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June 17, 2009

Dr. Michael Berndt
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Dear Dr. Berndt:

Subject: Draft Final Report Entitled "Demonstration of Mercury Capture in a Fixed Bed"
Work Order No. B25018; EERC Fund 14784

Enclosed please find the subject draft final report. If you have any questions or comments, please contact me by phone at (701) 777-5034, by fax at (701) 777-5181, or by e-mail at gdunham@undeerc.org.

Sincerely,

Grant E. Dunham
Research Manager

GED/sah

Enclosure

c/enc: Stan Miller, EERC
David Dunham, EERC

EERC
Energy & Environmental Research Center

DRAFT

DEMONSTRATION OF MERCURY CAPTURE IN A FIXED BED

Draft Final Report

(for the period of February 10, 2009, through June 30, 2009)

Prepared for:

Michael Berndt

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Division of Lands and Minerals
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Work Order No. B25018

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June 2009

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NOMENCLATURE

acfm	actual cubic feet per minute
alpm	actual liter per minute
ARL	Analytical Research Laboratory
ASTM	ASTM International
CMM	continuous mercury monitor
CVAA	cold-vapor atomic absorption
DAC	data acquisition and control
dscm	dry standard cubic meter
dslm	dry standard liter per minute
EERC	Energy & Environmental Research Center
EPA	U.S. Environmental Protection Agency
HEPA	high-efficiency particulate air
Hg^0	elemental mercury
Hg^{2+}	oxidized mercury forms
ICP–AES	inductively coupled plasma–atomic emission spectroscopy
lpm	liter per minute
MNDNR	Minnesota Department of Natural Resources
MPCA	Minnesota Pollution Control Agency
MSDS	material safety data sheet
NIST	National Institute of Standards and Technology
NaOH	sodium hydroxide
PSA	P S Analytical
psia	pounds per square inch absolute
sccm	standard cubic centimeter per minute
SIC	sulfur-impregnated carbon
$SnCl_2$	stannous chloride
W.C.	water column

DEMONSTRATION OF MERCURY CAPTURE IN A FIXED BED

1.0 PROJECT SUMMARY

The Minnesota Department of Natural Resources (MNDNR) contracted with the Energy & Environmental Research Center (EERC) to conduct a test program to evaluate the performance of activated carbons for mercury capture by fixed-bed mercury adsorption for flue gas generated by taconite-processing facilities.

The work included a 1000-hr test using two parallel columns with fixed beds consisting of multiple stages of selected granular activated carbons. The tests were conducted under constant process conditions for an actual duration of 1009 hours and were completed June 1, 2009.

The purpose of the test was to evaluate carbon performance for mercury abatement in a flue gas environment that simulates that generated in a taconite-processing facility. The test conditions were selected to match the nominal operating conditions downstream of a particulate scrubber.

The test configuration consisted of two columns (designated as Column 1 and Column 2) operated in parallel. Each column has an inside diameter of 3.75 inches and a total depth of 18 inches of carbon adsorbent. The columns were arranged in six bed segments ranging in depth from 1 to 6 inches. Sampling ports were located at the outlet of each bed segment to provide mercury breakthrough concentration data as a function of bed depth and time. The Column 1 carbon was a lignite-based granular brominated carbon (DARCO® Hg-LH 4×8) from Norit Americas. The Column 2 adsorbent was NUCON MERSORB HT-1.5 coal-based sulfur impregnated carbon (SIC).

The simulated flue gas composition and operating conditions were selected to represent those of a coal-fired kiln used to process taconite for steel production. The simulated flue gas fed to both columns provided a constant mercury concentration. The target mercury concentration was 6.0 µg/dscm. Mercury breakthrough measurements were taken at specific bed depths from both columns during the entire run with a continuous mercury monitor (CMM). The same CMM was used to periodically measure the inlet mercury concentration.

Following completion of the test, samples of carbon from each stage were recovered. All stages that were exposed to mercury were analyzed for total mercury content. Good agreement between the independent CMM breakthrough data and the laboratory data along with good mass balance closures provided a strong basis for forming the conclusions.

