLE ID		ng/trap	ng/trap	ng/trap	ng/trap	corr., liter		ug/m ³	ug/m ³	ug/m ³	ug/m ³
FMS-9-030700 FB		nc	0.1473	0.0859	0.145	30.0		na	0.0048	0.0078	0.0126
FMS-13-030700 FB		nc	2.78	0.0205	0.015	30.0		na	0.0154	0.0029	0.0183
FMS-3-031000 FB		0.0419	0.0395	0.0000	0.490	30.0		0.0014	0.0163	0.0013	0.0177
FMS-5-031300 FB		na	0.3990	0.0000	0.377	30.0		na	0.0126	0.0000	0.0126
FMS-4-031400 FB		0.0239	0.0000	0.0000	0.416	30.0		0.0008	0.0139	0.0000	0.0139
FMS-8-031400 FB		0.132	0.0356	0.0823	0.371	30.0		0.0044	0.0124	0.0039	0.0163
FMS-4-031500 FB		nc	0.0000	0.0000	0.640	30.0		na	0.0213	0.0000	0.0213
FMS-8-031600 FB		nc	0.2026	0.0000	0.789	30.0		na	0.0263	0.0068	0.0330
		2.78±test									
	n=8							FMSS mean	0.0022	0.0154	0.0028
								STDEV	0.0019	0.0064	0.0031
								MDL-A	0.0058	0.0192	0.0093
One KCLA value was removed by t-test and replaced by mean.											

CLEAN NATURAL GAS FLUEGAS MATRIX BLANK											
RESULT		PHg	KCLA	KCLB	ICA	T-Vol		[pHg]	[Hg ⁰]	[Hg ⁺²]	THg
FMSS SAMPLE ID		ng/trap	ng/trap	ng/trap	ng/trap	corr., liter		ug/m ³	ug/m ³	ug/m ³	ug/m ³
FMS-1-030600		inval	0.675	0.000	1.45	26.7		na	0.0390	0.0224	0.0615
FMS-3-030600		nc	0.020	0.102	1.23	30.9		na	0.0246	0.0011	0.0257
FMS-4-030600		nc	0.123	0.123	1.52	31.2		na	0.0335	0.0050	0.0385
FMS-1-030700		nc	0.225	0.000	0.697	28.7		na	0.0089	0.0050	0.0139
FMS-2-030700		nc	0.000	0.123	0.944	30.3		na	0.0158	0.0012	0.0170
FMS-3-030700		nc	0.286	0.164	0.726	22.0		na	0.0176	0.0176	0.0353
FMS-4-030700		nc	0.082	0.000	0.784	31.2		na	0.0098	-0.0002	0.0096
FMS-5-030700		nc	0.000	0.020	1.16	30.7		na	0.0225	-0.0022	0.0203
		Flowrate RSD 11.1%									
EERC Run 0	3/6/00-3/7/00							FMSS mean	NA	0.0215	0.0063
	n=8							STDEV	NA	0.0107	0.0089
	Natural Gas			No spikes							
See MDL-B Calculation								NA	0.032	0.027	0.051

COAL FLUEGAS MATRIX MID LEVEL NO SPIKES

RESULT FMSS SAMPLE ID	PHg			KCLA			KCLB			ICA			T-Vol			Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result				
	ng/trap	ng/trap	ng/trap	ng/trap	ng/trap	ng/trap	ng/trap	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³
FMS-5-030800	0.055	150.3	0.368	0.368	8.85	19.3							0.0006	0.443	7.79	8.24	94.5%	8.24	94.6%	
FMS-6-030800	0.21	181.3	0.368	6.01	20.2								0.0081	0.282	8.98	9.26	96.9%	9.27	97.0%	
FMS-7-030800	0.10	149.6	0.182	4.22	19.5								0.0031	0.201	7.69	7.89	97.4%	7.90	97.5%	
FMS-8-030800	inval	175.1	0.306	5.76	19.6								na	0.279	8.97	9.24	96.9%	9.25	97.0%	
FMS-9-030800	0.29	181.3	0.678	7.74	20.3								0.0119	0.365	8.95	9.31	96.0%	9.32	96.1%	
FMS-10-030800	0.27	152.1	0.000	5.15	19.6								0.0113	0.247	7.76	8.00	96.8%	8.01	96.9%	
FMS-11-030800	26.02	26.0	0.492	48.10	15.0							temp-control	4.74	3.20	4.77	4.97	35.5%	4.97	35.6%	
FMS-12-030800	33.06	45.2	0.464	26.76	20.5							temp-control	4.64	4.29	0.76	2.05	37.0%	2.05	37.1%	

