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Evaluation of Sorbent Trap Materials and Methods for Flue Gas Mercury Measurement

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PRODUCT DESCRIPTION

Glass tubes containing sorbent (sorbent traps) are used as alternatives to continuous mercury (Hg) monitors on coal-fired power plants and other emission sources. This project involved evaluation of sorbents in the laboratory, following a stack gas sampling method included in the vacated U.S. Environmental Protection Agency (EPA) Clean Air Mercury Rule, Appendix K. Sorbents evaluated include iodated, brominated, and non-halogenated types with either coconut shell or bituminous coal precursor materials. Gas exposure tests helped evaluate sorbent performance, using simulated coal combustion flue gas. This report summarizes research to determine the performance of activated carbon sorbents currently used in Hg flue gas monitoring.

Results and Findings

The project included a number of sorbent materials that provide good adsorption of Hg for use in Appendix K sampling. Tests indicated that the halogenated sorbent types provided better performance for long-term exposure. The most recent tests evaluated possible interferences, particularly SO_3, that might limit the application of sorbent tube sampling for Hg compliance measurements. The tests indicated that within the expected flue gas conditions, the sorbent tube materials most commonly used today will meet or exceed the EPA performance criteria for Appendix K compliance measurements.

Challenges and Objective(s)

The sorbent trap method has been the subject of concerted development efforts over the past several years by EPA, EPRI, and sorbent vendors. At the time this project commenced, only one vendor's sorbent was in use in sorbent traps; however, the performance-based Appendix K method allows for the use of other sorbents. Given the potential market demand for Appendix K monitoring, it was advantageous to have a greater choice of sorbent trap providers. Furthermore, there was little information available about the performance of any of the potential Appendix K sorbents across the wide range of flue gas conditions encountered at coal-fired power plants. EPRI therefore initiated a multi-year effort to evaluate performance of sorbents for use in Hg monitoring.

Applications, Values, and Use

This project facilitated the development of sorbent tube sampling of Hg by identifying multiple sources of materials and expanding the number of available suppliers. As the sorbent tube application for Hg measurement has matured, it has provided a valuable alternative to the more expensive continuous emission monitors being developed. This work has continued to validate and increase confidence in the Appendix K approach to Hg measurement. As Hg control technologies improve and emissions levels continue to decrease, the detection limit achieved by sorbent tube methods will become increasingly important for many emission sources.

EPRI Perspective

EPRI has been the primary source for objective evaluation of sorbent materials produced by different vendors for use in Hg measurement systems. In addition, EPRI's involvement in both field and laboratory testing of the Appendix K method has allowed the Institute to identify and research potential interferences that could impact method accuracy.

Approach

Two types of gas exposure tests were conducted to evaluate sorbent performance: adsorption tests and desorption tests. Adsorption tests were designed to evaluate the ability of sorbents to capture and retain Hg on the first (leading) sorbent bed in a multi-bed sorbent trap. These tests were conducted by exposing sorbent traps to a simulated Hg-laden coal combustion flue gas. Desorption tests were designed to detect loss of Hg from a pre-spiked third sorbent bed, a quality control measure required by the method. Appendix K specifies acceptance criteria for retention of the Hg spike during sampling; if the spike is not retained, the test is invalidated. Desorption tests were conducted by exposing pre-spiked Appendix K traps to a simulated Hg-free flue gas containing SO_3.

Keywords

Mercury Measurement
Appendix K Method
Performance Specification 12B
Flue Gas Emissions
Sorbent Performance
Sorbent Tube Sampling

ABSTRACT

This report summarizes research to evaluate the performance of activated carbon sorbents used in mercury flue gas monitoring. Glass tubes containing sorbent (sorbent traps) are used as alternatives to continuous mercury monitors on coal-fired power plants and other emission sources. Sorbents were evaluated in the laboratory, following a stack gas sampling method included in the vacated U.S. Environmental Protection Agency Clean Air Mercury Rule, Appendix K. Sorbents evaluated include iodated, brominated, and non-halogenated types with either coconut shell or bituminous coal precursor materials. Gas exposure tests were run to evaluate sorbent performance, using a simulated coal combustion flue gas.

Two types of gas exposure tests were conducted to evaluate sorbent performance: adsorption tests and desorption tests. Adsorption tests were designed to evaluate the ability of sorbents to capture and retain mercury on the first (leading) sorbent bed in a multi-bed sorbent trap. These tests were conducted by exposing sorbent traps to a mercury-laden simulated coal combustion flue gas. Desorption tests were designed to detect loss of mercury from a pre-spiked, third sorbent bed, a quality control measure required by the method. Appendix K specifies acceptance criteria for retention of the mercury spike during sampling; if the spike is not retained, the test is invalidated. The desorption tests were conducted by exposing pre-spiked Appendix K traps to a mercury-free simulated flue gas.

Adsorption and desorption tests were conducted with simulated flue gases containing sulfur dioxide and sulfur trioxide, to evaluate the impact of these gas components on sorbent mercury capture and retention. Laboratory tests were conducted to determine optimum conditions for spiking mercury onto Appendix K traps, including spike placement and spiking temperature.

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INTRODUCTION AND BACKGROUND

Sorbent traps are used to measure mercury (Hg) in flue gas from combustion sources such as coal-fired power plants. Flue gas is pulled through a glass tube containing a sorbent material with an affinity for mercury. After sampling, the collected mercury is separated from the sorbent by heating, combustion of the sorbent, or solvent extraction, and is measured by a mercury detector.

The U.S. Environmental Protection Agency (EPA) published a sorbent trap method, referred to as "Appendix K", as an alternative to continuous mercury monitors (CMM) for measuring vapor phase mercury concentrations in stacks of coal-fired power plants. Typically, paired samples are collected over periods of days to a week

Appendix K was contained in the Clean Air Mercury Rule (CAMR) of 2005, which was subsequently vacated by the D.C. Circuit Court; thus, it is not an official EPA method. However, it should be noted that at the time of this report, EPA has proposed a new rule for cement kilns. If this proposed rule ("National Emission Standards for Hazardous Air Pollutants From the Portland Cement Manufacturing Industry") is promulgated in the Federal Register, all of the 40 CFR Part 75 Appendix K requirements and associated performance specifications for sorbent trap monitoring would be moved to 40 CFR part 60 Appendix B, Performance Specification 12B.

The sorbent trap method has been the subject of concerted development efforts over the past several years by EPA, EPRI, and sorbent vendors. At the time this project commenced, only one vendor's sorbent was in use in sorbent traps; however, the performance-based Appendix K method allows for the use of other sorbents. Given the potential market demand for Appendix K monitoring, it was advantageous to have more choice of sorbent trap providers. Furthermore, there was little information available about the performance of any of the potential Appendix K sorbents across the wide range of flue gas conditions encountered at coal-fired power plants. Therefore, EPRI initiated a multi-year effort to evaluate performance of sorbents for use in mercury monitoring. This report summarizes the research conducted by URS for EPRI from 2005 through 2009.

Project Objectives

The objective of this program was to identify two or three mercury sorbent materials most suitable for use in Appendix K and evaluate sorbent performance of the selected sorbent materials in laboratory tests using simulated coal combustion flue gas. Data obtained from Appendix K traps must meet several acceptance criteria to be considered valid and reportable. The laboratory tests were designed to test the ability of each sorbent to meet these criteria after exposure to different flue gas conditions.

Background

A discussion of the Appendix K method and the general technical approach for the project is provided below.

Description of the Appendix K Method

Appendix K sampling technology involves the use of manual sampling equipment to sample flue gas from a stack duct, in order to measure vapor-phase mercury A known volume of flue gas is pulled across a pair of sorbent traps housed in a heated probe. The sorbent traps contain three discrete layers or beds of sorbent, each of which has a different purpose. After the sampling period, the sorbent traps are removed from the duct and each sorbent bed is analyzed for mercury content. The Appendix K method thus provides an average mercury concentration for the flue gas over the sampling period, which can last for several days to more than a week. The mercury analysis can take place either on site (allowing for quicker turnaround) or at an off-site laboratory, in which case there may be a delay of several days to obtain a measurement.

A sorbent trap monitoring system, pictured in Figure 1-1, consists of the components below:

- Sorbent traps: paired sorbent traps are placed within a probe and inserted directly into the flue gas stream.
- Sampling probe assembly
- Moisture removal device
- Vacuum pump
- Dry gas meter
- Sample flow rate and controller
- Temperature sensor
- Barometer
- Data logger

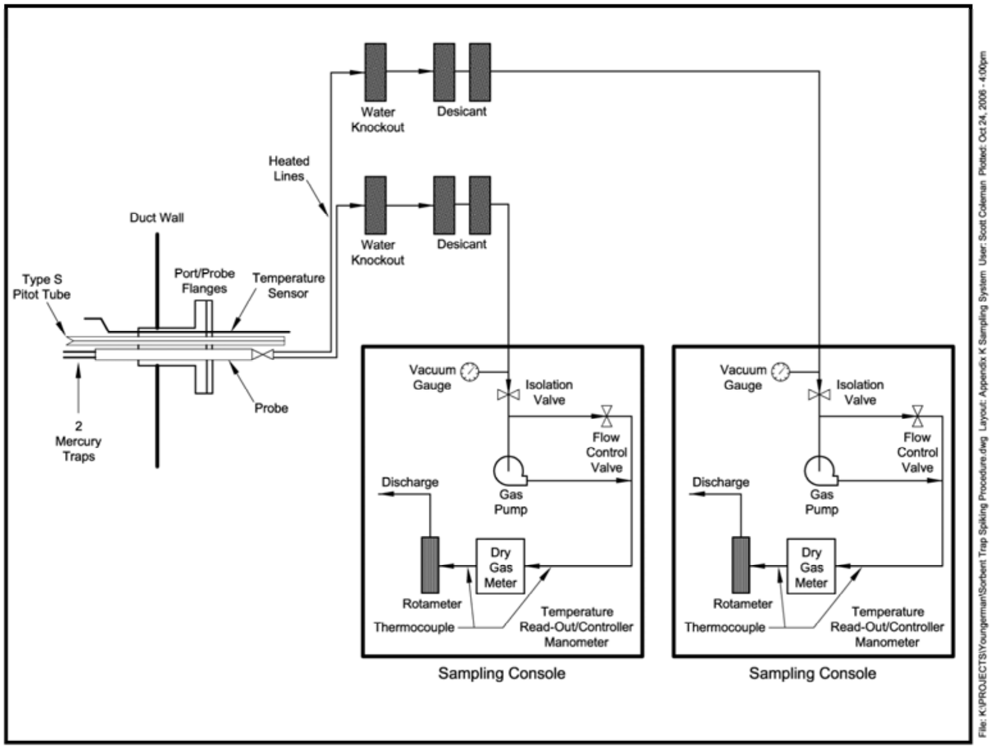

Figure 1-1
Diagram of a Sorbent Trap Sampling System

The Appendix K method specifies quality control (QC) requirements, including:

- Pre-test and post-test leak checks less than 4% of sampling rate;
- Proportional sampling within 25% of initial sampling rate;
- Breakthrough of mercury to second bed below 5% of the mass on the first bed;
- Paired sorbent trap agreement with less than 10% relative deviation;
- Spike recovery study giving recovery between 85 and 115% at three spike levels;
- Daily multipoint analyzer calibration with each reading within 10% of true value and r^2 greater than 0.99;
- Analysis of independent calibration standard within 10% of true value;
- Recovery of spiked mercury from the third bed between 75 and 125%; and
- A relative accuracy test audit (RATA).