The results showed that for the conditions evaluated, an 18-inch bed of either brominated granular activated carbon or sulfur-impregnated activated carbon was adequate to provide essentially 100% control of the inlet mercury for more than 1000 hours.

2.0 BACKGROUND

It has been estimated that taconite processing in Minnesota releases approximately 350 to 400 kg of mercury to the atmosphere each year (1–4). Even though this amount is small compared to global emission rates, it represents Minnesota's second largest industrial source of mercury to the atmosphere. Therefore, the Minnesota Pollution Control Agency (MPCA) may regulate mercury emissions from taconite plants.

There are a number of methods to remove mercury from a flue gas stream. With taconite plants, it may be simple to inject additives such as halogens to enhance oxidation of the mercury in the induration furnace and promote capture of the oxidized mercury in the wet particulate scrubbers. Short-term tests (5) indicated 5%–13% capture for a straight-grate facility and 18%–32% for a grate–kiln facility when NaCl was added to the process. The addition of bromide salts was more effective, but still saw reductions of only 62%–64%. The best results (80% capture) were observed when a proprietary U.S. Environmental Protection Agency (EPA) oxidant was added to the scrubber solution. Each of these methods provides some level of mercury capture, but not at a high level (>95%). In addition, there is a possibility of increased corrosion in the system as a result of injecting these additives into the system, and an increase in particulate matter emissions due to additional fine particulate formation.

A fixed bed of activated carbon is preferred over other forms of mercury control for several reasons. Unlike injection of activated carbon upstream of the wet scrubber, a fixed bed will not add to the particulate loading to the scrubber or increase particulate matter emissions at the stack. With a fixed bed, nothing is added to the system that may affect the quality of the product or the life of the system, such as corrosion downstream. There will also be no increase of halogens in the scrubber water.

3.0 OBJECTIVES AND APPROACH

The overall goal of this project was to evaluate the ability of a fixed bed of sorbent material to capture mercury in a flue gas stream that simulates that generated during the processing of taconite for steel production.

The approach used to meet this objective was to utilize two adsorption columns, each configured with an 18-inch depth of carbon adsorbent. Column 1 was loaded with Norit America's DARCO Hg-LH 4×8 brominated lignite-based activated carbon. Column 2 was loaded with MERSORB HT-1.5 coal-based SIC adsorbent. The bed segment configurations in each column were identical. Both carbon adsorbents were selected based on past performance in capturing mercury.

The operating parameters for this test were selected to simulate real process conditions in terms of bed velocity, temperature, flue gas composition, and mercury concentration. The one component missing from the simulated flue gas is particulate matter. Mercury-sampling tests have shown that the dust in the taconite process flue gas is very reactive toward mercury, causing oxidation of elemental mercury (6). Mercury measurements taken at the outlet of the scrubber do not show this oxidation effect to the same degree, indicating the reactive dust has been removed by the scrubber. In support of this, chemical analysis of impactor samples collected at the stack of one taconite facility show the particulate matter downstream of the scrubber is mostly potassium and chloride, most likely as KCl salt (7). Based on these findings, it is likely there is very little reactive dust that would reach the fixed-carbon bed, and any that does would most likely enhance mercury capture rather than hinder it. For these reasons, it was decided to not include reactive dust as a variable in this test matrix. Using this system rather than pulling a slipstream of real flue gas from one of the taconite facilities also allows for uninterrupted exposure of the samples for an extended period of time in a controlled laboratory environment where all process variables can be controlled.

3.0 SCOPE OF WORK

3.1 Column Configurations

The configuration for the test consisted of two columns operated in parallel; each column had a total depth of 18 inches in six discrete bed segments.

Table 1 provides a segment-by-segment loading schedule for the two columns evaluated during the test. Column 1 consisted of a total of 18 inches of a lignite-based DARCO Hg-LH 4×8, which is brominated. Column 2 consisted of 18 inches of MERSORB HT-1.5 coal-based SIC adsorbent. The bed segment depth configurations were identical for the two columns.

