

PERFORMANCE EVALUATION OF INERTIAL SEPARATION PROBES FOR VAPOR-PHASE MERCURY MEASUREMENTS

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ABSTRACT

Measuring vapor-phase mercury from coal-fired flue gas using a continuous mercury analyzer can be complicated by the presence of particulate matter. Initially, standard sampling filters were used to remove particulate matter as the sample gas was extracted from the duct. Significant variations in the measured mercury concentration were attributed to capture of vapor-phase mercury by particulate deposited on the sampling filter. In the past few years, probes designed to inertially separate the bulk of the particulate matter from a gas sample have been successfully used with mercury analyzers to minimize particulate-related sampling artifacts.

In preparation for a Clean Coal program, a performance evaluation and comparison of commercially available inertial separation probes is being conducted. Probes designed by Baldwin Environmental, EPM Environmental (Thermo Electron Corporation), and Apogee Scientific were included in the evaluation. The probes were tested to determine their potential to oxidize mercury at various operating temperatures, their ability to accurately measure elemental and oxidized mercury spikes, and the effect of activated carbon injection into the probes (<0.15 sec residence time) at various operating temperatures. All tests were conducted in actual flue gas. This paper presents a description of each of the probes and the test results to date.

INTRODUCTION

The U.S. Environmental Protection Agency (EPA) is required to announce mercury MACT regulations under Title III of the Clean Air Act Amendments of 1990 by December 15, 2003. In anticipation of these regulations, We Energies and ADA Environmental Solutions (ADA-ES) submitted a proposal to DOE as part of the Clean Coal Power Initiative. The proposed project, which was selected for award, is to design, install, evaluate and operate the TOXECON™ technology for integrated emission control of mercury and particulate matter. The system will be designed to treat the gas from We Energies Presque Isle Units 7-9. In addition to the goal of achieving 90% continuous mercury removal using powdered activated carbon injection (PAC), one of the demonstration goals is to develop and demonstrate a mercury continuous emission monitor (CEM).

There are several key components to a robust and reliable mercury CEM. These include systems to extract, pretreat, transport, and measure mercury in flue gas. Some of these components, specifically the measurement system, are commercially available and operate fairly well in some gas matrices. However, other components, such as the pretreatment system, are still in development and have not been proven on a variety of flue gas streams.

During the evaluation program described in this paper, three commercially available extraction probes were evaluated at We Energies Pleasant Prairie Power Plant. All three probes utilize inertial separation to separate flue gas from particulate. Apogee Scientific, Baldwin Environmental, and Thermo Electron Corporation provided the probes. ADA-ES contracted with Southern Research Institute to provide the mercury analyzer and the elemental mercury spiking system. A system for spiking oxidized mercury was provided by EPA's NRML/APPC.

In general, all three probes demonstrated reliable performance in delivering a mercury-laden sample for total vapor-phase mercury measurements. This paper describes the equipment used for the evaluation, the test protocol, and the results.

EQUIPMENT DESCRIPTIONS

Inertial Separation Probes

Fly ash and other particulate, such as injected sorbents, can capture or convert vapor-phase mercury if present on a sampling filter upstream of a mercury measurement system. Inertial separation probes provide a method of separating particulate from the gas sample while minimizing gas-particle interactions.

Three commercially available inertial separation probes were evaluated in a short test program. Table 1 identifies the probe manufacturers and key characteristics. Figure 1 shows photographs of each of the three probes. Another probe supplier, Mott Corporation in Farmington, Connecticut, was not included in the evaluation program because they do not provide heated systems.

Table 1. Inertial Probes Included in Evaluation Program.