Spiked sorbent traps that meet the QA/QC criteria of Appendix K can be purchased from a vendor. The recovered sorbent traps must be analyzed by a certified laboratory that meets the requirements of ISO 17025. Alternatively, if the laboratory performs a spike recovery study (described in Section 10.3 of Appendix K) and repeats this procedure annually, ISO certification is not required. Any analytical system capable of quantitatively recovering and quantifying total gaseous mercury from sorbent media is acceptable, provided it meets the specifications of Section 8 of Appendix K. Examples of recovery techniques include acid digestion and thermal desorption. Examples of analytical quantification techniques include atomic fluorescence (AF) and atomic absorption (AA).

It should be noted that the EPA does not stipulate nor endorse any particular sorbent for use in Appendix K. While there was only one supplier of packed, spiked sorbent tubes at the commencement of this study, there are currently several vendors offering packed, spiked sorbent tubes. Each utility is responsible for validating and certifying the sorbent selected to be used at its plant(s), so it is important for a utility to ensure that proper analytical procedures are used for a given sorbent type.

There are several different types of activated carbon that are in use for Appendix K measurements and it is likely that additional types will be developed over the next few years. The site-specific flue gas characteristics will have an impact on sorbent selection, as some sorbents may exhibit better performance than others at extremes of sulfur dioxide (SO_2) or sulfur trioxide (SO_3) concentration, moisture, gas exposure time, and possibly stack gas temperature.

Appendix K Trap Design

As Appendix K does not specify the sorbent material to be used in the traps, selection of a sorbent is based on the material's ability to achieve the quality assurance and quality control parameters identified in Section 8 of Appendix K. A sorbent viable for use in this method needs to be free of background mercury contamination, able to adsorb mercury from mercury-laden flue gas, and able to retain pre-spiked mercury during exposure to flue gas.

Figure 1-2 illustrates the design of a trap used in Appendix K sampling. The sorbent medium used to collected mercury must be configured in three distinct and identical segments (typically 1.0 g of sorbent). Appendix K requires each trap contain two beds of clean sorbent followed by a third bed that has been pre-spiked with a known amount of mercury.

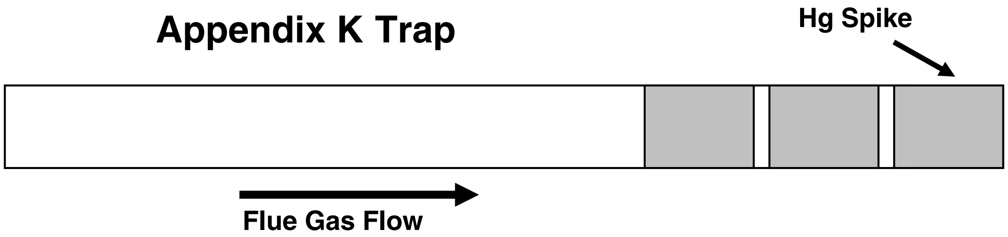

Figure 1-2
Diagram of an Appendix K Trap

The purpose of each trap section, and acceptance criteria for each section are as follows:

- Section 1 is designated for primary capture of gaseous mercury.
- Section 2 is designated as a backup section for determination of vapor-phase mercury breakthrough. The Appendix K acceptance criterion allows less than 5% of the total mercury captured on the first sorbent bed to break through to the second sorbent bed:

  % breakthrough = (ng Hg measured on Section 2 / ng of Hg measured on Section 1) *100

- Section 3 is designated for QA/QC purposes. This section is spiked with a known amount of gaseous elemental mercury prior to sampling (any spiking approach capable of quantitatively delivering a known mass of elemental mercury to the trap is acceptable). Recovery of spiked mercury post-sampling must fall between 75 and 125%.

  % Recovery of Section 3 Hg = (ng Hg on bed 3 / true ng value of mercury spiked) * 100

Project Technical Approach

Several potential sorbents were identified and subjected to analytical pre-screening tests. The sorbents that passed the analytical pre-screening tests were configured in gas exposure tests designed to evaluate sorbent performance in simulated flue gas at flow rates and temperatures that would be encountered during a typical Appendix K run.

Report Organization

Section 2 of this report provides the details of the experimental procedures that were used, and Section 3 presents the results. Section 4 discusses the conclusions of the test program.

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EXPERIMENTAL DESIGN

This section describes the experimental procedures used to conduct this sorbent evaluation study. This study has consisted of four phases of testing. Table 2-1 describes each phase and the year the tests were completed.

Table 2-1
Summary of Test Phases

Test Phase	Description	Year Completed
I	Initial Sorbent Screening	2006
II	Spike Optimization	2007
III	Impact of SO_2	2008
IV	Impact of SO_3	2009

The first part of this section summarizes the experimental approaches for Phases I, II, and III. These phases were reported previously [1,2,3], thus the experimental details of these phases will be discussed in less detail in this report. The second part of this section describes the experimental details of the Phase IV tests, which have not been reported previously.

Phases I through III

A discussion of prescreening sorbent selection and various types of tests conducted during Phases I through III is provided below, followed by a brief summary of the testing conducted in each phase.

Sorbent Prescreening and Test Descriptions

In Phase I of this project, a list of potential sorbents for evaluation in this study was generated; the list is provided in Appendix A. Sorbents were selected from this list for evaluation based on cost, commercial availability, and sorbent composition. The selected sorbents were analyzed for background mercury concentration. Sorbents with no detectable background mercury were retained for further testing. The sorbents were also screened for their ability to adsorb mercury. This screening test was performed by spiking a known quantity of mercury on the sorbents and analyzing for mercury recovery.

Sorbents that passed the analytical pre-screening tests were subjected to laboratory adsorption and desorption tests designed to evaluate sorbent performance after exposure to a simulated flue gas at sample flowrates, temperatures, and time periods similar to those that would be encountered in an Appendix K test. Sorbent performance was judged on the ability of the sorbent to capture and retain mercury on the first sorbent bed (adsorption) and to retain mercury on the pre-spiked third bed when exposed to simulated flue gas (desorption). As the work progressed, some sorbents were eliminated from the test program based on poor performance and new sorbents were added to the test program based on commercial availability. Thus, the sorbents evaluated were not constant over the different phases of the test program.

Adsorption Tests

Adsorption tests, used to determine the ability of a sorbent to capture mercury, were conducted by exposing sorbent traps containing two beds of clean sorbent to a mercury-laden simulated synthetic flue gas. Miniature impingers with catch solutions analogous to a EPA Method 29 sampling train were placed downstream of the sorbent traps to monitor mercury levels in the trap effluent gas. The ability of each sorbent to adsorb and retain mercury from the simulated flue gas was based on results of the mercury mass recovery on sorbent bed 1, and the percentage of mercury that broke through the first sorbent bed to the second sorbent bed and the amount passing through both beds into the catch solutions. A sorbent acceptable for use in Appendix K will not allow more than 5% of the captured mercury to pass through the first sorbent bed.

Desorption Tests

Desorption gas exposure tests made use of three-section Appendix K traps spiked with elemental mercury on the third bed. The tests were conducted by exposing the pre-spiked traps to a mercury-free flue gas. According to the Appendix K acceptance criteria, tests are invalid if the mercury mass recovered from the third bed after sampling falls outside ± 25% of the true (spiked) value. Two types of desorption tests were conducted. In real-time flue gas exposure screening tests, desorption of mercury from the third spiked bed was monitored using an online monitor to determine the rate of desorption over time. This test configuration only allowed one trap at a time to be monitored. The tests were run until 25% of the pre-spiked mercury mass was desorbed or for seven days, whichever timeframe was the shortest. Figure 2-1 shows a diagram of the apparatus used to conduct the real-time screening tests.

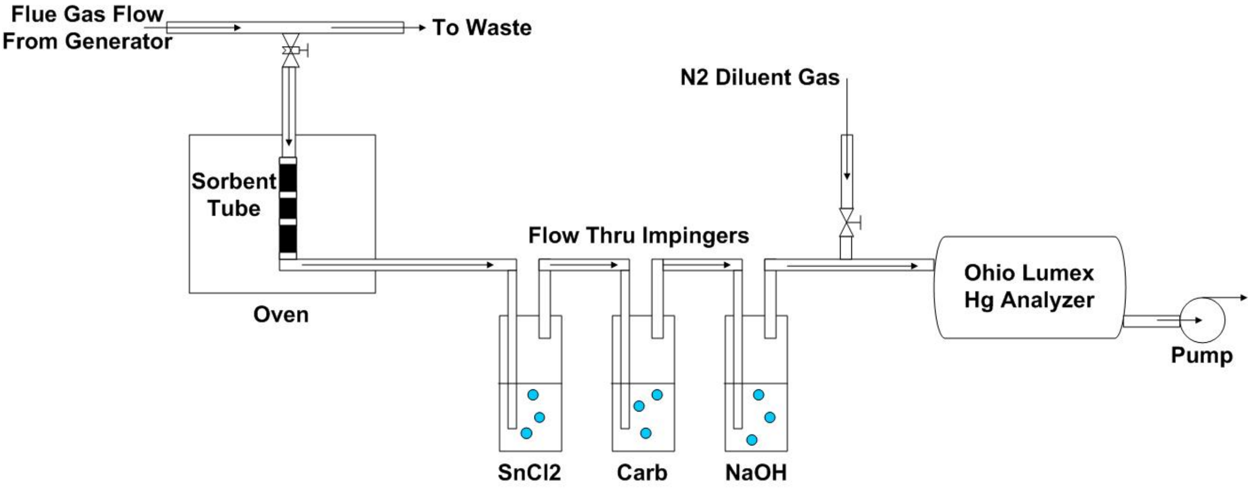

Figure 2-1
Schematic of Real-Time Flue Gas Exposure Test Apparatus

The second type of desorption test used a multi-trap configuration that allowed multiple traps to be exposed to mercury-free flue gas in parallel. Mercury desorption was evaluated by measuring the mercury mass on the pre-spiked third bed after gas exposure, and mercury loss was confirmed by the presence of mercury in downstream impinger catch solutions. Figure 2-2 shows a diagram of the multi-trap gas exposure test apparatus. This apparatus was also used to conduct adsorption gas exposure tests.

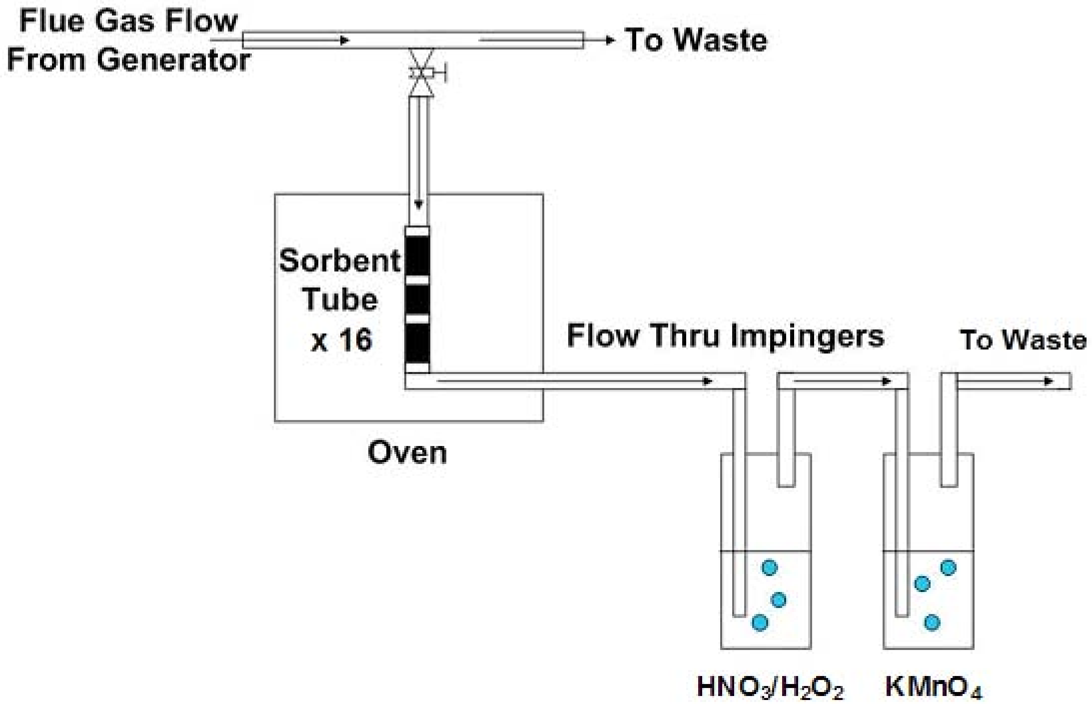

Figure 2-2
Schematic of Multi-trap Gas Exposure Test Apparatus

The main differences between the two desorption test configurations were the mercury measurement equipment located downstream of the traps and the number of traps tested simultaneously. In both test configurations, sorbent traps were placed in an oven and approximately 400 mL/min of simulated flue gas was pushed through each sorbent trap. The effluent gas exited the oven and bubbled through catch solutions configured analogous to a Method 29 train, which were used to capture any mercury that passed through or desorbed from the sorbent traps.