Flowrate RSD 9.29%

EERC Run 4

3/8/2000-18:37

n=6

Bitcoal ESP with spikes

See MDL-C Calculation

Quads No Tests

FMSS Mean 0.0070
STDEV 0.0050
FMSS Precision 71.3%

8.36 8.66 96.4%
0.667 0.682 1.0%
8.0% 7.9% 1.1%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg]			[Hg ⁰]			[Hg ⁺²]			THg			%Hg ⁺²			THg+PHg			%Hg ⁺²		
	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³	ug/m ³
D2-30	0.210	0.480	7.49							7.97			93.9%			7.9770			94.0%		
D2-40	0.300	0.450	8.27							8.72			94.8%			8.7270			94.8%		
OH mean	0.255	0.465	7.88							8.35			94.3%			8.35			94.4%		
OH Precision, %RPD	35.3%	6.5%	9.9%							9.0%			0.92%			9.0%			0.91%		
Accuracy																					
as % recovery	NA	65.2%	106.0%							103.8%			102.2%			103.7%			102.2%		

COAL FLUEGAS MATRIX WITH HIGH LEVELSPIKES

COAL FLUEGAS MATRIX WITH HIGH LEVELSPIKES										Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result			
RESULT	PHg	KCLA	KCLB	ICA	T-Vol		[pHg]	[Hg ⁰]	[Hg ⁺²]	THg	%Hg ⁺²	THg+PHg	%Hg ⁺²
FMSS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter		ug/m ³	ug/m ³	ug/m ³	ug/m ³		ug/m ³	
FMS-1-031700	nc	820.9	63.4	245.0	26.6		nc	9.18	33.2	42.4	78.3%	42.39	78.3%
FMS-2-031700	nc	800.2	129.0	237.0	26.6		nc	8.88	34.9	43.8	79.7%	43.79	79.7%
FMS-3-031700	nc	809.2	14.6	214.3	27.3		nc	7.84	30.2	38.0	79.3%	38.06	79.4%
FMS-4-031700	nc	823.4	67.1	256.9	27.4		nc	9.37	32.5	41.9	77.6%	41.95	77.6%
FMS-5-031700	nc	870.7	18.0	258.7	25.4		nc	10.2	35.0	45.2	77.4%	45.24	77.5%
FMS-6-031700	nc	921.9	12.2	234.6	27.2		nc	8.62	34.4	43.0	79.9%	43.01	80.0%
FMS-7-031700	nc	831.5	12.7	191.6	27.0		nc	7.08	31.3	38.3	81.5%	38.36	81.5%
					Flowrate RSD	2.56%							

EERC Run 16

3/17/2000-10:06

n=8

Bitcoal BH with spikes
See MDL-C Calculation
Quads No Tests

FMSS Mean	NA	8.74	33.1	41.8	79.2%	41.8	79.3%
STDEV	NA	1.02	1.86	2.69	1.5%	2.69	1.5%
FMSS Precision	NA	11.7%	5.6%	6.4%	1.9%	6.4%	1.9%

Ontario Hydro Simultaneous Result									
OH SAMPLE ID	[pHg]	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³	%Hg ⁺² THg+PHg		
D9-1o	0.030	7.64	36.6	44.2	82.7%	44.24	82.7%		
D9-2o	0.020	10.18	38.8	49.0	79.2%	49.02	79.2%		
OH mean	0.025	8.91	37.7	46.6	80.9%	46.6	81.0%		
OH Precision, %RPD	40.0%	28.5%	5.9%	10.3%	4.3%	10.3%	4.3%		
Accuracy as % recovery	NA	98.1%	87.7%	89.7%	97.9%	89.7%	97.9%		