Manufacturer	Material	Filter Diameter (inches)	Design Flow Rate (acfm)	Other
Apogee Scientific	Stainless Steel	5/8	10-12	Venturi flow meter
Baldwin Environmental	Stainless Steel with Durinert™ Coating	5/8	6-10.5	Venturi flow meter Heated eductor air
Thermo Electron (EPM)	Stainless Steel	1/4	1.5-2	Heated filter blowback option, dilution probe option

The filter element in the inertial separation probe is a sintered stainless steel porous tube. A filter housing tube surrounds the filter element, creating a minimum-volume annular volume for sample collection. A high velocity gas flow is developed axially through the porous tube using a vacuum eductor installed downstream of the filter element. The sample gas is drawn radially through the porous tube at a very low face velocity, passing through the housing annulus and into the sample port. The high velocity axial gas flow separates the bulk of the particulate matter from the sample stream. Any remaining fine particles are filtered through the porous tube. The inner surface of the porous tube is self-cleaning by the scouring effect of the particles in the main gas stream. A sketch of an inertial filter is shown in Figure 2.

Two of the inertial probes tested were provided with venturi flow meters to monitor flow. All three probes used vacuum eductors to draw flow through the filter element.



Apogee QGIS Probe



Baldwin Probe (shown with cover open)



Thermo Electron (EPM Environmental) (shown with covers removed)

Figure 1. Photographs of the inertial separation probes included in the evaluation program.

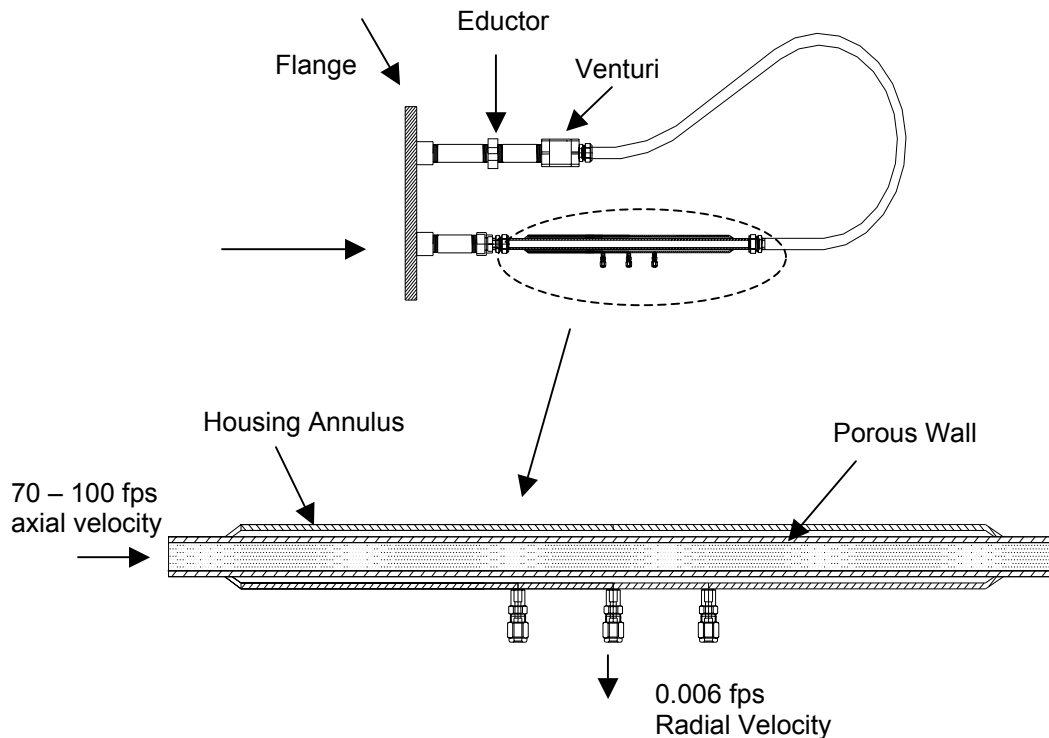


Figure 2. General arrangement sketch of the inertial probe assembly and filter.

Sample Pretreatment System

ADA-ES fabricated the flue gas pretreatment system. The system consists of two sampling trains, each incorporating two impingers followed by a thermoelectric cooler to dry the sample. For the total vapor-phase mercury sampling train, a solution of mildly acidified stannous chloride is continuously introduced to the first impinger and spent solution is continuously removed. The stannous chloride converts all vapor-phase mercury to the elemental form. A solution of sodium hydroxide is introduced into the second impinger. For elemental mercury measurements, the stannous chloride impinger is replaced with a solution of potassium chloride to remove oxidized forms of mercury. The impingers are located at the sample port on the extraction probe to minimize transport losses.