Simulated Flue Gas System

A schematic of the system used to generate the simulated synthetic flue gas for all gas exposure tests is shown in Figure 2-3. Gas settings and pressures were monitored daily during each test to ensure that the make-up of the simulated flue gas remained the same and within specifications to produce the expected gas component concentrations. The main components of the simulated synthetic flue gas were sulfur dioxide (SO_2), nitric oxide (NO_x), hydrochloric acid (HCl), carbon dioxide (CO_2), oxygen (O_2), and water vapor (H_2O).

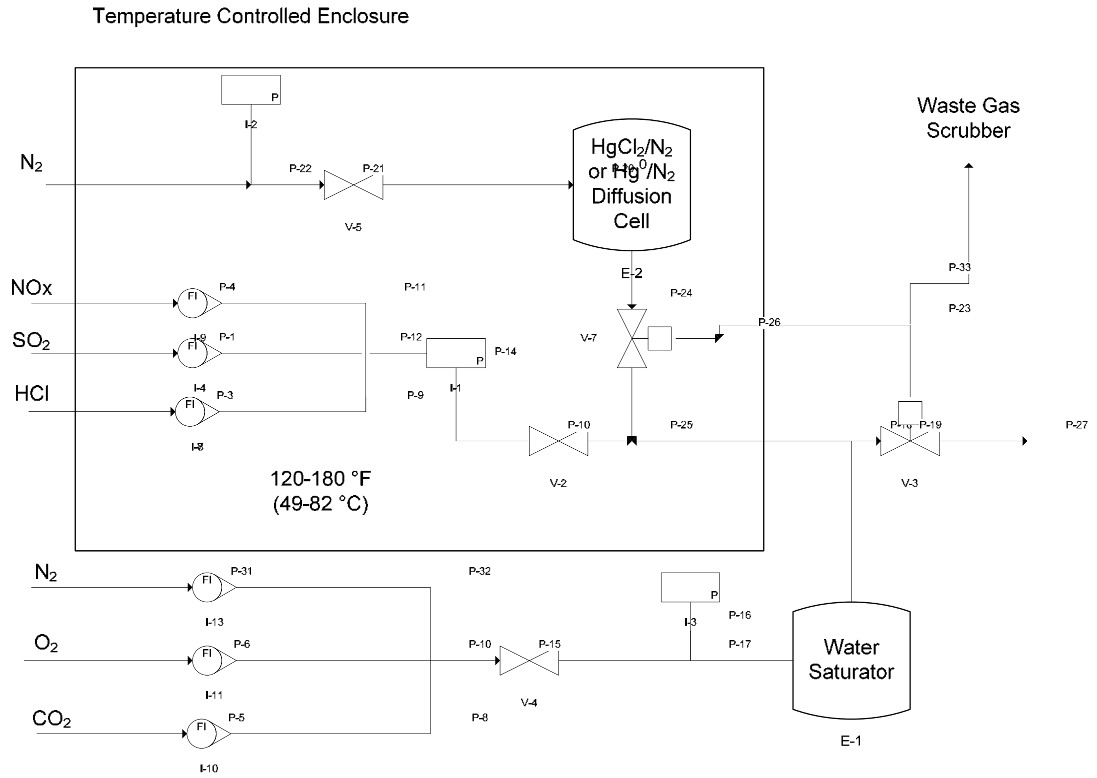

Figure 2-3
Schematic of URS Bench Scale Test Unit Used to Generate Simulated Flue Gas

Spike Placement

Spike placement on the third sorbent bed was a parameter evaluated in the desorption tests. Three types of spike configurations were tested: front-half (FH), back-half (BH) and batch (batch) spikes. Front-half and back-half spikes are prepared via the stannous chloride ($SnCl_2$) dynamic spiking method, as described in an earlier report [2]. In a front-half spike configuration, a known mercury mass is loaded onto the front portion of the third sorbent bed. Figure 2-4 illustrates the location of the mercury on the third sorbent bed in an Appendix K trap spiked using a front-half configuration in relation to the direction of flue gas flow during testing.

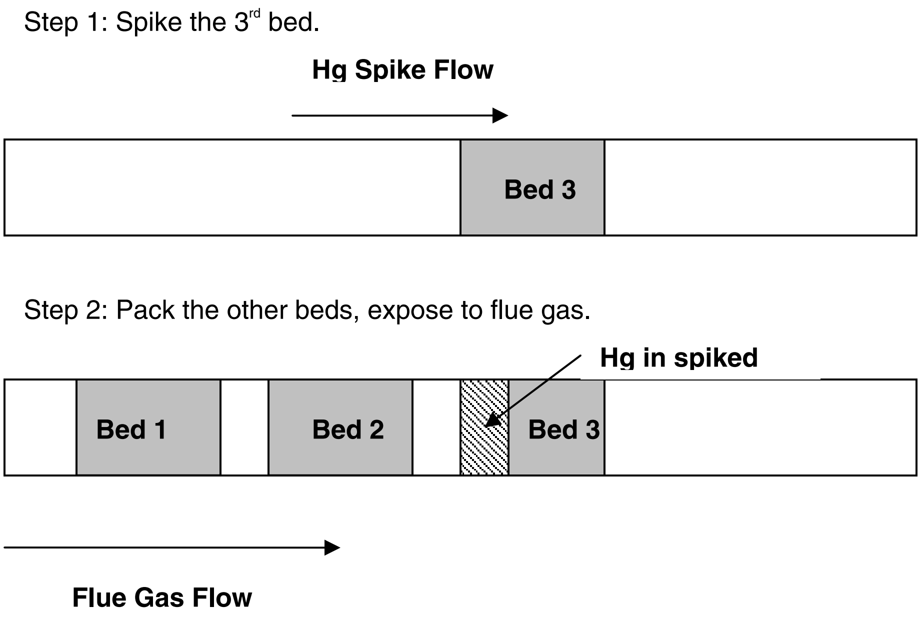

Figure 2-4
Schematic of a Trap Spiked in the Front-Half Configuration

In a back-half spike configuration, a known mercury mass is loaded onto the back portion of the third sorbent bed. Figure 2-5 illustrates the location of the mercury on the third sorbent bed in an Appendix K trap spiked using a back-half configuration in relation to the direction of flue gas flow during testing.

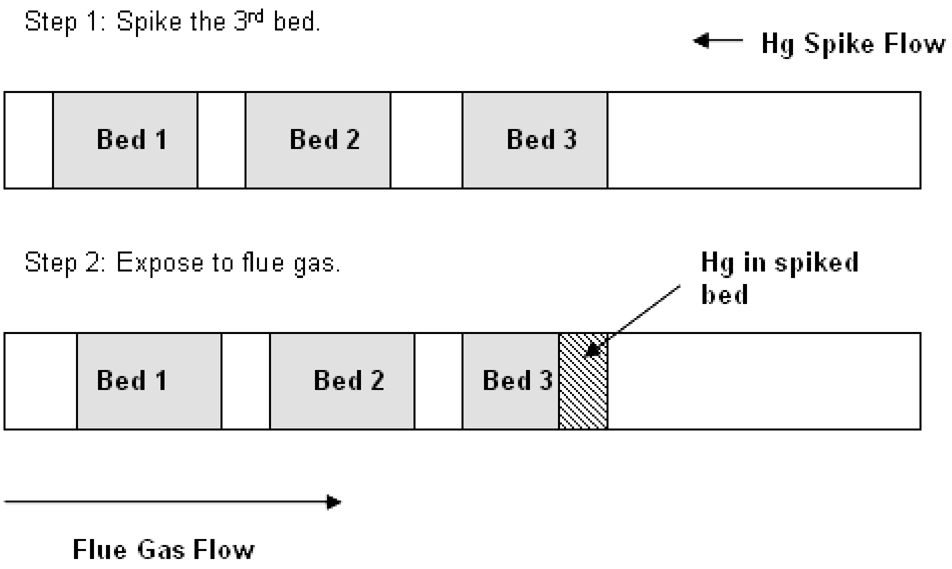

Figure 2-5
Schematic of a Trap Spiked in the Back-Half Configuration

Batch spikes were prepared by sorbent vendor SKC. In a batch spiking process, batches of carbon sorbent are spiked with mercury-laden gas using a fluidized bed apparatus, and it is assumed that the mercury adsorbs homogeneously to all carbon particles in the batch. Figure 2-6 illustrates the location of the mercury on the third sorbent bed in an Appendix K trap spiked using a batch methodology in relation to the direction of flue gas flow during testing.

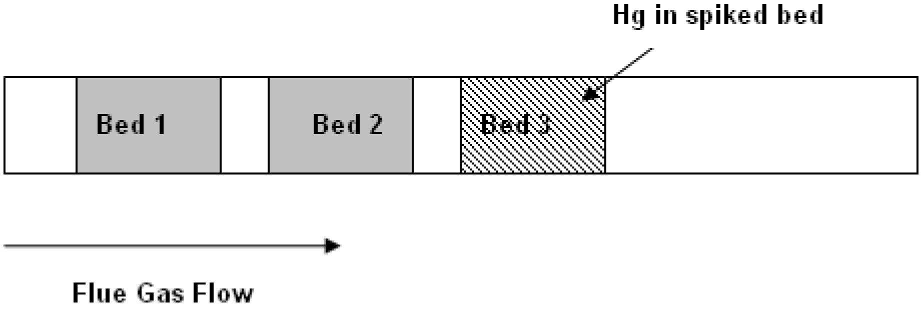

Figure 2-6
Schematic of a Trap Containing Sorbent Spiked using a Batch Methodology

Phase I – Initial Screening Tests

In Phase I, four tests were conducted to screen the performance (adsorption and desorption) of several sorbents that passed the initial analytical screening tests. A high level mercury spiking test was designed to evaluate the mercury capacity of the carbons at favorable conditions; thus, mercury was loaded onto granulated sorbent traps in air and at room temperature. Two adsorption gas exposure tests were conducted with flue gas conditions simulating an unscrubbed stack and scrubbed stack. The results allowed for a comparison of the effects of high versus low SO_2 and moisture on the mercury adsorption capacity of the sorbents. One desorption test, a mercury mobilization test, was conducted with flue gas conditions simulating an unscrubbed stack gas. Table 2-2 summarizes the flue gas parameters used in each test. Table 2-3 summarizes the trap configuration used in the mercury mobilization test. The unscrubbed and scrubbed stack tests were adsorption gas exposure tests, thus tested traps contained two beds of clean sorbent and no pre-spiked bed.