COAL FLUEGAS MATRIX MID LEVEL FLOWRATE TESTS

RESULTS							
RESULT	PHg	KCLA	KCLB	ICA	T-Vol	Flowrate	
FMS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter	slpm	
FMS-1-030800	inval	88.81	0.35	5.79	9.1	0.22	
FMS-2-030800	11.2	178.9	1.37	12.04	23.6	0.44	
FMS-3-030800	inval	234.2	0.78	16.56	35.1	0.44	
FMS-4-030800	13.8	199.2	0.35	19.61	24.4	0.57	
							Flowrate, RSD 33.9%
EERC Run 3 n=4	3/8/2000-11:37 Bit-coal ESP no spikes See Quad Flowrate Test Mid Level		FMSS Mean STDEV FMSS Precision		0.516 0.066 12.8% 0.579 0.181 31.3% 7.49 0.74 9.9%		
Ontario Hydro Simultaneous Result							
OH SAMPLE ID	[pHg]	[Hg ⁰]	[Hg ⁺²]	THg	%Hg ⁺²	THg+PHg	%Hg ⁺²
D2-1o	na	0.510	8.98	9.49	84.7%	10.60	94.6%
D2-2o	1.70	0.560	8.37	8.93	83.4%	10.04	93.7%
OH mean	1.70	0.535	8.68	9.21	84.1%	10.32	94.2%
OH Precision, %RPD	NA	9.3%	7.0%	6.1%	1.6%	5.4%	1.0%
Accuracy as % recovery							99.0%

COAL FLUEGAS MATRIX MID LEVEL TEMPERATURE TESTS

COAL FLUEGAS MATRIX MID LEVEL TEMPERATURE TESTS												
RESULT	PHg	KCLA	KCLB	ICA	T-Vol	Temp	[pHg]	[Hg ⁰]	[Hg ⁺²]	Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result		
FMSS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr. liter	deg C	ug/m ³	ug/m ³	ug/m ³	THg	THg+PHg	%Hg ⁺²
FMS-5-031500	nc	217.7	0.089	3.03	26.0	75.7	nc	0.10	8.39	8.49	8.50	98.8%
FMS-6-031500	nc	243.6	0.036	5.88	26.2	88.4	nc	0.21	9.31	9.52	9.53	97.8%
FMS-7-031500	nc	215.3	0.191	3.09	26.3	96.4	nc	0.10	8.19	8.29	8.30	98.8%
FMS-8-031500	nc	250.1	0.347	4.54	26.4	105.7	nc	0.16	9.49	9.65	9.66	98.4%
Floware, RSD Temp, RSD												
						0.7%						
						13.9%						
EERC Run 12 n=4	3/15/2000-13:55						NA	0.142	8.84	8.99	9.00	98.4%
	Bitcoal BH no spikes						NA	0.052	0.65	0.69	0.69	0.47%
	See Quad Temperature						NA	36.3%	7.3%	7.7%	7.7%	0.5%
	Test Mid Level											
Ontario Hydro Simultaneous Result												
							[pHg]	[Hg ⁰]	[Hg ⁺²]	THg	THg+PHg	%Hg ⁺²
							OH SAMPLE ID			ug/m ³	ug/m ³	
							D7-3o	0.010	9.63	9.98	9.99	96.5%
							D7-4o	0.010	10.5	10.9	10.89	96.6%
							OH mean	0.010	10.1	10.4	10.4	96.5%
							OH Precision, %RPD	0.0%	5.6%	8.74%	8.6%	0.1%
Accuracy as % recovery												
							NA	39.6%	87.8%	86.1%	86.2%	102.0%