Sample Transport

The mercury instrument was installed in a sampling trailer on the ground level below the sample extraction location. The sample was transported to the instrument through 50 feet of heated (300°F) ¼-inch PFA tubing. The flow through each sample line was maintained at nominally 1.5 slpm. The mercury analyzer drew a slipstream from the main sample flow for analysis.

Mercury Analyzer

A Tekran Model 2537A Mercury Vapor Analyzer was used for this program. The instrument uses two mercury traps containing ultra-pure gold adsorbent. The amalgamated mercury is

thermally desorbed for detection using Cold-Vapor Atomic Fluorescence Spectrometry (CVAFS). Continuous sampling is achieved by alternating the traps between collecting and desorbing mercury, with one trap always in the collection mode and one trap always in the measurement mode. Flow through the trap was maintained at 250 cc/min.

Mercury Analyzer Calibration System

The Tekran instrument has provisions for two methods of calibration: manual injection or an internal elemental mercury permeation tube for automatic instrument calibrations. The permeation tube option was used during these tests.

Elemental and Oxidized Mercury Spiking Systems

Two systems were used during the program to introduce elemental and oxidized mercury into the probes upstream of the inertial filter. The elemental spiking system was a PSA 10.534 Mercury Calibration System fabricated by P S Analytical. The PSA 10.534 provides a wide range of mercury concentrations by altering the temperature of a mercury reservoir and varying the gas flowrate through the reservoir. The mercury reservoir is constructed by impregnating elemental mercury on an inert substrate. The gas passing over the substrate becomes saturated with mercury at the reservoir temperature.

The oxidized mercury source was a Hot-Vapor Calibration (HOVACAL) system manufactured by IAS GmbH (distributed in the U.S. by EcoChem Analytics). For this system, a solution of mercuric chloride is injected onto a heated head to evaporate the solution. Nitrogen carries the gaseous mercuric chloride into the bulk gas. A photo of the HOVACAL installed at the site is shown in Figure 3. The heated head, disconnected from the injection port on the probe, is shown in the right of the photo. The liquid calibration solution is delivered to the head with a peristaltic pump. The pump flow rate is verified using a loss-of-weight balance (shown in upper right of photo). The peristaltic pump feed rate, nitrogen flow rate, and the head temperature are controlled with a touch-screen interface shown on the front of the HOVACAL unit.