Table 2-2
Summary of Test Parameters for Phase I Tests

Gas Condition	Test ID Unscrubbed Stack	Scrubbed Stack	Mobilization Test
SO_2 (ppm)	1550	200	1000
NO_x (ppm)	340	100	200
HCl (ppm)	45	<1	50
CO_2 (%)	10	11	10
O_2 (%)	5	5	5
H_2O (%)	5	15	7
Hg (µg/Nm^3)	15.6-17.4	15.7-20.2	0
SO_3 (ppm)	0	0	0
Test Temperature (^oF)	265	250	265
# sorbents tested	5	7	6
Gas flowrate across traps (mL/min)	400	400	400
Trap exposure time (days)	9.1	9.8	10.5

Table 2-3
Summary of Trap Configuration for Mercury Mobilization Desorption Test

Sorbent Vendor/Sample ID	Sorbent Impregnate	Spike Placement	Spike Temperature	Hg Spike (ng)
Norit - Darco	none	BH, FH	Room Temp	55,000
Carbochem - TOG	none	BH	Room Temp	55,000
Frontier Geosciences	iodated	BH	Room Temp	55,000
SKC Inc.	iodated	BH, FH	Room Temp	55,000
Ohio Lumex	iodated	BH	Room Temp	55,000
Sorbent Technologies	brominated	BH, FH	Room Temp	55,000

BH – back half, FH – front half

Phase II - Spike Optimization Tests

Phase I results raised the concern that the third-section mercury spike, required in Appendix K traps, could be desorbed during sampling. Spike loss is a particular concern for Appendix K tests, which can be a week or more in duration. A series of laboratory tests was planned to monitor the rate of mercury loss, and to determine whether desorption could be reduced by optimizing spiking conditions (spike placement and spiking temperature).

Traps were spiked with elemental mercury, varying the spike location on the sorbent bed and the temperature of the sorbent during spiking. The mobility of the mercury spike was tested in both types of desorption gas exposure tests. In the first set of tests, the rate of mercury loss was monitored continuously while traps were exposed to flue gas using the real-time screening gas exposure test configuration. A second round of gas exposure testing was conducted to ensure the reproducibility of the results obtained from the first set of tests. Additional spiked traps were exposed to flue gas using the multiple-trap configuration, in which total mercury loss was determined after seven days of exposure. Table 2-4 summarizes the test parameters for the Phase II desorption tests. Table 2-5 summarizes the trap configurations evaluated in the real-time screening gas exposure test, and Table 2-6 summarizes the trap configurations evaluated in the additional desorption multi-trap gas exposure test.

Table 2-4
Summary of Test Parameters for Phase II Desorption Tests

Gas Condition	Real-time Tests	Multiple Trap Desorption Tests
SO_2 (ppm)	1000	1000
NO_x (ppm)	200	200
HCl (ppm)	50	50
CO_2 (%)	12	12
O_2 (%)	6	6
H_2O (%)	6	6
Hg (µg/Nm^3)	0	0
SO_3 (ppm)	0	0
Test Temperature (°F)	300	300
# sorbents tested	2	2
Gas flowrate across traps (mL/min)	400	400
Trap exposure time (days)	*	7

*Traps were exposed until 25% of the pre-spiked mercury desorbed, or for seven days, whichever timeframe was shortest

Table 2-5

Summary of Trap Configurations for Real-Time Screening Gas Exposure Test

Sorbent Vendor/Sample ID	Sorbent Impregnate	Spike Placement	Spike Temperature	Hg Spike (ng)
Sorbent Technologies	brominated	BH	Room Temp	40,000
Sorbent Technologies	brominated	BH	250º F	40,000
Sorbent Technologies	brominated	FH	Room Temp	40,000
Ohio Lumex	iodated	BH	Room Temp	40,000
Ohio Lumex	iodated	BH	250º F	40,000
Ohio Lumex	iodated	FH	Room Temp	40,000

BH – back half, FH – front half

Table 2-6
Summary of Trap Configurations for Phase II Desorption Multi-Trap Gas Exposure Tests

Sorbent Vendor/Sample ID	Sorbent Impregnate	Spike Placement	Spike Temperature	Hg Spike (ng)
Sorbent Technologies	brominated	FH	Room Temp	40,000
Ohio Lumex	iodated	FH	Room Temp	40,000

FH – front half

Phase III – Impact of SO_2 on Sorbent Performance

Observations made during the Phase II studies, as well as results from earlier EPRI test programs[4], indicate that mercury desorption could be associated with the concentration of flue gas constituents, particularly SO_2. Thus, an additional phase of laboratory testing was conducted to determine the concentration of SO_2 that might cause desorption of mercury from pre-spiked beds. Multi-trap, gas exposure desorption tests were conducted in simulated flue gas containing SO_2 levels ranging from 200-1000 ppm; all other experimental parameters were held constant. Table 2-7 summarizes the test parameters for the four desorption tests conducted in Phase III. Table 2-8 summarizes the trap configurations evaluated.

Table 2-7

Summary of Test Parameters for Phase III Impact of SO_2 Desorption Tests

	Impact of SO_2 Test 1	Impact of SO_2 Test 2	Impact of SO_2 Test 3	Impact of SO_2 Test 4
SO_2 (ppm)	1000	500	350	200
NO_x (ppm)	200	200	200	200
HCl (ppm)	50	50	50	50
CO_2 (%)	12	12	12	12
O_2 (%)	6	6	6	6
H_2O (%)	6	6	6	6
Hg (µg/Nm^3)	0	0	0	0
SO_3 (ppm)	0	0	0	0
Test Temperature (°F)	300	300	300	300
# sorbents tested	2	4	4	4
Gas flowrate across traps (mL/min)	400	400	400	400
Trap exposure time (days)	7	7	7	7

Table 2-8
Summary of Trap Configurations for Impact of SO_2 Desorption Tests

Sorbent Vendor/Sample ID	Sorbent Impregnate	Spike Placement	Spike Temperature	Hg Spike (ng)
Sorbent Technologies	brominated	FH	Room Temp	40,000
Ohio Lumex	iodated	FH	Room Temp	40,000
Frontier Geosciences	iodated	FH	Room Temp	40,000
SKC Inc.	iodated	FH	Room Temp	40,000
SKC Inc.	iodated	batch	NA	60,000

FH – front half

Phase IV – Impact of SO_3 on Sorbent Performance

Over the past few years, field data obtained by several research groups have shown a significant negative effect of SO_3 on mercury capture when powdered activated carbon (PAC) is injected into flue gas ducts for mercury control. In Phase IV, which was completed in 2009, laboratory tests were conducted to determine the impact of SO_3 on both adsorption of mercury to a clean sorbent bed and desorption of mercury from the pre-spiked beds.

Multi-tube gas exposure tests were conducted to compare the effects of SO_3 on sorbent performance. A baseline test with no SO_3 present was conducted to verify that selected baseline conditions would not adversely affect sorbent performance. Baseline conditions were selected based on results from previous tests. Adsorption and desorption gas exposure tests were conducted in simulated flue gas containing 20 ppm SO_3. The Ohio Lumex and SKC iodated sorbents, and Sorbent Technologies brominated sorbent were evaluated in this test phase.

The experimental apparatus and spike placement for Phase IV tests were the same as described above for Phases I through III. In the baseline and SO_3 gas exposure tests, traps were exposed to approximately 400 mL/ min of simulated flue gas at 300°F. Catch solutions were placed downstream of each trap to capture any mercury present in the sorbent trap effluent.

Table 2-9 summarizes the test matrix. An additional parameter evaluated in this study was exposure time. Separate sets of sorbent traps were exposed to simulated flue gas for four and seven days. Each sorbent was tested in duplicate in each gas exposure test conducted. The experimental design is described for each test type in the sections below.

Table 2-9
Summary of Test Matrix for Impact of SO_3 Tests

Sorbent Vendor	Sorbent Impregnate	Baseline Test		Adsorption Test (20 ppm SO_3)		Desorption Test (20 ppm SO_3)	
		4 day	7 day	4 day	7 day	4 day	7 day
Sorbent Technologies	brominated	X	X	X	X	X	X
Ohio Lumex	iodated	X	X	X	X	X	X
SKC Inc.	iodated	X	X	X	X	X	X

Baseline Test

Baseline test conditions were selected that would not affect sorbent performance, so that the impact of SO_3 could be determined. The SO_2 and mercury levels were selected based on data obtained in previous phases of this program. Phase III results showed that SO_2 concentrations higher than 500 ppm could cause desorption of mercury from the third spiked bed of an Appendix K trap, but successful results could be achieved at 200 ppm SO_2. In addition, analytical problems were encountered earlier when measuring high mercury mass loadings on tested sorbent beds (40,000 ng Hg); thus, lower mercury mass loadings were selected (5,000 ng Hg). Table 2-10 lists the components and concentrations of the simulated flue gas used in the baseline test.

A schematic of the system used to generate the baseline (i.e., no SO_3 present) simulated flue gas is shown above in Figure 2-3. Gas settings and pressures were monitored daily to ensure the make-up of the simulated flue gas remained the same and within specifications to produce the expected concentrations. Colorimetric gas detection tubes (Dräger tubes) were used to spot check the SO_2 concentrations during testing. Measurements were within ±15% of the targeted SO_2 concentration.

Table 2-10
Summary of Baseline Test Simulated Flue Gas Components

Flue gas Component	**Concentration**
SO_2 (ppm)	200
NO_x (ppm)	200
HCl (ppm)	50
CO_2 (%)	12
O_2 (%)	6
H_2O (%)	6
Hg (µg/Nm^3)	1.1-1.4
SO_3 (ppm)	0

The baseline test was conducted using the multi-trap, gas exposure test apparatus shown previously in Figure 2-2. Simulated flue gas was generated and plumbed into a heated gas manifold composed of glass columns, Teflon® fittings, tubing, and valves. The heated gas manifold was plumbed to deliver gas to 12 Appendix K traps that were placed in a temperature controlled oven. Approximately 400 mL/min of simulated flue gas was pushed through each sorbent trap. The gas flow across the traps was controlled using a Teflon® valve placed upstream of each sorbent trap. The measured flow rates across the sorbent traps for the baseline tests ranged from 350 – 430 mL/min. The effluent gas exited the oven and bubbled into a glass impinger containing 10% v/v hydrogen peroxide/ 5% v/v nitric acid solution, then into a glass impinger containing 4% w/v potassium permanganate/ 10% v/v sulfuric acid solution. The catch solutions were configured analogous to a Method 29 train, and were used to capture any mercury that passed through or desorbed from the sorbent traps.

The temperature of the oven and one trap effluent gas stream were monitored several times daily. The trap effluent gas temperature was measured by a thermocouple installed in a Teflon® tee placed immediately downstream of one trap. The temperature of the trap effluent gas ranged from 294 – 300°F. The average temperature of the trap effluent gas was 298°F.

Mercury concentrations were verified in the simulated flue gas each day with 1 – 2 hours of measurements using an EPRI semi-continuous mercury monitor. The mercury concentration range listed in Table 2-10 represents the highest and lowest concentration measured over the seven-day testing period. Mercury concentrations were stable over the seven-day testing period.

Only one baseline test was conducted, thus adsorption and desorption were not tested separately at baseline conditions. All traps contained three one-gram beds of sorbent, separated by glass wool. The third bed of each trap was spiked in the front-half configuration (see Figure 2-4) with 5,000 ng of mercury. These traps were prepared by URS Corporation using the stannous chloride ($SnCl_2$) dynamic spiking method, as described in an earlier report [2].