3.2 Gas Composition and Conditions

The simulated flue gas composition used for the test is provided in Table 2. The levels of the gas components and the temperatures are based on actual measurements (8) and suggestions from John Engesser (Principal Engineer for Mineral Development, MNDNR Division of Land and Minerals).

Table 1. Column Bed Configurations by Bed Segment

Bed Segment No.	Bed Segment Sorbent Depth, in.	Cumulative Sorbent Depth at Bed Segment Outlet, in.	Column 1 Segment Loadings	Column 2 Segment Loadings
1	1	1	DARCO Hg-LH	MERSORB HT-1.5
2	2	3	DARCO Hg-LH	MERSORB HT-1.5
3	3	6	DARCO Hg-LH	MERSORB HT-1.5
4	3	9	DARCO Hg-LH	MERSORB HT-1.5
5	3	12	DARCO Hg-LH	MERSORB HT-1.5
6	6	18	DARCO Hg-LH	MERSORB HT-1.5

Table 2. Simulated Flue Gas Inlet Composition and Conditions

Conditions at Hg Adsorption Model Column Inlet	Units	Value
Temperature	°F	180
H_2O	mol% (wet)	16.0
CO_2	mol% (dry)	3
O_2	mol% (dry)	18
N_2	mol% (dry)	Balance
NO	ppmv (dry)	250
NO_2	ppmv (dry)	10
SO_2	ppmv (dry)	20
HCl	ppmv (dry)	2
Hg	µg/dscm	6.0
Superficial Gas Face Velocity	ft/min	23.58

3.3 Comparison of Test Conditions to Full-Scale Conditions

Process conditions from a full-scale system that is used to capture mercury in a similar application were used to select the superficial gas velocity, carbon bulk density, configuration, and residence time per inch of bed. These, along with the temperature, were all held constant between Columns 1 and 2 and the full-scale system. There are no scale-up factors to apply. The mercury entering the system was in elemental form as is the expected case for full-scale operation downstream of a scrubber. The mercury inlet concentration averaged 6.2 µg/dscm.

4.0 EXPERIMENTAL SYSTEM AND PROCEDURES

A system designed and built for evaluating activated carbon for mercury abatement in a high-mercury-concentration flue gas was modified for use in this test program.

4.1 Test Columns

Figure 1 is a conceptual design of a column used for the testing. The system included two parallel columns made up of six separate glass stages separated by perforated Teflon support disks/gaskets. Each stage section was 3.75 inches inside diameter. Steel clamps that were torqued