COAL FLUEGAS MATRIX HIGH LEVEL TEMPERATURE TESTS

COAL FLUEGAS MATRIX HIGH LEVEL TEMPERATURE TESTS													
RESULT	PHg ng/trap	KCLA ng/trap	KCLB ng/trap	ICA ng/trap	T-Vol corr., liter	Temp deg C	Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result						
							[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³	%Hg ⁺² THg+PHg
FMS-1-031600	nc	819.0	15.3	215.6	26.5	75.3	nc	8.10	31.4	39.5	79.5%	39.55	79.5%
FMS-2-031600	nc	711.1	14.9	220.0	26.4	86.7	nc	8.33	27.5	35.9	76.7%	35.89	76.8%
FMS-3-031600	nc	732.2	6.89	170.6	26.3	96.6	nc	6.47	28.1	34.6	81.2%	34.58	81.3%
FMS-4-031600	nc	650.1	61.3	216.7	26.4	105.9	nc	8.20	27.0	35.2	76.6%	35.18	76.7%
Flowrate, RSD Temp, RSD													
						0.4%	14.5%						
FMSS Mean							NA	7.78	28.5	36.3	78.5%	36.30	78.6%
FMSS STDEV							NA	0.874	2.00	2.23	2.2%	2.23	2.23%
FMSS Precision							NA	11.2%	7.02%	6.15%	2.8%	6.1%	2.84%
Test High Level													
Bitcoal BH with spikes													
See Quad Temperature													
n=4													
Ontario Hydro Simultaneous Result													
OH SAMPLE ID		[pHg]	[Hg ⁰]	[Hg ⁺²]	THg		%Hg ⁺² THg+PHg						
D8-1o		0.02	8.23	30.0	38.2		78.4%						
D8-2o		0.02	8.67	33.7	42.4		79.5%						
OH mean		0.02	8.45	31.9	40.3		79.0%						
OH Prediction, %RPD		0.0%	5.2%	11.7%	10.4%		1.4%						
Accuracy as % recovery													
NA		92.0%	89.5%	90.0%	90.0%		99.4%						

COAL FLUEGAS MATRIX HIGH LEVEL FLOWRATE TESTS

COAL FLUEGAS MATRIX HIGH LEVEL FLOWRATE TESTS										Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Results				
RESULT	PHg	KCLA	KCLB	ICA	T-Vol	Flowrate	[pHg]	[Hg ⁰]	[Hg ⁺²]	THg	%Hg ⁺²	THg+PHg	%Hg ⁺²	
FMSS SAMPLE ID	ng/trap	ng/trap	ng/trap	corr., liter	slpm		ug/m ³	ug/m ³	ug/m ³	ug/m ³		ug/m ³	THg+PHg	
FMS-9-031600	nc	467.1	7.10	123.7	14.6	0.24	na	8.44	32.4	40.8	79.3%	40.89	79.3%	
FMS-10-031600	nc	836.7	7.67	193.9	26.3	0.47	na	7.36	32.1	39.5	81.3%	39.55	81.4%	
FMS-11-031600	nc	1060.1	12.5	362.5	38.0	0.60	na	9.52	28.2	37.7	74.7%	37.77	74.8%	
FMS-12-031600	nc	1352.5	132.7	534.8	50.8	0.88	na	10.5	29.2	39.8	73.5%	39.80	73.6%	
Flowrate, RSD						48.8%								
EERC Run 15 3/16/2000-16:13 n=4	FMSS Mean						NA	8.960	30.50	39.46	77.2%	39.50	77.3%	
	Bitcoal - BH with spikes						NA	1.360	2.09	1.29	3.7%	1.29	3.7%	
	See Quad Flowrate Test High Level						NA	15.2%	6.9%	3.3%	4.8%	3.3%	4.8%	
	Ontario Hydro Simultaneous Result													
	OH SAMPLE ID	[pHg]	[Hg ⁰]	[Hg ⁺²]	THg	%Hg ⁺²	THg+PHg	%Hg ⁺²						
D8-50	0.050	8.35	27.5	35.82	76.8%	35.86	76.7%							
D8-60	0.030	10.41	31.6	42.05	75.2%	42.09	75.2%							
OH mean						0.04	9.38	29.6	38.9	75.9%	39.0	76.0%		
						0.014	1.46	2.95	4.41	1.01%	4.41	1.02%		
OH Precision, %RPD						50.0%	22.0%	14.1%	16.0%	1.9%	16.0%	1.9%		
Accuracy as % recovery						NA	95.5%	103.2%	101.4%	101.7%	101.4%	101.7%		

CLEAN NATURAL GAS FLUEGAS MATRIX WITH Hg° SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMSS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-6-030700	nc	0.532	0.184	389.2	29.6
FMS-7-030700	nc	0.307	0.020	392.1	30.1
FMS-8-030700	nc	1.043	0.020	302.1	24.5
Flowrate RSD					11.0%