Sorbent traps were exposed to the simulated baseline gas for four and seven day periods. Each sorbent type was tested in duplicate for both gas exposure times. Table 2-11 summarizes the sorbent trap configurations tested at each exposure duration.

Table 2-11
Summary of Baseline Test Sorbent Trap Configurations

Sorbent Vendor	Sorbent Impregnate	Spike Placement	Hg Spike (ng)	# of replicates tested	
				4 day	7 day
Sorbent Technologies	brominated	FH	5,000	2	2
Ohio Lumex	iodated	FH	5,000	2	2
SKC Inc.	iodated	FH	5,000	2	2

FH – front half

At the conclusion of the baseline test, the duplicate sorbent tube pairs were split into two sets. One set of sorbent traps were prepared for analysis using EPRI's modified version of the ASTM 6414 wet digestion method, and were analyzed on a flow injection mercury system equipped with cold vapor atomic fluorescence detection. The second set was analyzed using an Ohio Lumex RP-M324 Thermal Decomposition Furnace attached to a RA-915 Mercury Analyzer. All Method 29 catch solutions were prepared for analysis using ASTM 6784 "Ontario Hydro Method" and analyzed on a flow injection mercury system equipped with cold vapor atomic fluorescence detection.

SO_3 Gas Exposure Tests

Two types of SO_3 gas exposure tests were conducted in this study. Adsorption SO_3 gas exposure tests were designed to evaluate the ability of sorbents to capture and retain mercury on the first sorbent bed of a sorbent trap in the presence of SO_3. These tests were conducted by exposing sorbent traps containing two beds of clean sorbent to a mercury- and SO_3-laden simulated flue gas. Sorbent traps were exposed to the simulated baseline gas for four or seven day periods. Each sorbent type was tested in duplicate for both gas exposure durations. Table 2-12 lists the composition of the simulated flue gas for the adsorption SO_3 gas exposure tests.

Table 2-12
Summary of Adsorption SO_3 Test Simulated Flue Gas Components

Flue gas Component	Concentration
SO_2 (ppm)	200
NO_x (ppm)	200
HCl (ppm)	50
CO_2 (%)	12
O_2 (%)	6
H_2O (%)	6
Hg (μg/Nm^3)	0.8 - 5.0
SO_3 (ppm)	18.26

Desorption SO_3 gas exposure tests were designed to evaluate the propensity of the mercury to desorb from the pre-spiked third bed of an Appendix K trap in the presence of SO_3. These tests were conducted by exposure of pre-spiked Appendix K traps to a mercury-free, SO_3-laden simulated flue gas. Table 2-13 the composition of the simulated flue gas for the desorption SO_3 gas exposure tests.

Table 2-13
Summary of Desorption SO_3 Test Simulated Flue Gas Components

Flue gas Component	Concentration
SO_2 (ppm)	200
NO_x (ppm)	200
HCl (ppm)	50
CO_2 (%)	12
O_2 (%)	6
H_2O (%)	6
Hg (µg/Nm^3)	0
SO_3 (ppm)	21.26

The third bed of each trap tested in the desorption SO_3 gas exposure tests was spiked in the front-half configuration (see Figure 2-4) with 5,000 ng of mercury. These traps were prepared by URS Corporation via the stannous chloride ($SnCl_2$) dynamic spiking method, as described in an earlier report [2]. Table 2-14 summarizes the configuration of traps tested in the desorption SO_3 gas exposure tests. Sorbent traps were exposed to the simulated baseline gas for four or seven day periods in both the adsorption and desorption SO_3 gas exposure tests. Each sorbent type was tested in duplicate for both exposure durations.

Table 2-14
Summary of Desorption SO_3 Test Trap Configurations

Sorbent Vendor	Sorbent Impregnate	Spike Placement	Hg Spike (ng)	# of replicates tested	
				4 day	7 day
Sorbent Technologies	brominated	FH	5,000	2	2
Ohio Lumex	iodated	FH	5,000	2	2
SKC Inc.	iodated	FH	5,000	2	2

FH – front half

SO_3-laden simulated flue gas was made by mixing SO_3 with preheated baseline simulated flue gas. The baseline gas was preheated to 400-500ºF in a heated glass mixing tube. SO_3 was generated by passing a known mass of SO_2 over a bed of platinum (Pt) black powder dispersed on quartz wool heated to1000ºF.

In order to transport SO_3 to the sorbent tubes without sulfuric acid condensing in the transport tubes, the flue gas had to be maintained at a temperature above the acid dew point of sulfuric acid. That temperature depends on factors such as gas pressure, gas composition and SO_3 concentration. At the conditions tested, the dewpoint is between 282-284ºF. In the SO_3 gas exposure tests, transport of the simulated flue gas took place at temperatures ranging from 310ºF

- 750ºF, thus a few modifications were made to the multi- trap gas exposure test experimental apparatus shown in Figure 2-2:

- Most Teflon® connections were replaced with quartz.
- Several borosilicate glass pieces were replaced with quartz pieces.
- Most of the gas transport/mixing pieces and trap manifold were placed inside an oven to promote even heating.

Figure 2-7 shows a diagram of the modified test apparatus used to conduct the SO_3 multi-trap gas exposure tests.

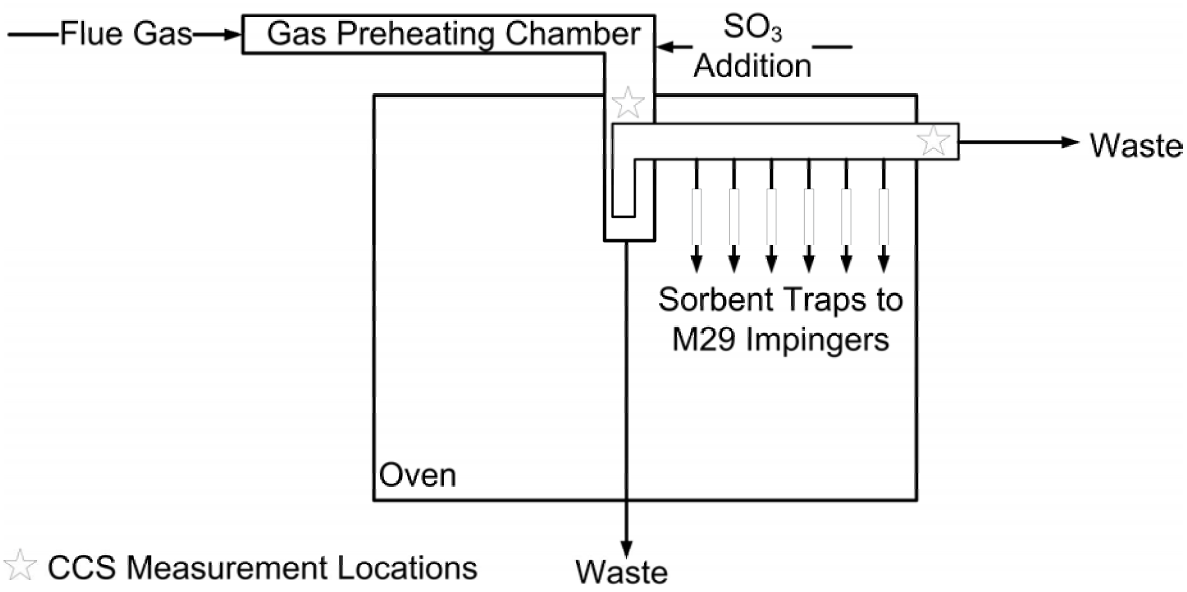

Figure 2-7
Schematic of SO_3 Gas Exposure Test Apparatus

Simulated SO_3-laden flue gas was mixed and transported to a heated trap manifold plumbed to deliver gas to six Appendix K traps. The gas temperature in the heated trap manifold was monitored daily at the inlet and outlet, and temperatures were maintained above 305ºF. A slipstream of approximately 400 mL/min of simulated flue gas was pulled through each sorbent trap. The effluent gas exited the oven and bubbled into a glass impinger containing 10% v/v hydrogen peroxide/ 5% v/v nitric acid solution, then into a glass impinger containing 4% w/v potassium permanganate/ 10% v/v sulfuric acid solution. The catch solutions were configured analogous to a Method 29 train, and were meant to capture any mercury that passed through or desorbed from the sorbent traps. The gas flow across the traps was controlled using a Teflon® valve placed downstream of the catch solutions. Flowrates and gas volumes were measured with mass flow meters and totalizers placed downstream of the catch solution impingers.

The temperature of one trap effluent gas stream was monitored daily. The trap effluent gas temperature was measured by a thermocouple installed in a Teflon® tee placed immediately downstream of one trap. The temperature of the trap effluent gas ranged from 280 – 307°F over the four tests. The average temperature of the trap effluent gas was 293°F.

In the adsorption tests, mercury concentrations were verified in the simulated flue gas each day with 1 – 2 hours of measurements, using an EPRI semi-continuous mercury monitor. The mercury concentration range listed in Table 2-12 represents the lowest and highest concentration measured over the four and seven-day testing periods. The mercury concentrations fluctuated over this range during both test periods.

SO_3 was measured at the exit of the trap manifold at the beginning and end of each SO_3 gas exposure test using a Controlled Condensation System (CCS) method. SO_3 concentrations listed in Tables 2-12 and 2-13 represent an average of the two measurement events. SO_3 transport across the heated trap manifold was verified by measuring the SO_3 concentration at the trap manifold inlet and outlet. The CCS measurement locations are shown in Figure 2-7. All trap manifold inlet and outlet measurements agreed within 7% of each other.

At the conclusion of each test, sorbent tubes were prepared for analysis using EPRI's modified version of the ASTM 6414 wet digestion method. All sorbent traps and catch solutions were analyzed on a flow injection mercury system equipped with cold vapor atomic fluorescence detection.

3
RESULTS

This section provides a comprehensive discussion of the results from this five-year study. Phases I through III have been reported previously [1, 2, 4]; thus, this report provides only a high-level discussion of conclusions from the results of these studies. Results from Phase IV, which have not been reported previously, are discussed in more detail.

Findings from Previous Phases of the Study

Phase I – Initial Screening Tests

The objective of the Phase I study was to identify several potential Appendix K sorbents for field testing. The selected sorbents were first subjected to a analytical pre-screening tests to quantify background mercury contamination levels and the ability of the sorbents to adsorb mercury. Some of the sorbents were dropped from the study because of high background mercury contamination. The contamination appeared to be sorbent-specific, not vendor specific. Background contamination did not appear to be related specifically to the base material of the granulated carbons.

Results from the adsorption tests indicated that several of the granulated carbon sorbents were capable of adsorbing large amounts of mercury during long term exposure to mercury-laden gas. In an air stream, all sorbents, except one (SKC Anasorb coconut shell carbon), were capable of adsorbing at least one million ng of Hg per gram of sorbent. One non-impregnated coconut shell carbon (SKC Anasorb) adsorbed only one-tenth the amount of mercury as the other non-impregnated coconut shell carbon (Carbochem CG3070). The SKC sorbent was the only one that had significant (>5%) breakthrough to the second carbon bed. These results indicated that a vendor's manufacturing process is as important as the base component of the activated carbon.

The adsorption capacities of the sorbents decreased when tested in simulated unscrubbed flue gas. The performance of the sorbents was characterized according to the amount of mercury adsorbed on the first bed and also by the amount of mercury that broke through to the second bed. When exposed to a simulated unscrubbed flue gas (high SO_2, low moisture content) for 10 days, only one (Frontier GeoSciences, Inc.) of the five tested sorbents had less than 5% breakthrough of mercury to the second bed. However, the pre-packed Frontier Geosciences tube contained twice the amount of carbon as the other tubes.