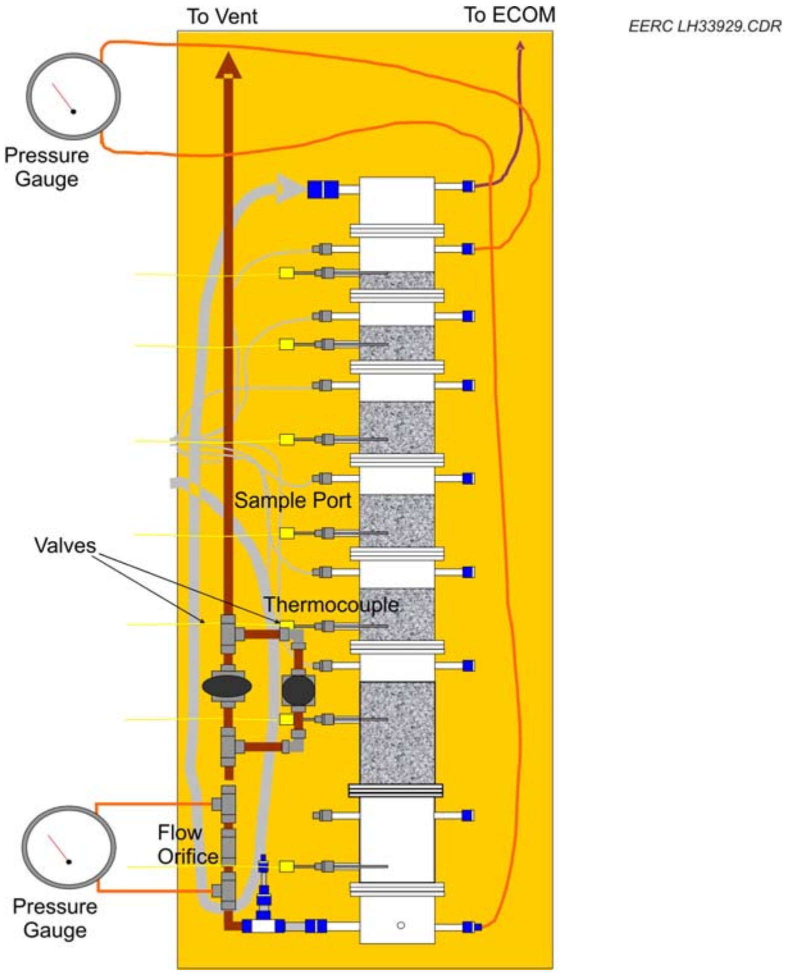


Figure 1. Conceptual schematic of one column.

to a consistent value to ensure a uniform seal held the stages together. Each column was supported the entire length by a threaded rod, which was attached at the top and bottom of the cabinet. Sampling ports were located near the top of each stage. Teflon tubing connected the individual solenoid valves to the ports located in a heated control box between the columns. The common sampling header for this system was also in the heated box. Figure 2 is a conceptual design of the hot box that maintained some of the switching valves and the sampling system at an elevated temperature. The ⅛-inch sample lines were joined in a common header. The headers were connected to heated-head sample pumps. The output from the pumps supplied sample to the CMM conditioning/conversion system.