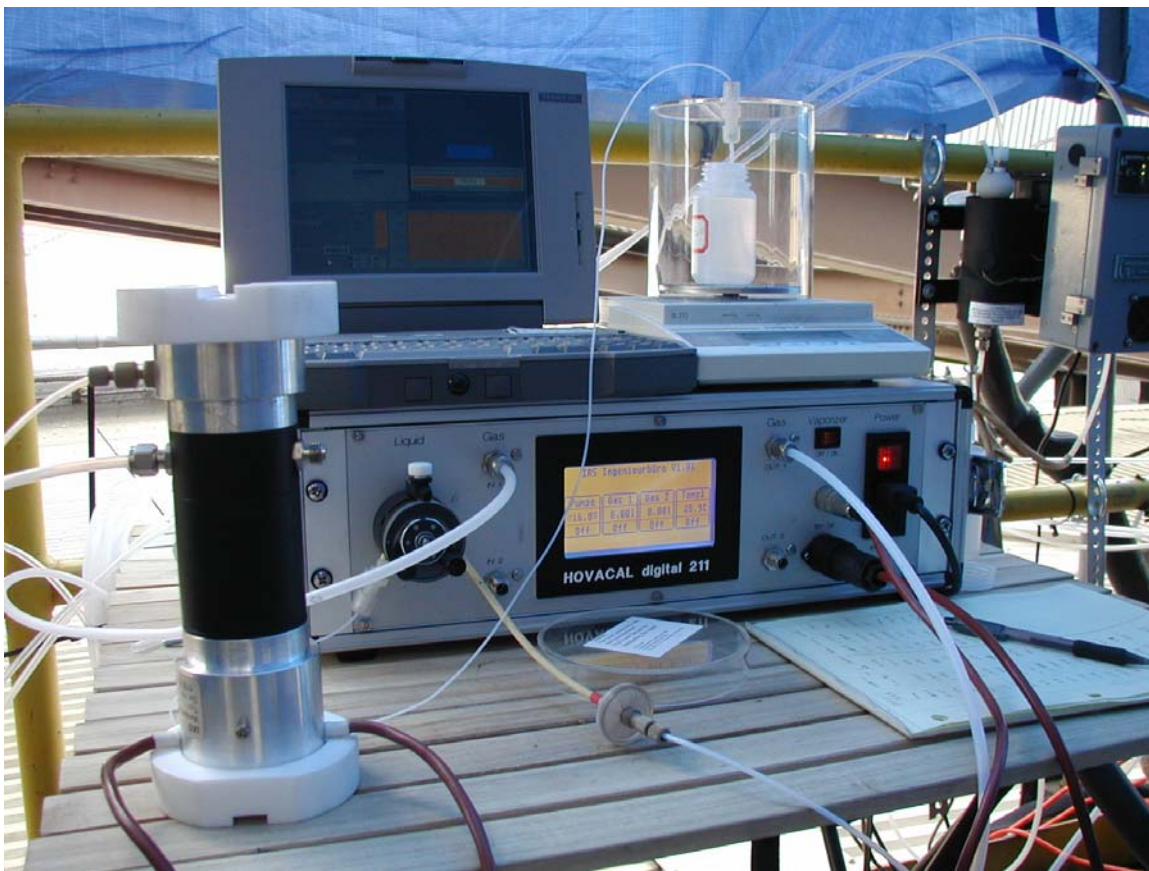


Figure 3. HOVACAL installed at Pleasant Prairie.

EVALUATION PROGRAM

The goal of the evaluation program was to determine the effectiveness of inertial separation probes for both elemental and oxidized mercury transport in a relatively clean gas stream, and to qualitatively assess their ability to inertially separate particles with a high affinity for mercury with minimal sampling artifacts. The general plan was to alternately spike the probes with elemental and oxidized mercury ahead of the filter and then measure the resulting mercury concentrations, and to dope the probes with activated carbon and monitor the effect.

Probe Preparation

Flow Measurement

The oxidized and elemental mercury spiking systems were configured to introduce a small volume of concentrated vapor-phase mercury into the bulk probe flow upstream of the inertial filter. The flow through the probe was monitored to determine the expected mercury concentration in the bulk flow downstream of the spike.

Each probe was retrofitted with a venturi flow meter to monitor the gas flow through the probes. The venturi flow meters were installed at the end of the exhaust return pipe to

eliminate the need to modify the overall probe design or operation. Since compressed air is introduced into the vacuum eductors upstream of the exhaust venturis, it was also necessary to measure the compressed air flow to determine the actual flow through the inertial separation portion of the probes. Two of the probes also included integral venturi flow meters.

Gas Spiking Ports

The Apogee and Thermo probes were retrofitted with spiking ports immediately upstream of the inertial filter to allow spiking with elemental and oxidized mercury. Because high concentrations of oxidized mercury can be difficult to transport, the length of the spike transport line was minimized to prevent losses. The spiking port on two of the probes (the small probe and one of the larger probes) was retrofitted so that the HOVACAL head could be connected directly to the port. The spiking port for the other large probe was located inside the heated enclosure requiring nominally 18 inches of PFA Teflon™ and 6 inches of coated stainless steel transport line prior to entering the bulk probe flow.

Probe Extensions (Stingers)

A 6-foot stinger was installed on the inlet of each extraction probe. For the two higher-flow probes, 3/4-inch pipe was used. Baldwin provided a Durinert™-coated stinger for use with their probe. One-half inch tube was used for the stinger on the lower-flow probe.