EERC Run 1
3/7/2000-13:35
n=3
Natural Gas
Hg° spikes

FMSS Mean	NA	12.8	0.0233	12.8	0.18%	12.9	0.18%	99.66%
STDEV	NA	0.44	0.016	0.428	0.13%	0.43	0.13%	0.139%
FMSS Precision	NA	3.45%	70.2%	3.34%	73.1%	3.3%	73.1%	0.140%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³	%Hg ⁰
D1-1o	0.02	10.05	0.23	10.3	2.2%	10.30	2.2%	10.30	97.57%
na	na	na	na	na	na	na	na	na	na

OH mean 0.02 10.05 0.23 10.3 2.2% 10.30 2.2% 97.57%

OH Precision, %RPD NA NA NA NA NA NA NA NA

Accuracy
as % recovery NA NA NA NA NA NA

CLEAN NATURAL GAS FLUEGAS MATRIX WITH Hg⁺² SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMSS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-10-030700	nc	381.7	0.000	66.8	27.7
FMS-11-030700	nc	403.4	0.123	61.0	28.6
FMS-12-030700	nc	371.9	0.020	46.5	25.6
Flowrate RSD					5.6%

#####

EERC Run 2
3/7/2000-16:40
n=3
Natural Gas
Hg⁺² spikes

FMSS Mean	NA	2.10	14.1	16.2	77.7%	18.2	87.0%
STDEV	NA	0.30	0.38	0.08	1.73%	0.077	1.90%
FMSS Precision	NA	14.2%	2.65%	0.48%	2.23%	0.43%	2.18%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³	%Hg ⁺²
D1-2o	1.94	2.11	6.38	8.49	61.2%	10.43	75.1%
na	na	na	na	na	na	na	na

OH mean 1.94 2.11 6.38 8.49 61.2% 10.4 75.1%

OH Precision, %RPD NA NA NA NA NA NA NA NA

Accuracy
as % recovery NA NA NA NA NA NA

COAL FLUEGAS MATRIX MID LEVEL NO SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-1-031000	2.65	193.6	0.426	11.0	25.9
FMS-2-031000	1.52	206.5	1.35	7.57	25.3

Flowrate RSD 1.6%

EERC Run 5
3/10/2000-16:24
n=2
Bitcoal ESP
No Spikes

FMSS Mean	0.079	0.347	7.86	8.20	93.1%	8.44	95.7%
STDEV	0.030	0.089	0.50	0.42	1.4%	0.42	1.3%
FMSS Precision	53.5%	36.4%	9.09%	7.16%	2.12%	6.97%	1.93%

Ontario Hydro Simultaneous Result							
OH SAMPLE ID	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³	%Hg ⁺² THg+PHg
D4-10	0.370	0.46	6.63	7.09	90.5%	7.32	93.5%
D4-20	0.400	0.46	6.99	7.45	91.0%	7.68	93.8%
OH mean	0.385	0.46	6.81	7.27	90.8%	7.50	93.7%
OH Precision, %RPD	7.8%	0.0%	5.3%	5.0%	0.5%	4.8%	0.33%
Accuracy as % recovery	20.6%	75.4%	115.4%	112.9%	102.6%	112.5%	102.2%

COAL FLUEGAS MATRIX WITH HIGH LEVEL SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-1-031300	15.1	745.2	63.5	87.8	25.1
FMS-2-031300	inval	776.2	43.7	120.7	25.1

Flowrate RSD 0.1%

EERC Run 6
3/13/2000-9:20
n=2
Bitcoal ESP
With Spikes

FMSS Mean	0.60	4.14	32.5	36.6	87.3%	37.2	88.7%
STDEV	NA	0.924	0.291	1.22	2.1%	1.22	2.2%
FMSS Precision	NA	31.6%	1.27%	4.70%	3.4%	4.6%	3.43%

Ontario Hydro Simultaneous Result							
OH SAMPLE ID	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺²	THg+PHg ug/m ³	%Hg ⁺² THg+PHg
D5-10	0.66	4.67	30.4	35.07	85.3%	35.65	86.7%
D5-20	0.48	7.02	25.5	32.54	77.0%	33.12	78.4%
OH mean	0.57	5.85	28.0	33.8	81.2%	34.4	82.6%
OH Precision, %RPD	31.6%	40.2%	17.5%	7.5%	10.1%	7.4%	10.0%
Accuracy as % recovery	105.1%	70.8%	116.1%	108.3%	107.6%	108.1%	107.5%