With simulated scrubbed flue gas (low SO_2, high moisture content), five of the eight sorbents tested passed the Appendix K criterion of •5% mercury breakthrough to the second bed. This was an improvement in performance over the unscrubbed gas condition. Of the five sorbents that passed, three were iodated sorbents (Frontier GeoSciences, Inc., Ohio Lumex, and SKC), and the other two were unimpregnated, coal-derived sorbents. The Ohio Lumex and SKC iodated sorbents were not tested at the unscrubbed stack gas condition, so it is unknown if they would have passed the •5% breakthrough criterion as did the Frontier GeoSciences sorbent.

Both unimpregnated coconut shell carbon sorbents performed poorly in both field simulation adsorption tests. Impregnating iodine onto the coconut shell carbon improved performance in all tests where a comparison was available. Bromination of a Norit Darco coal-based sorbent did not increase the sorbent's ability to capture and hold mercury; rather, the impregnation of the bromine decreased the sorbent's ability to hold the mercury.

Results from the mercury mobilization (desorption) gas exposure tests showed that back-half spikes lost greater than 80% of the spiked mercury after ten days of exposure to a mercury-free simulated flue gas. According to Appendix K criteria, these tubes would have been considered invalid. A few sorbents were tested using the front-half spike configuration and the results showed this configuration allowed for more mercury to be retained on the spiked bed.

Phase II - Spike Optimization

The objective of the Phase II effort was to understand factors that effect mercury spike retention and optimize the spiking procedures. Simulated flue gas was used to evaluate loss of mercury spikes under conditions simulating sampling of dry stack, eastern bituminous coal flue gases. Both iodated (Ohio Lumex) and brominated (Sorbent Technologies) sorbent traps spiked on the back half of the third sorbent bed began to lose mercury within one to two days of flue gas exposure. Once mercury desorption began, 25% or more of the pre-spiked mercury desorbed within two to three days. Tests were not continued long enough to determine the time required for complete loss of mercury from the tubes as this was not a primary objective of the tests.

Increasing the sorbent temperature during dynamic mercury spiking from ambient to 250°F did not increase mercury retention on either the brominated or the iodated sorbent beds. Desorption profiles obtained from traps spiked at 250°F were similar to those spiked at room temperature. These results indicate that the 250°F spiking temperature was not effective in increasing the extent of irreversibly-bound mercury at the sorbent surface.

By changing the location of the spike from the back portion to the front portion of the sorbent bed mercury retention was improved for both the brominated and iodated sorbent. Appendix K requires a mercury spike on a third section of a sorbent trap to contain within 75 to 125% of its initial value after field testing. Post-test spike retention for all brominated traps spiked using the front half configuration ranged from 88 – 101%, meeting these criteria. Conversely, for the iodated traps spiked using the same configuration mercury retention ranged from 32 – 82% and only one of the six iodated traps tested passed the Appendix K criteria. These results suggest that the rate of mercury desorption and subsequent transport across the iodine-treated carbon sorbent is much faster than that for the bromine-treated carbon sorbent. They also suggest more chemisorption occurring with the latter.

An observation from these tests suggested a possible impact of flue gas components on sorbent trap performance. No mercury was lost from a front half spike of a brominated sorbent bed when two additional beds were placed upstream during flue gas exposure (i.e., normal Appendix K configuration), while 99% of the mercury was lost from an identical bed when the two upstream sorbent beds were not present. These results suggest that the upstream beds were protecting the spiked mercury from flue gas species that promote mercury desorption. Because the results suggested an impact of flue gas species, they raised concerns that sorbent trap performance might be impacted by flue gases containing high levels of the interfering species. Corroborating this observation, in Phase I of this study, mercury retention on an Appendix K trap improved for a gas stream with lower SO_2 levels (200 ppm vs. 1600 ppm SO_2).

Phase III – Impact of SO_2

Phase III tests evaluated the effect of SO_2 on the retention of mercury on the pre-spiked third bed of an Appendix K trap. Traps were exposed to simulated flue gas containing SO_2 levels ranging from 200-1000 ppm, for seven days at 300°F. Sorbents from four vendors (Ohio Lumex, SKC, Frontier Geosciences, and Sorbent Technologies) were evaluated, including both iodated and brominated sorbents and front-half and batch-spiked configurations. For each test, the amount of mercury that desorbed from each pre-spiked sorbent bed was determined by measuring mercury in the catch solutions.

For traps spiked in the front-half configuration, none of the four sorbents tested showed any loss of mercury at SO_2 concentrations at or below 500 ppm. Only two sorbents (Ohio Lumex and Sorbent Technologies) were tested at 1000 ppm SO_2. Losses were observed with the Ohio Lumex iodated sorbent, while no measurable loss was observed with the Sorbent Technologies brominated sorbent. Thus, the brominated sorbent may be a better choice for long term (e.g., seven days) mercury measurement in gases containing levels of SO_2 greater than 500 ppm. As only one iodated sorbent (Ohio Lumex) was tested at 1000 ppm SO_2, it is not known if all iodated sorbents will lose mercury at elevated SO_2 concentrations.

A comparison of mercury loss from front-half spiked and batch spiked beds of the same sorbent (SKC) showed that the batch-spiked beds lost mercury when exposed to 350 and 500 ppm SO_2, while the front-half spiked beds prepared with the SKC sorbent showed no measurable loss at these SO_2 levels.

The Phase III results demonstrated that SO_2 can have a negative impact on the retention of mercury on the pre-spiked beds. For the traps that experienced Hg loss, the amount of desorption increased with increasing SO_2 concentration in the flue gas.

Phase IV – Impact of SO_3

The impact of SO_3 on sorbent performance (adsorption and desorption of mercury) was evaluated in two separate gas exposure tests. Adsorption tests were designed to evaluate the impact of SO_3 on the ability of the sorbents to capture and retain mercury on bed 1 of a sorbent trap. Desorption tests were designed to evaluate the mobility of the mercury from the pre-spiked, third sorbent bed in an Appendix K trap. A baseline test was conducted first to ensure the selected baseline test conditions (i.e., no SO_3) did not negatively impact both adsorption and desorption of mercury.

Baseline Test Results

In the baseline test, pre-spiked Appendix K traps were exposed to baseline simulated flue gas (i.e., no SO_3) for four and seven days. The mercury measurement results were used to calculate the following data:

- The percent breakthrough of mercury from sorbent bed 1 to sorbent bed 2
- The percent recovery of pre-spiked mercury on sorbent bed 3
- Mercury mass balance for each trap/catch solution unit

The percent breakthrough of mercury from the first sorbent bed to the second sorbent bed is calculated as:

% Breakthrough = (Hg on bed 2/Hg on bed 1) * 100

A mercury mass balance was determined as follows:

- Hg mass on all sorbent beds and impinger /theoretical Hg mass

The theoretical mass of mercury exposure was calculated by multiplying the measured daily gas mercury concentration by the daily flow measurements across each trap. The sum of these daily tabulated mercury masses equals the theoretical mass of mercury to which each trap was exposed.

The purpose of the baseline test was to verify that the selected test conditions would not affect the performance of the sorbents selected for evaluation in the SO_3 gas exposure tests. The main difference between the baseline test conditions and the SO_3 gas exposure test conditions is the addition of SO_3 to the gas. Selected baseline conditions would be considered acceptable for use in the SO_3 gas exposure tests if the following conditions were met:

- Percent breakthrough values met the Appendix K criteria of •5%.
- Percent recovery of the pre-spiked mercury met the Appendix K criteria of ±25% of the true value
- No detectable mercury in the catch solutions
- The percent of mercury recovered across each trap/catch solution unit was within the range of 80 – 120%

Table 3-1 presents the results of the baseline gas exposure test. Each sorbent type was tested in duplicate at each gas exposure duration. Duplicate pairs were split and analyzed using two different mercury analytical methodologies: a wet digestion/AFS and a thermal desorption/AAS technique. The percent breakthrough, percent recovery of the pre-spiked mercury, catch solution results and the percent of mercury recovered across each trap/catch solution unit (% total Hg recovered) are presented. An entry of "BD" indicates the mercury measurement in the combined catch solution impinger was below the detection limit, meaning less than 3% of the total mercury passed through the sorbent trap or desorbed from the pre-spiked bed.

Table 3-1
Summary of Baseline Test Results

Sorbent ID	Trap ID	Exposure Time Days	Analytical Method	Sorbent Halogen[1]	% BT[2]	% Recovery of pre-spiked Hg	Catch Solution (ng Hg)	% Total Hg Re-covered
Ohio Lumex	1	4	wet	I	0.6%	102%	BD[3]	94%
Ohio Lumex	2	4	thermal	I	0.3%	103%	BD	94%
Ohio Lumex	3	7	wet	I	0.2%	99%	BD	96%
Ohio Lumex	4	7	thermal	I	0.2%	104%	BD	108%
SKC	1	4	wet	I	0.3%	98%	BD	93%
SKC	2	4	thermal	I	0.5%	100%	BD	88%
SKC	3	7	wet	I	0.1%	93%	BD	97%
SKC	4	7	thermal	I	0.2%	99%	BD	109%
Sorbent Technologies	1	4	wet	B	0.2%	104%	BD	103%
Sorbent Technologies	2	4	thermal	B	0.6%	101%	BD	97%
Sorbent Technologies	3	7	wet	B	0.5%	110%	BD	104%
Sorbent Technologies	4	7	thermal	B	0.2%	104%	BD	98%

[1] I = iodated, B = brominated
[2] Percent breakthrough
[3] BD = below analytical detection limits

The following observations were made from the baseline tests:

- The percent breakthrough values were all less than 1% and met the Appendix K criteria
- The percent recoveries of the pre-spiked mercury ranged from 93 – 110%. All traps met the Appendix K criteria of +/-25%
- There was no detectable mercury in the catch solutions.
- The total mercury recoveries ranged from 88 – 109%

The baseline test results show all three sorbents performed very well at the baseline conditions. All traps would have met Appendix K criteria and been considered valid, reportable data. It was concluded that the selected baseline conditions would be appropriate for use in the SO_3 gas exposure tests.

Another notable observation was the good agreement between the two mercury analytical methods. Comparison of the percent recovery results of the pre-spiked mercury for duplicate pairs of each sorbent type (i.e., traps 1 & 2, and traps 3 & 4) show that all pairs matched within 6% , which is within the analytical error of both methodologies.

Adsorption SO_3 Test Results

In the SO_3 adsorption tests, two-bed traps were exposed to simulated baseline flue gas containing approximately 20 ppm SO_3 for four or seven days. The mercury measurements were used to calculate the percent breakthrough of mercury and mercury mass balance for each trap/catch solution unit.

A negative impact of SO_3 on the ability of the sorbent to adsorb mercury from the flue gas was judged to have occurred if either of two conditions were met:

- The percent breakthrough of mercury from bed 1 to bed 2 was greater than 5%, or
- Detectable amounts of mercury were measured in the catch solutions

Table 3-2 presents the results of the adsorption SO_3 gas exposure tests. Each sorbent type was tested in duplicate at each gas exposure time. All traps were analyzed using the wet digestion technique. The calculated percent breakthrough, the catch solution results and the percent of mercury recovered across each trap/catch solution unit (% total Hg recovered) are presented. An entry of "BD" indicates the mercury measurement in the combined catch solution impingers was below the detection limit. This combined detection limit represented 5 – 10% of the mercury that theoretically entered the sorbent trap via the simulated flue gas.

The following observations were made regarding the results from the SO_3 adsorption gas exposure tests:

- The percent breakthrough values for all traps were less than 2% and met the Appendix K criteria of •5%.
- There was no detectable mercury in the catch solutions.
- Exposure time did not affect sorbent performance. Results were equivalent for all three sorbents at both 4-day and 7-day exposure times.
- The total percent of mercury recovered was low and ranged from 42 – 100%.