Activated Carbon Injection Line

A 1/4-inch stainless steel carrier line was installed along the stinger for activated carbon injection. One end of this line extended into the tip of the stinger. The opposite end terminated outside of the flange to allow doping with activated carbon.

A photograph of the Baldwin probe with the exhaust venturi, probe extension, and calibration line is shown in Figure 4.

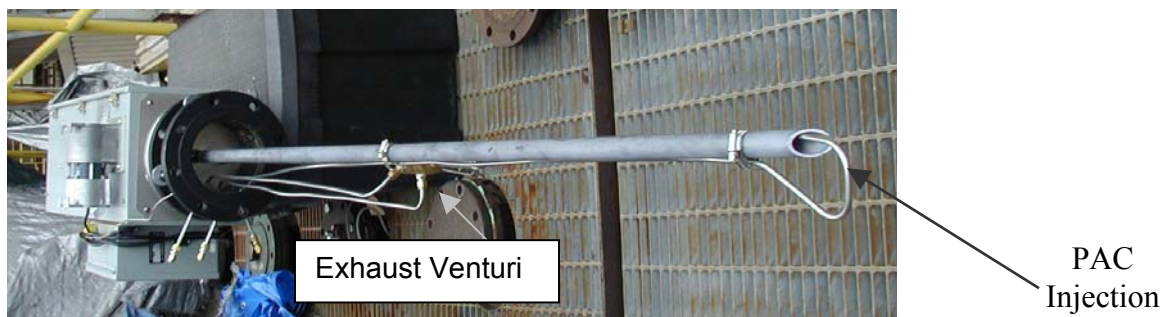


Figure 4. Photo of Baldwin probe with additional monitoring equipment installed.

Test Protocol

The exhaust venturi flow meters and integral venturi flow meters were calibrated using a laminar flow element prior to testing. These calibrations were used to determine the expected vapor-phase mercury concentrations during elemental and oxidized mercury spike test periods. The probes were installed and operating at the manufacturer's recommended operating conditions (flow and temperature) for at least 24 hours prior to testing.

At the beginning of each test day, the Tekran instrument was calibrated with the on-board permeation device. Following calibration, baseline mercury measurements were made to establish the vapor-phase mercury concentration and speciation in the duct. Pleasant Prairie burns a PRB coal. The mercury concentration was fairly stable and the mercury was primarily in the elemental form (typically >80%).

After establishing the baseline conditions, the output from the PSA 10.534 was connected to the calibration port on the extraction probe. Based upon the saturator temperature and flowrate through the PSA 10.534, the mercury introduced to the probe (ng/min) could be calculated. The expected mercury concentration was determined by adding the mercury mass injection rate divided by the probe flow to the baseline duct concentration. Each probe was evaluated at 300 and 400°F.

Following performance evaluations with elemental mercury, the probes were spiked with oxidized mercury from the HOVACAL system. The concentration of oxidized mercury was calculated using the injection rate of the HgCl₂ solution and the flow rate through the probe. Tests were conducted at 300 and 400°F to establish the temperature stability of the probes for oxidized mercury measurements.

The final test was designed to determine the inertial separation effectiveness. Because the three probes were installed at the outlet of the electrostatic precipitator at Pleasant Prairie, the fly ash loading to the probes was quite low. This provided a good opportunity to evaluate the probe for elemental and oxidized mercury measurements without concern for in-duct reactions with the fly ash. However, this test location and the nature of the fly ash at Pleasant Prairie (relatively low affinity for mercury) did not challenge the probes for their ability to adequately separate particulate matter while minimizing the sampling artifacts.

For the final test, NORIT FGD activated carbon was introduced by batch injection into the tip of the probe extension. The injection rate was equivalent to nominally 45 lb/MMacf, which is roughly twice the maximum injection rate economically feasible for an ESP application. The injection was maintained for 30 seconds and repeated at two different operating temperatures (300 and 400°F). Total and elemental mercury measurements were made during and following injection. The residence time in the probe extensions was less than 0.15 seconds for most of the test conditions.