COAL FLUEGAS MATRIX WITH HIGH LEVEL SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-3-031300	3.3	629.8	6.39	170.6	25.7
FMS-4-031300	2.2	705.7	7.49	197.1	25.7
				Flowrate RSD	0.1%

EERC Run 7
n=2
3/13/2000-14:50
Bitcoal ESP
With Spikes

Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result					
	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺² THg+PHg ug/m ³
	0.125	6.63	24.8	31.4	78.4%
	0.082	7.66	27.8	35.4	77.9%
					78.9%
					78.4%
FMSS Mean	0.104	7.15	26.3	33.4	78.2%
STDEV	0.030	0.72	2.10	2.83	42.5%
FMSS Precision	41.0%	14.3%	11.3%	12.0%	0.58%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺² THg+PHg ug/m ³
D5-3o	0.290	6.97	24.8	31.8	77.6%
D5-4o	0.310	6.64	32.6	39.3	82.7%
OH mean	0.300	6.81	28.7	35.5	80.1%
OH Precision, %RPD	6.7%	4.8%	27.1%	21.0%	6.3%

OH Precision, %RPD 6.7% 4.8% 27.1% 21.0% 6.3%

Accuracy as % recovery	34.6%	105.0%	91.5%	94.0%	97.5%
					94.1%
					97.6%

COAL FLUEGAS MATRIX WITH HIGH LEVEL SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-6-031300	23.0	655.9	6.2	86.9	25.1
FMS-7-031300	inval	522.1	156.7	181.4	25.2
				Flowrate RSD	0.3%

EERC Run 8
3/13/2000-18:53
Bitcoal ESP
With Spikes

Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result					
	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺² THg+PHg ug/m ³
	0.914	3.45	26.4	29.9	86.2%
	nc	7.18	26.9	34.1	77.2%
					88.4%
					78.9%
FMSS Mean	0.91	5.32	26.7	32.0	81.7%
STDEV	NA	NA	0.38	3.02	40.2%
FMSS Precision	NA	NA	2.0%	13.3%	11.0%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺² THg+PHg ug/m ³
D5-5o	0.66	7.56	23.8	31.40	74.1%
D5-6o	0.60	7.66	30.6	38.21	78.4%
OH mean	0.63	7.61	27.2	34.8	76.2%
OH Precision, %RPD	9.5%	1.3%	24.7%	19.6%	5.6%

OH Precision, %RPD 9.5% 1.3% 24.7% 19.6% 5.6%

Accuracy as % recovery	145.0%	69.9%	98.1%	91.9%	107.2%
					92.1%
					107.4%

COAL FLUEGAS MATRIX WITH HIGH LEVEL SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMSS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-1-031400	83.2	448.9	0.22	12.7	16.7
FMS-2-031400	63.0	503.8	1.26	12.8	23.6
FMS-3-031400	95.9	596.7	30.3	16.2	27.3

Flowrate RSD 23.9%

EERC Run 9

3/14/2000-10:30
n=3
Bitcoal ESP - heavy particulate
With Spikes

Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result						
	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺² THg+PHg ug/m ³	%Hg ⁺² THg+PHg %Hg ⁺²
FMSS Mean	3.72	0.618	23.8	24.4	84.0%	97.5%
STDEV	1.18	0.114	2.85	2.97	1.24%	0.15%
FMSS Precision	31.6%	18.5%	12.0%	12.2%	1.47%	0.16%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺² THg+PHg ug/m ³	%Hg ⁺² THg+PHg %Hg ⁺²
D6-1o	1.21	0.91	21.6	23.7	78.2%	91.1%
D6-2o	6.89	1.26	24.1	32.3	66.7%	74.7%
OH mean	4.05	1.09	22.8	28.0	72.5%	82.9%
OH Precision, %RPD	140.2%	32.3%	11.0%	30.6%	15.9%	19.7%

Accuracy

as % recovery	91.9%	56.9%	104.0%	87.1%	115.9%	117.6%
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COAL FLUEGAS MATRIX WITH HIGH LEVEL SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMSS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-5-031400	138.8	420.7	0.736	20.6	20.8
FMS-6-031400	143.3	168.2	0.399	8.85	12.2
FMS-7-031400	128.9	272.4	0.337	16.5	21.4