Almost all mass balance results fell outside of the desired 80 – 120% range. These suspect data were investigated further. Several factors could potentially cause low mercury recoveries:

- The measured masses on beds 1 and 2 could be biased low.
- The catch solutions may not have effectively captured mercury that broke through the trap.
- The calculated theoretical mercury masses may be biased high. This theoretical mercury mass is used to calculate the total percent mercury recoveries.

Table 3-2
Summary of Adsorption SO_3 Test Results

Sorbent ID	Trap ID	Exposure Time Days	Analytical Method	Sorbent Halogen[1]	%BT[2]	Catch Solution (ng Hg)	% Total Hg Recovered
Ohio Lumex	1	4	wet	I	0.3%	BD[3]	65%
Ohio Lumex	2	4	wet	I	0.2%	BD	63%
Ohio Lumex	3	7	wet	I	0.2%	BD	70%
Ohio Lumex	4	7	wet	I	1.6%	BD	70%
SKC	1	4	wet	I	0.2%	BD	62%
SKC	2	4	wet	I	0.2%	BD	63%
SKC	3	7	wet	I	0.1%	BD	66%
SKC	4	7	wet	I	0.3%	BD	100%
Sorbent Technologies	1	4	wet	B	0.3%	BD	80%
Sorbent Technologies	2	4	wet	B	0.6%	BD	42%
Sorbent Technologies	3	7	wet	B	0.2%	BD	63%
Sorbent Technologies	4	7	wet	B	NM	BD	NM

[1] I = iodated, B = brominated
[2] Percent breakthrough
[3] BD = below analytical detection limits

The analytical methodologies were investigated by double-checking the quality assurance/quality control (QA/QC) results and details of preparatory steps. One of the most important QA/QC checks used to ensure the accuracy during the analysis of sorbent trap beds is the analysis of a spiked trap called the "lot spike". A lot spike is retained from a batch of spiked traps, set aside (i.e., not exposed to flue gas), and analyzed for mercury with the tested traps. The mercury recovery results of all lot spikes fell within the accepted range of 85 -115%, thus ruling out problems with the analytical processes. Furthermore, all other analytical QA/QC results fell within accepted ranges.

The catch solution results were considered reliable based on the acceptable analytical QA/QC results, and on the results of a matrix spike inserted in the test apparatus (i.e., spiked impinger solutions were exposed to flue gas). The matrix spike results show mercury was captured effectively from the simulated flue gas.

Based on these findings, the low mercury mass balance results were likely caused by a high bias in the calculated theoretical mercury masses. The measured mercury gas concentrations are used to calculate the theoretical masses. Measurements of the gaseous mercury in the simulated flue gas were only taken for 1 -2 hours each day of the test. The average gas concentration obtained during this short measurement time is used to calculate a theoretical mass over a 4- or 7-day test period. If the mercury concentration is not stable, a bias will be present in the calculated theoretical mercury. The mercury concentrations that were measured changed daily, and ranged from 0.8 – 5.0 $\mu g/m^3$ throughout the tests due to problems with the mercury generator.

Another notable observation is that most of the total mercury mass balance recoveries fell in the range of 62 – 70%, and the mass of mercury collected on bed 1 was roughly the same for traps tested in parallel. These data support the likelihood that the low recoveries were a due to errors in quantifying the low concentration of mercury delivered to the traps.

In spite of the low mass balance recovery results, very little mercury broke through sorbent bed 1 in the presence of SO_3. All traps would have met the Appendix K criteria for adsorption (i.e. less than 5% breakthrough to bed 2). The breakthrough data and catch solution results show that 20 ppm SO_3 did not affect the ability of any of the three sorbents to absorb and retain mercury from the simulated flue gas after seven days of gas exposure.

Desorption SO_3 Test Results

In the SO_3 desorption test, pre-spiked, three-bed Appendix K traps were exposed to a mercury-free, simulated baseline flue gas containing approximately 20 ppm SO_3. The mercury measurement results of the traps and the catch solutions were used to determine if desorption had occurred. The mercury measurement results were used to calculate the following data:

- The percent recovery of pre-spiked mercury on sorbent bed 3
- Mercury mass balance for each trap/catch solution unit

A mercury mass balance was calculated for each trap/catch solution unit by dividing the sum of the mercury measured in the sorbent and catch solution by the theoretical mass of the pre-spiked mercury. Desorption was determined to have occurred when both of the following conditions were met:

- Detectable amounts of mercury were measured in the catch solutions.
- The percent of Hg recovered across the trap/catch solution unit was within the range of 80 – 120%

Table 3-3 presents the results of the SO_3 desorption gas exposure tests. Each sorbent type was tested in duplicate at each gas exposure time. All traps were analyzed using the wet digestion technique. The percent of pre-spiked mercury recovered, the catch solution results and the percent of mercury recovered across each trap/catch solution unit (% total Hg recovered) are summarized. An entry of “BD” indicates the mercury measurement in the combined Method 29 impingers was below the detection limit. This combined detection limit represented 4 – 11% of the pre-spiked mercury.

Table 3-3

Summary of Desorption SO_3 Test Results

Sorbent ID	Trap ID	Exposure Time Days	Analytical Method	Sorbent Halogen[1]	% Recovery of bed 3 Hg	Catch Solution (ng Hg)	%Total Hg Recovered
Ohio Lumex	1	4	wet	I	113%	BD[2]	113%
Ohio Lumex	2	4	wet	I	111%	BD	111%
Ohio Lumex	3	7	wet	I	113%	BD	113%
Ohio Lumex	4	7	wet	I	110%	BD	110%
SKC	1	4	wet	I	113%	BD	113%
SKC	2	4	wet	I	112%	BD	112%
SKC	3	7	wet	I	111%	BD	111%
SKC	4	7	wet	I	125%	BD	125%
Sorbent Technologies	1	4	wet	B	120%	BD	120%
Sorbent Technologies	2	4	wet	B	120%	*	120%
Sorbent Technologies	3	7	wet	B	116%	BD	116%
Sorbent Technologies	4	7	wet	B	118%	BD	118%

[1] I = iodated, B = brominated
[2] BD = below analytical detection limits
*sample was contaminated during the testing process

The following observations were made regarding the SO_3 desorption tests:

- There was no detectable mercury in the catch solutions.
- Exposure time did not affect sorbent performance. Results were equivalent for all three sorbents at both the 4-day and 7-day exposure times.
- All but one total mercury recovery result fell in the desired 80 – 120% range

These results show all three sorbents performed well under the test conditions. All traps would have met Appendix K criteria (±25% recovery of pre-spiked Hg) and been considered valid, reportable data. The pre-spiked mercury recovery results and the catch solution results show that 20 ppm SO_3 did not affect the mobility of mercury from the required third bed spike after seven days of gas exposure.

4

CONCLUSIONS

Invalidation of an Appendix K sample is a concern for power plant operators, as the EPA's regulations require that missing or invalid sampling data must be substituted with conservative (high) assumed emission values. The objective of this study was to identify and evaluate sorbent materials for use in Appendix K. The approach was to identify candidate sorbents and perform a laboratory evaluation of the sorbents' ability to adsorb and retain mercury.

In Phase I of this study, a large number of granulated sorbent materials were identified as potential candidates for evaluation. Approximately one dozen sorbent materials were selected for an analytical pre-screening test to quantify background mercury contamination levels and the ability of the sorbents to adsorb mercury. All selected sorbent materials were derived from coal and coconut shell, and some of the sorbents contained impregnates such as sulfur, bromine, or iodine.

Sorbents that passed the analytical pre-screening tests were configured in gas exposure tests designed to evaluate sorbent performance in simulated flue gas at flow rates and temperatures that would be encountered during a typical Appendix K run. A sorbent ideal for use in the Appendix K method would be able to effectively capture and retain mercury from flue gas after a week of gas exposure. The Appendix K method outlines acceptance criteria that are used to validate sorbent performance, and failure to meet these criteria results in data invalidation.

Two types of gas exposure tests were conducted to evaluate sorbent performance, adsorption tests and desorption tests. Adsorption tests were designed to evaluate the ability of sorbents to capture and retain mercury on the first sorbent bed in a sorbent trap. Desorption tests were designed to evaluate the mobility of the mercury from the pre-spiked third sorbent bed in an Appendix K trap. The conclusions of this five-year test program have been organized below by test type (i.e., adsorption tests, desorption tests).

Adsorption Tests

In Phase I, the ability of various sorbents to capture and retain mercury was evaluated in scrubbed (low SO_2, high H_2O content) and unscrubbed (high SO_2, low H_2O content) simulated flue gas exposure tests. The performance of the sorbents was characterized by the amount of mercury adsorbed on the first bed and also by the amount of mercury that broke through to the second bed. An important observation from this study was that several of the sorbents tested at both conditions showed improved performance (i.e., less breakthrough) in the simulated scrubbed stack gas.

The Frontier Geosciences iodated sorbent was the only commercially available sorbent tested in both gas conditions. It appeared to perform the best at the highest SO_2 concentration, however these results were misleading since the pre-made traps contained twice the amount of carbon as the other sorbents.

The main difference between the simulated scrubbed and unscrubbed stack gases was the concentration of SO_2 (200 ppm SO_2 vs. 1600 ppm SO_2, respectively). The scrubbed stack gas contained less SO_2; thus, these results suggested that SO_2 affected the ability of the tested sorbents to adsorb mercury from simulated flue gas. This conclusion was supported by results from an earlier EPRI test program that also indicated that SO_2 may reduce the adsorption and/or retention of mercury on carbon sorbents [4].

The possibility of SO_2 impeding the ability of sorbents to capture mercury from flue gas will become less of a concern if plants are required to lower stack SO_2 levels in the future. However, if breakthrough problems are encountered in the field, possible solutions could include the addition of more sorbent material to the first sorbent bed, or less gas exposure time.

Another important observation from the mercury adsorption tests was that several sorbents were capable of adsorbing large quantities of mercury from the simulated flue gas. Appendix K sorbents exposed to flue gas containing high levels of mercury (e.g., 20 $\mu g/m^3$) for a week duration will need to retain between 40,000 and 80,000 ng Hg. Several sorbents successfully captured mercury loadings in this range and were able to meet the Appendix K breakthrough criterion of •5%.

Although several non-impregnated sorbents performed satisfactorily in the simulated scrubbed stack gas; only commercially available, impregnated sorbents were tested for the remainder of the program (i.e., Phases II, III, and IV). These sorbents included the SKC, Ohio Lumex, and Frontier Geosciences iodated sorbents and Sorbent Technologies brominated sorbent.

Phase IV tests investigated the impact of SO_3 on the ability of sorbents to capture mercury from simulated flue gas containing. Traps were exposed to flue gas containing 20 ppm SO_3 for four and seven days. The SKC and Ohio Lumex iodated sorbents, and Sorbent Technologies brominated sorbent were evaluated. Results showed that 20 ppm SO_3 did not impact sorbent performance at either exposure duration. All traps tested would have met the Appendix K criteria and the results would have been considered reportable data. This result was surprising, since several research groups have documented issues with SO_3 impeding the mercury removal efficiency of powdered activated carbon (PAC) when injected into SO_3-laden flue gas.

One observation made at the conclusion of each SO_3 adsorption test was that liquid had collected on the glass walls of the trap upstream of the first sorbent bed, and the sorbent beds were wet. These tests were conducted at 300ºF, which is very close to the acid dew point temperature for 20 ppm SO_3 in a flue gas containing 5-6% moisture (284ºF). The observed moisture in the traps was likely SO_3 that had condensed from the flue gas as sulfuric acid. The wet sorbent beds suggest that some percentage of the SO_3 did contact the sorbent beds as sulfuric acid.