RESULTS

The baseline vapor-phase mercury concentration in the flue gas during testing ranged from 9 to 12 $\mu\text{g}/\text{Nm}^3$. The flue gas flowrate through the larger probes ranged from 10 to 11.5 acfm. The flowrate through the smaller probe ranged from 2.25 to 3.0 acfm. The large probes were operated at their maximum design velocity and the small probe was operated slightly above the design velocity of the system.

Elemental Mercury Spiking

All three probes performed well when elemental mercury was introduced upstream of the inertial filter. The recovery of total mercury was quite good and well within the measurement uncertainty of the probe flow. Both of the uncoated stainless steel probes demonstrated some oxidation of the elemental mercury spike. The larger probe demonstrated somewhat more oxidation (11% at 300°F and 19% at 400°F). The smaller uncoated probe demonstrated slight (3 to 5%) oxidation at 300 and 400°F. The Durinert™-coated probe did not produce any measurable oxidation at either temperature. An example of the mercury concentrations measured during an elemental mercury-spiking period is shown in Figure 5.

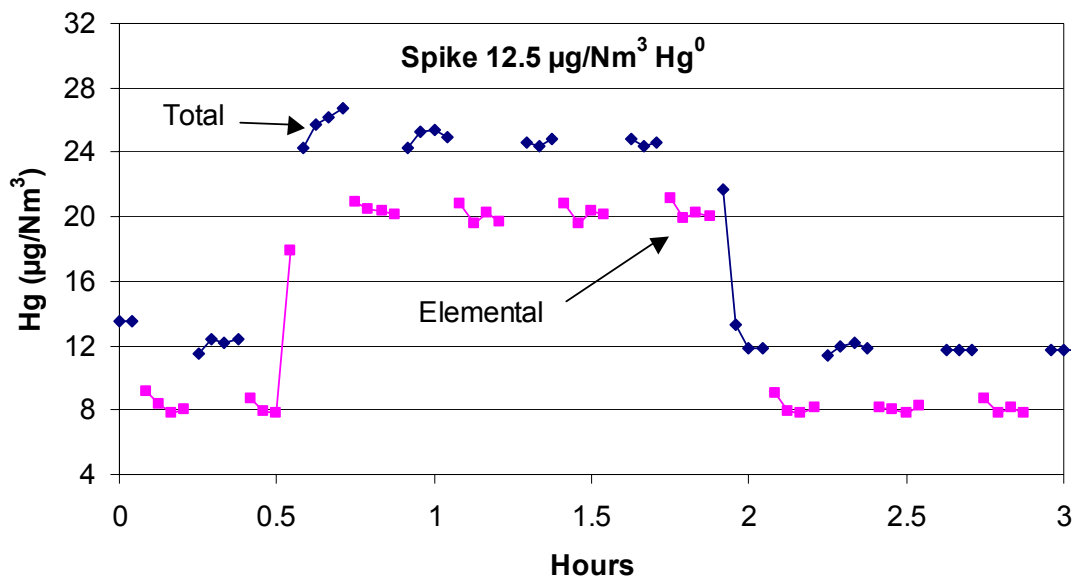


Figure 5. Example of an elemental mercury spike.

Oxidized Mercury Spiking

The recovery of oxidized mercury was very good (>90% of the expected concentration) on both probes that could be close-coupled to the HOVACAL head. For the larger probe, the recovery was slightly lower at 300°F than 400°F. There was no measurable difference in performance between 300 and 400°F for the smaller probe. The smaller probe also resulted in a higher fraction of the sample measured as oxidized (>95% of expected). The oxidized mercury measured with the larger probe was 82 to 85% of the expected concentration. An

example of the mercury concentrations measured during an oxidized mercury-spiking period is shown in Figure 6.