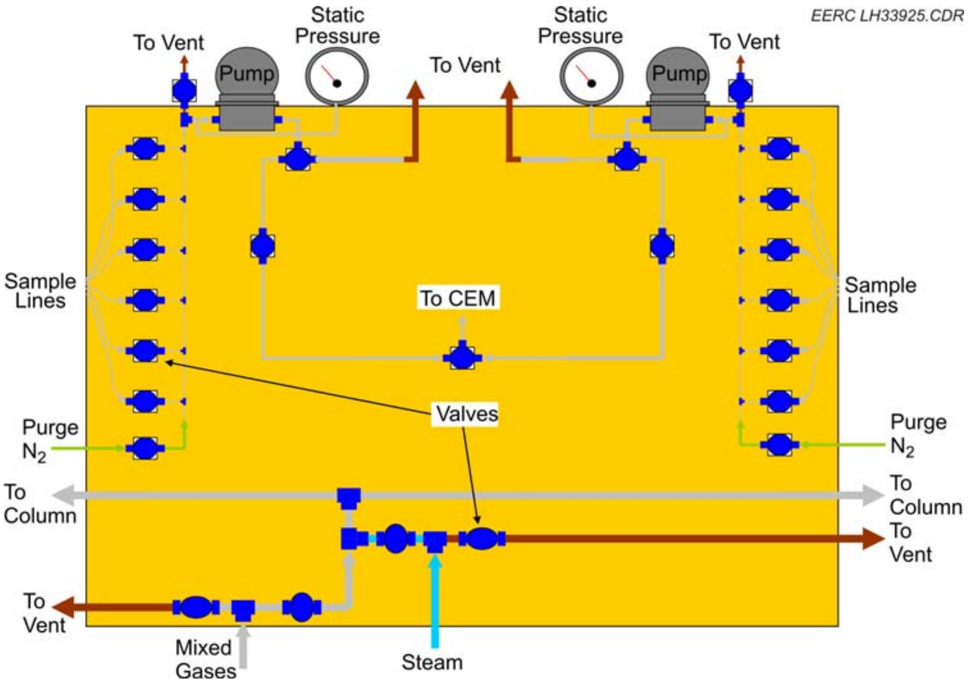


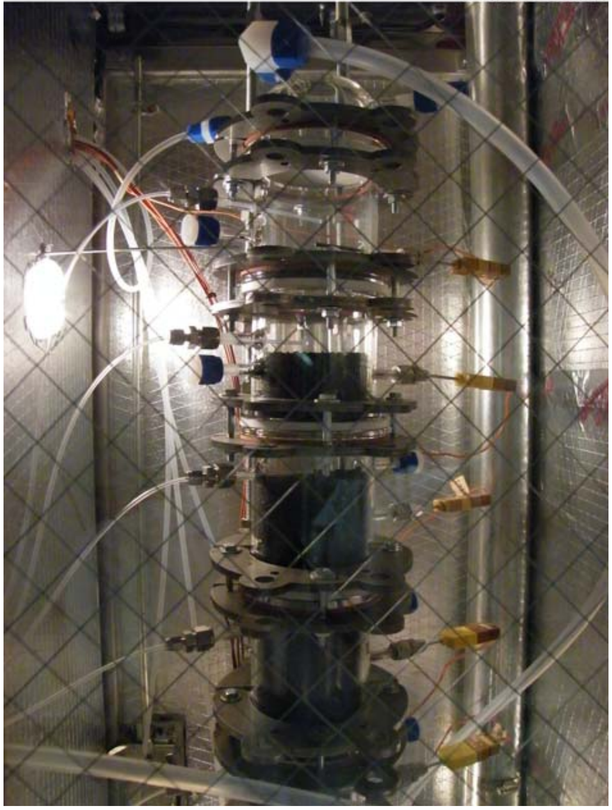
Figure 2. Schematic of the hot box showing sampling configurations.

A data acquisition and control (DAC) system individually controlled each valve. The temperature of each bed was monitored by a thermocouple located near the bottom of each stage section. The cabinets holding the reactor columns were maintained at temperature by using high-temperature blowers to circulate hot air. Heaters were located in an overhead plenum and controlled by external temperature controllers. Figure 3 is a photo of the front of the cabinets holding the columns and the hot box in between. A photo of an example of the carbon bed setup is shown in Figure 4.

The moisture level in the flue gas was controlled by injecting water into a steam generator with a peristaltic pump. The water delivery rate was automatically controlled with the DAC system by monitoring loss of weight of the water reservoir. The mass feed rate of water into the steam generator was independently calibrated by monitoring the pump feed rate. The water feed rate was also monitored with a manual variable-area flowmeter.

EERC LH31318.CDR

Figure 3. Photo of the column cabinets and the hot box.



EERC LH33933.CDR

Figure 4. Photo of a loaded column.

The DAC system was used to select the specific sampling points. A Teflon 3-way valve was used to select between columns. Gases not being sent to the CMM were vented. The common sample headers could also be purged with N_2, either with the exit purge gas going to vent or to the CMM. This provided a means to monitor and clean the system.

4.2 DAC System

National Instruments components were used to construct the DAC system, which was written in LabVIEW graphical language. The front panel of the program acted as the user interface. The DAC system was used to do the following:

- Monitor and control gas flows into the system with mass flow controllers
- Monitor and control the water supply rate to the steam generator
- Monitor flow out of each column
- Monitor system temperatures (beds, cabinets, and steam generation system)
- Control all of the sampling valves
- Integrate with the bed outlet CMM to synchronize sampling
- Log process data every 5 minutes

Metering of component gases into the mixing manifold through the mass flow controllers was controlled by the DAC system. The mass flow controllers were initially calibrated with a Gilibrator bubble-type flowmeter to produce a response curve to convert an input, in units of standard cubic centimeters per minute (sccm), into voltage sent to the flow controller. This calibration was performed with nitrogen for the trace gases that were in a balance of nitrogen and with the gas itself for the CO_2 and O_2. The mass flow controllers have internal closed-loop control to maintain the desired flow and an output representing the actual flow. This voltage was read by the DAC system and converted back into a sccm value, displayed, and recorded.