Flowrate RSD 28.3%

EERC Run 10

3/14/2000-16:00
n=3
Bitcoal ESP - heavy particulate
With Spikes

Frontier's Fluegas Mercury Sorbent Speciation (FMSS) Result						
	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺² THg+PHg ug/m ³	%Hg ⁺² THg+PHg %Hg ⁺²
FMSS Mean	8.14	0.81	15.6	16.42	62.0%	95.0%
STDEV	3.13	0.14	4.09	4.22	5.4%	0.52%
FMSS Precision	38.4%	17.5%	26.2%	25.7%	8.75%	0.55%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg] ug/m ³	[Hg ⁰] ug/m ³	[Hg ⁺²] ug/m ³	THg ug/m ³	%Hg ⁺² THg+PHg ug/m ³	%Hg ⁺² THg+PHg %Hg ⁺²
D6-3o	10.3	1.58	19.7	21.3	66.1%	92.6%
D6-4o	7.53	1.9	23.6	25.4	69.5%	92.7%
OH mean	8.89	1.72	21.6	23.4	67.8%	92.7%
OH Precision, %RPD	30.6%	15.7%	18.0%	17.8%	4.9%	0.2%

Accuracy

as % recovery	91.6%	47.4%	72.1%	70.3%	91.4%	102.5%
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COAL FLUEGAS MATRIX MID LEVEL NO SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-1-031500	nc	236.0	0.242	2.37	26.1
FMS-2-031500	nc	269.4	0.039	2.88	26.1
FMS-3-031500	nc	243.6	0.574	2.30	27.3

Flowrate RSD 2.7%

EERC Run 11

3/15/2000 -9:40
n=3
Bitcoal BH
No Spikes

FMSS Mean	NA	0.0798	9.45	9.53	98.7%	9.57	99.2%
STDEV	NA	0.014	0.767	0.781	0.05%	0.78	0.074%
FMSS Precision	NA	17.1%	8.12%	8.20%	0.05%	8.2%	0.08%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg]	[Hg ¹]	[Hg ²]	THg	%Hg ²	THg+PHg	%Hg ²
	ug/m ³	ug/m ³	ug/m ³	ug/m ³		ug/m ³	

D7-10	0.060	0.400	9.78	10.18	95.7%	10.22	96.1%
D7-20	0.020	0.470	10.7	11.15	95.4%	11.19	95.8%

OH mean	0.040	0.435	10.2	10.7	95.6%	10.7	95.9%
	0.028	0.049	0.64	0.69	0.18%	0.69	0.20%
OH Precision, %RPD	100.0%	16.1%	8.8%	9.1%	0.26%	9.1%	0.30%

Accuracy as % recovery	NA	18.3%	92.3%	89.3%	103.3%	89.4%	103.4%
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COAL FLUEGAS MATRIX WITH HIGH LEVEL SPIKES

RESULT	PHg	KCLA	KCLB	ICA	T-Vol
FMS SAMPLE ID	ng/trap	ng/trap	ng/trap	ng/trap	corr., liter
FMS-5-031600	nc	699.5	9.80	246.1	23.5
FMS-6-031600	nc	812.6	6.76	268.5	25.9
FMS-7-031600	nc	599.5	6.10	227.2	26.3

Flowrate RSD 6.0%

EERC Run 14

3/16/2000-12:31
n=3
Bitcoal BH
With Spikes

FMSS Mean	NA	9.8	28.3	38.1	74.1%	38.1	74.1%
STDEV	NA	1.03	4.62	5.63	1.31%	5.63	1.30%
FMSS Precision	NA	10.5%	16.3%	14.8%	1.76%	14.8%	1.75%

Ontario Hydro Simultaneous Result

OH SAMPLE ID	[pHg]	[Hg ¹]	[Hg ²]	THg	%Hg ²	THg+PHg	%Hg ²
	ug/m ³	ug/m ³	ug/m ³	ug/m ³		ug/m ³	

D8-30	na	8.54	29.9	38.4	77.7%	38.43	77.8%
D8-40	0.02	9.70	32.4	42.1	76.9%	42.13	77.0%

OH mean	0.02	9.12	31.1	40.3	77.3%	40.3	77.4%
OH Precision, %RPD	NA	12.7%	8.2%	9.2%	1.03%	9.2%	1.04%

Accuracy as % recovery	NA	107.6%	90.8%	94.6%	95.8%	94.6%	95.8%
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