The oxidized mercury spike test for the probe with the calibration port extension was not as successful as the other two probes. Nominally 50% of the spiked concentration was measured when using this probe, and only 30 to 40% of the expected concentration was measured as oxidized mercury. Due to the difficulties transporting oxidized mercury, poor performance may be due to difficulties transporting the spike sample to the calibration port on the probe and not necessarily due to any other aspect of the probe design. Modifications to allow direct coupling of the HOVACAL head to this probe are required to allow an appropriate evaluation of oxidized mercury spiking.

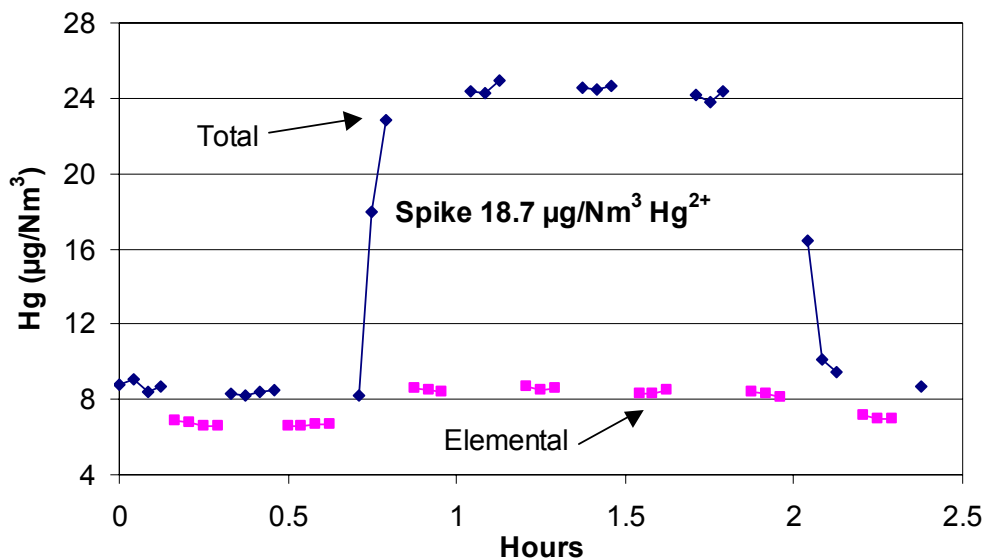


Figure 6. Example of an oxidized mercury spike.

Doping with Activated Carbon

Following characterization with elemental and oxidized mercury spikes, each of the probes was challenged with activated carbon to qualitatively assess the ability of the probes to inertially separate particulate matter with an affinity for mercury.

Both large probes demonstrated a rapid drop in mercury concentration following injection. The concentration immediately following injection was 15 to 60% lower than the initial concentration. The total mercury concentration on one of the large probes returned immediately to the baseline concentration following injection. However, some activated carbon remained on the filter as evidenced by the increased oxidation across the probe (typically 20% additional oxidation following injection). This behavior is shown for probe "C" in Figure 7. In the figure, a batch of activated carbon is added in three distinct episodes. Following testing, the sampling train is moved to another probe. Note the decrease in the fraction of elemental mercury following activated carbon injection. Also note that the

elemental mercury is equal to the total mercury when measured through probe “B”. No carbon injection episodes are shown for probe “B”.

The total mercury concentration measured with the coated probe did not immediately return to baseline concentrations but remained suppressed for up to an hour following activated carbon injection. The fraction of oxidized mercury also increased across this probe following each injection episode.

No drop in mercury concentration or increase in oxidation was noted across the small probe.