The acid dew point temperatures for SO_3 concentrations typically found in coal fired power plants can range from 230º – 300ºF. The Appendix K method requires the probe to be kept at a high enough temperature to prevent water condensation in the traps; thus, the probe temperature during sampling likely is at the low end of the acid dewpoint temperature range (i.e., 240º - 250ºF). If traps are packed such that the flue gas must travel a few inches through the glass tube before contacting the first sorbent bed, the flue gas could cool enough for SO_3 to condense as sulfuric acid before having the chance to impact the performance of the sorbent. Transport of SO_3 is very difficult to achieve at temperatures close to the acid dew point and is very sensitive to temperature fluctuations. It is possible the same phenomenon would occur in a field sampling situation. Condensation of the SO_3 could help mitigate any possible effects of gaseous SO_3 on sorbent performance in Appendix K traps.

The above observation may explain the discrepancy between the lack of SO_3 impact in this study and the reported impact with PAC injection. When PAC is injected in a duct for mercury control, the carbon is well dispersed and immediately contacts the gas containing the SO_3. The gas also does not have a chance to cool before contacting the carbon; thus, the speed of contact of the carbon with SO_3-laden gas could be the reason impacts on PAC performance have been observed. In a fixed bed application such as Appendix K, the front portion of the bed would contact any SO_3 present in the flue gas, but depending how the sorbent reacts with SO_3, the back portion of the bed may not see SO_3 until after a certain test duration.

It is important to note that only one SO_3 level was tested in this study. While the tested level (20 ppm) is considered high for coal-fired power plant flue gas, further evaluation would need to be conducted at additional SO_3 concentrations and at varying gas moisture percentages and temperatures to conclude definitively that no impact of SO_3 will occur. The results of this study show that there is likely no impact on sorbent performance when exposed to 300ºF stack gas containing 20 ppm SO_3 and 5-6% moisture.

Desorption Tests

The results of the Phase I mercury mobility tests showed that sorbents had difficulty retaining mercury when a mercury-free flue gas was passed over the pre-spiked sorbent bed. Traps spiked in a back-half configuration lost more than 80% of the pre-spiked mercury when exposed to simulated flue gas for 10 days. These results were problematic since Appendix K measurements are invalidated if the mercury mass recovered from the third, spiked bed post sampling falls outside ± 25% of the true (spiked) value. These results led to additional efforts to measure the desorption profile of mercury from pre-spiked beds and optimize spiking conditions to prevent loss of spiked mercury .

In Phase II, the rate of mercury loss from traps spiked using the back-half configuration was monitored continuously while traps were exposed to the same simulated flue gas used in the Phase I mercury mobility tests. Mercury loss began within one to two days of flue gas exposure. Once desorption began, 25% or more of the pre-spiked mercury desorbed within two to three days. Sorbent traps showed improved mercury retention when the spiked mercury was moved to the front of the sorbent bed.

In Phase II, post-test mercury recovery results for all brominated traps spiked using the front-half configuration ranged from 88 – 101% recovery, and results for the iodated traps spiked using the same configuration ranged from 32 – 82%. These data indicate improved retention of mercury when compared to the Phase I results. Furthermore, in Phase III the mercury loss from front-half spiked and batch spiked beds exposed to the same flue gas conditions was compared and showed that the batch spiked beds lost mercury, while the front-half spiked beds showed no measurable loss. Only one batch-spiked sorbent (SKC) was tested; thus, it is unknown whether this observation would apply to other sorbents spiked using a batch process.

It was determined that the placement of the spike on the sorbent bed has a major impact on the extent of mercury loss from the trap. Placing the mercury spike on the front face of the sorbent bed (the part first contacted by the flue gas) greatly improved mercury retention, but desorption still occurred from sorbent traps containing an iodated sorbent. Shortening the flue gas exposure time may mitigate these losses, and success with this strategy has been reported by some utilities.

The difference in retention between front and back half spikes may be due to multiple processes affecting the fate of mercury on the pre-spiked sorbent bed, including a combination of physisorption and chemisorption of mercury at the sorbent surface. Physisorption results from the electrical attraction of an adsorbate to a sorbent surface due to interactions such as van der Waals forces. Chemisorption involves the formation of strong bonds between the adsorbate and sorbent surface due to chemical reactions. Physisorption is typically reversible whereas chemisorption is not. The fact that mercury appears to migrate across the spiked bed when exposed to a mercury-free flue gas suggests physisorption is playing a role in the tube spiking process.

Chemisorption is usually thermodynamically favored over physisorption as temperature increases. Therefore, elevated sorbent temperature during dynamic spiking was investigated as a means to improve mercury retention. However, increasing the mercury spiking temperature from ambient temperature to 250ºF did not increase mercury retention on either the brominated or iodated sorbent beds. Desorption profiles obtained from traps spiked at 250ºF were similar to those spiked at room temperature.

Observations made during the Phase II laboratory studies suggested that mercury desorption could be associated with the concentration of flue gas constituents, particularly SO_2. In Phase III testing, the impact of SO_2 on desorption of mercury from the pre-spiked bed was investigated. Traps spiked in the front-half configuration were exposed to simulated flue gas containing SO_2 levels ranging from 200 – 1000 ppm, for seven days at 300ºF. The Ohio Lumex, Frontier Geosciences, and SKC iodated sorbents, and the Sorbent Technologies brominated sorbent were evaluated. Desorption test results showed that all four sorbents could retain mercury on a front-half spiked sorbent bed when exposed to a dry stack flue gas containing 500 ppm or less of SO_2 for seven days. These results are promising for future use of the sorbent tube method, as future EPA regulations could potentially require power plants to lower stack SO_2 emissions to well under 500 ppm SO_2.

Only two sorbents (Ohio Lumex and Sorbent Technologies) were tested at SO_2 levels greater than 500 ppm (1000 ppm SO_2). Losses were observed with the Ohio Lumex iodated sorbent, while no measurable loss was observed with the Sorbent Technologies brominated sorbent. Thus, the brominated sorbent may be a better choice for long-term (i.e., seven days) mercury measurement in gases containing levels of SO_2 greater than 500 ppm. As only one iodated sorbent was tested with 1000 ppm SO_2, it is not known if all iodated sorbents will lose mercury at elevated SO_2 concentrations.

In Phase IV, the impact of SO_3 on the desorption of mercury pre-spiked sorbent beds was investigated at one level of SO_3. Traps spiked using the front-half configuration were exposed to a simulated flue gas containing low SO_2 (i.e., 200 ppm) and 20 ppm SO_3 for four and seven days. The Ohio Lumex and SKC iodated sorbents, and Sorbent Technologies brominated sorbent were evaluated. The results showed that all three sorbents could retain mercury at either exposure time. All traps tested would have met the Appendix K criteria.

The same observation made at the conclusion of the SO_3 adsorption test was made at the conclusion of the SO_3 desorption test. Moisture had collected on the glass walls of the trap upstream of the first sorbent bed, and the sorbent beds were wet. These tests were conducted at the same temperature as the adsorption tests (300ºF) which was very close to the acid dew point temperature of 20 ppm SO_3 in flue gas containing 5-6% moisture (284ºF). Again, a portion of the SO_3 likely collected on the glass walls of the traps as sulfuric acid before contacting the sorbent beds. The wet sorbent beds show that some percentage of the SO_3 did contact the sorbent beds as sulfuric acid.

The same conclusions made from the SO_3 adsorption tests can be made from the results of the SO_3 desorption results. The SO_3 desorption tests were conducted at only one level of SO_3. The results of this study 20 ppm SO_3 in a dry stack gas containing 5-6% moisture does not lead to mercury desorption. Further testing would need to be conducted at additional SO_3 levels and at varying gas moisture percentages and temperatures in order to make definitive conclusions on the impact of SO_3 on sorbent performance.

Other Notable Observations

In Phase I of this study, a number of sorbents were evaluated in adsorption and desorption gas exposure tests. These sorbents were derived from various base components (i.e., coals and coconut shell) and contained various chemical impregnates such as sulfur, iodide, and bromide. The following notable observations were made during the EPRI research:

- Adding halogen to coconut shell-based sorbent improved performance over plain coconut shell sorbent
- Adding halogen to a sorbent does not always improve performance. For example, bromination of a lignite coal-based sorbent did not improve sorbent performance.
- Sorbents derived from the same base components but purchased from different vendors performed differently. These results indicated that a vendor's manufacturing process is as important as the base component of the activated carbon. The differences may be due in part to the amount or chemical species of impregnate added

The implications of these observations are that the ability of a sorbent to capture and retain mercury cannot be predicted from its base composition or chemical additive. Therefore, as vendors create and supply new sorbents, should be asked whether they have tested the new sorbent for mercury sorption and desorption efficiency.

5

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4. EPRI, 1998. *Laboratory Investigation and Model Development of Flue Gas Mercury Adsorption Using Solid Sorbents,* TR-110533.

A

COMPLETE LIST OF RECEIVED SORBENTS

Table A-1
Complete List of Received Sorbents

Vendor	Vendor ID	Base Component	Form	Mesh Size
Norit	Vapure 612	activated coal, used in gaseous applications	granular	6 X 12
Norit	Darco	steam activated Lignite coal, acid washed	granular	20 X 50
Norit	Darco LH	brominated lignite coal	granular	small
Norit	Exp. BWN4	proprietary	granular	large
Norit	ROX 0.8	acid washed extruded carbon	pellets	0.8mm
Norit	ROZ3	KI impregnated steam activated extruded carbon	pellets	3mm
Norit	RGM 0.8	steam activated extruded carbon	pellets	0.8mm
Norit	RGM 3	steam activated extruded carbon	pellets	3mm
Calgon	CAL	bituminous coal based activated carbon	granular	12 X 40
Calgon	DSR-A	reactivated activated (thermo process twice) carbon from water treatment process	granular	8 X 40
Calgon	OLC	coconut based activated carbon	granular	12 X 30
Calgon	SCCW	activated carbon (bituminous based carbon) impregnated with Fe and Zn	granular	14 X 40
Calgon	TOG	bituminous coal based carbon (similar to CAL - diff mesh size)	granular	20 X 50
Calgon	centaur	activated carbon with catalytic functionality (nitrogen oxides formed on surface)	granular	20 X 50
Calgon	HgR	sulfur impregnated activated carbon (bituminous based carbon)	granular	small
Calgon	HgRLH	iodated impregnated activated carbon (bituminous based carbon)	granular	small
Calgon	Sulfusorb 8	Cu impregnated activated bituminous coal carbon - (SCCW imp w/Cu)	granular	12 X 30
Mead Westvaco	Sample A 10-R-05	Wood derived Carbon	granular	12 X 40
Mead Westvaco	Sample A 719-R-04	Wood derived Carbon - same as above (10-R-05), different particle size	granular	20 X 50
Westvaco	Sample B 326-R-02	Wood derived Carbon - higher surface area, more organic Oxygen on surface	granular	20 X 50
Carbochem	SI 2050	impregnated activated carbon derived from bituminous coal	granular	20 X 50
Carbochem	CG 3070	coconut based activated carbon	granular	30 X 70
Supelco	Carbotrap	graphatized carbon black	granular	20 X 40
SKC	226-38-01	Petroleum based Charcoal	granular	20 X 40
SKC	226-38-03	HBr on Petroleum based Charcoal	granular	20 X40
SKC	-	KI + coconut shell charcoal	granular	20 X 40
SKC	2260101	coconut-shell charcoal	granular	20 X40
Ohio Lumex	-	KI + coconut shell charcoal	granular	small

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