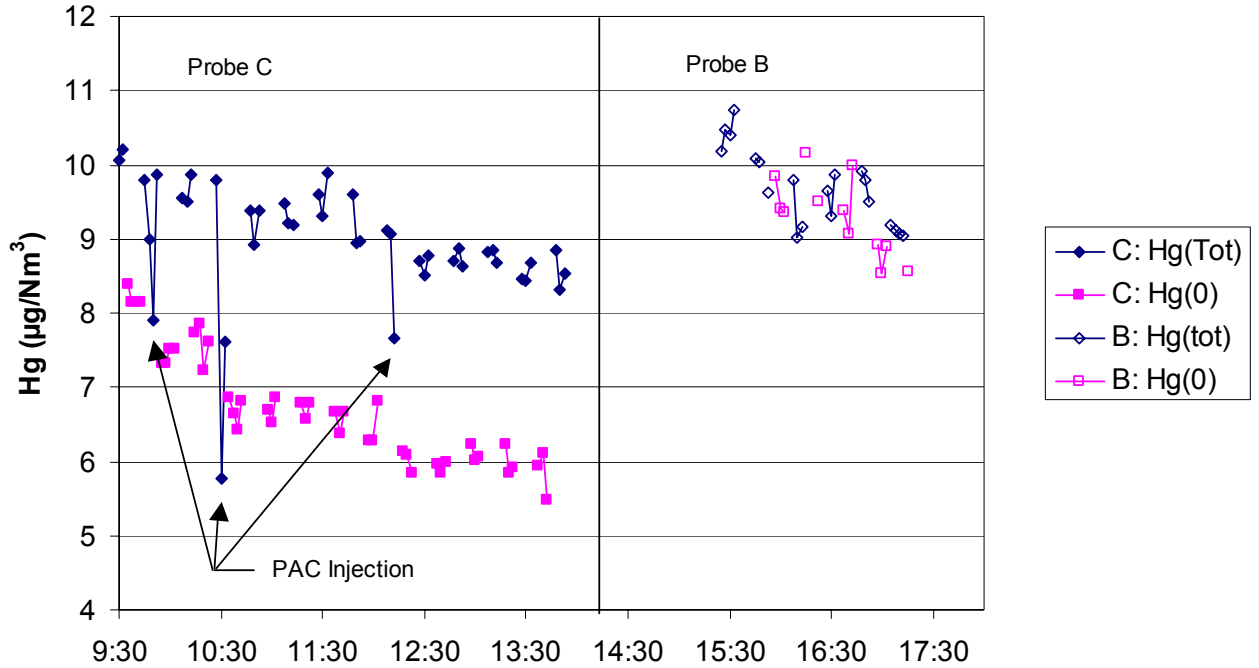


Figure 7. Effect of activated carbon injection into probe “C” on total and elemental mercury concentrations.

CONCLUSIONS

- All three probes demonstrated good performance and could be incorporated for use in a total vapor-phase mercury CEM system.
- The uncoated stainless steel probes demonstrated a potential to oxidize mercury, with the larger probe demonstrating more oxidation than the smaller probe (19% oxidation with large probe and 5% oxidation with small probe at 400°F).
- The recovery of oxidized mercury was very good (>90% of the expected concentration) on both probes that could be close-coupled to the HOVACAL oxidized mercury spiking system. Due to difficulties transporting oxidized mercury to the spiking port on the third probe, the oxidized mercury performance for this probe is inconclusive.
- The probes did not appear to be overly sensitive to operating temperature in the range of 300 to 400°F (there was a slight increase in oxidation noted on uncoated probes at 400°F compared to 300°F).
- Some activated carbon appeared to deposit on the larger probes as demonstrated by an initial drop in the measured mercury concentration and either a prolonged oxidation effect (activated carbon is known to oxidize mercury after reaching saturation capacity) or a period of suppressed total mercury following injection.
- The smaller probe appeared to be effective at minimizing the effect of high-activity particulate (activated carbon) on vapor-phase mercury measurements.

Additional Observations

- An elemental mercury doping system should be incorporated into all mercury CEMs and dynamic spiking should be done on a regular basis to assure proper system performance, particularly when probes are used in gas with active particulate matter when speciated mercury measurements are necessary.
- Dynamic spiking should be conducted in the flue gas matrix to minimize variations in flue gas chemistry that may affect the performance of the pretreatment system or transport assembly.
- Bottled calibration gases may not be appropriate for this application due to the low mercury concentration limits of bottled gases.
- Spiking with oxidized mercury should be conducted on a periodic basis to ensure proper system operation. Additional tests must be conducted to ensure stability of HOVACAL and develop a more robust system, or other systems must be demonstrated.

REFERENCES

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