The flow between the columns was balanced by manually adjusting a back-pressure valve at the exit of each column and by monitoring the pressure drop across a flow orifice at the exit of each of the columns. Identical 0.25-inch-diameter orifices were installed at the exit of each column to produce a ΔP of 1 to 2 inches W.C. at the desired flow conditions.

System temperatures were monitored with Type K thermocouples and logged by the DAC system. These were wired to input modules designed specifically for thermocouple voltage measurements and mounted on terminal bases intended to minimize thermal gradients in the input module, which would adversely affect the relative accuracy of the different channels. The most recent readings could be viewed on the DAC system screen. The temperatures monitored by the DAC system included the following:

- Individual bed segments
- Upper- and lower-level temperatures in the column enclosure cabinet
- Steam generation system (inlet, interior, and outlet)
- Mercury vapor source oven
- Sampling hot box

The sampling valves for mercury measurement were controlled by the DAC system so that individual points could be selected manually or a sequence of locations could be set so that multiple locations could be sampled for various times over a 24-hour period.

An alarm system was implemented to provide on- and off-site process alerts 24 hours a day, 7 days a week. The system consisted of two levels of alarms for the DAC system. Set points for triggering the alarms were determined during the initial part of the run. The alarm system did not include the CMMs.

<u>Level One: Process Alert and Alarms</u>

The set points for this level of alarm were designed to identify and alarm process instrumentation that was out of the desired range and required test personnel intervention and action to troubleshoot and return the modeling system to normal operation. Short-term excursions of this level were not expected to impact modeling data or personnel safety, but action was required to identify the source of the alarm and perform corrective action within

1–2 hours. Data generated during any system excursion required review and may have been excluded from the data analysis following evaluation.

Level Two: High (critical alarms)

These set points were established to identify immediate control system intervention to protect the modeling equipment, facility, or personnel. These were supported by Level One alarms and were designed to shut down and make safe some or all of the systems in the event of a significant loss of system function or rapidly changing conditions indicating an out-of-control situation.

4.3 Gas Delivery System

All compressed gases for producing the target gas concentrations were purchased from Airgas Specialty Gases. The liquid CO_2 was industrial-grade. The O_2 was obtained from the available house air supply. The CO_2 and air passed through a carbon trap and a HEPA (high-efficiency particulate air) filter before passing through the mass flow controller to ensure no mercury contamination. The SO_2/N_2, HCl/N_2, NO/N_2, and NO_2/N_2 gases were certified standard mixtures. Each of these cylinders came with a certificate of analysis that showed the actual concentration of the desired gas ±2% compared to National Institute of Standards and Technology (NIST)-traceable calibration standards. The flow rates for these gases were adjusted by the DAC system based on the actual concentrations in the cylinders input by the operator. Nitrogen was used as a moisture carrier gas, a mercury source sweep gas, and a mercury sample line purge gas. The source of nitrogen was from a central EERC cryogenic nitrogen supply facility.

Porter mass flow controllers were used to meter the flow of each gas into a gas-mixing manifold. Rotameters were used for backup and as a visual check. Each mass flow controller was calibrated during shakedown with a Gilibrator system, which is a primary standard for flow calibration. The atmospheric pressure and gas temperature were recorded for each calibration, along with the mass flow controller set point and actual measured gas flow rate. Linear calibration curves were then generated for flow at standard conditions (1 atm and 0°C) as a function of set point.

Mass flow controllers were controlled and monitored by the aforementioned LabVIEW program. Gas concentrations were entered into a Microsoft Excel® spreadsheet, which computed the required flow rates for each gas. These flow rates were entered into the LabVIEW program. This software used calibration data for each mass flow controller to adjust the digital-to-analog converter.

The moisture level was controlled by injecting deionized water into a steam generator with a peristaltic pump. The water feed rate was controlled by the DAC. The water feed rate was also monitored with a manual variable-area flowmeter. The steam generator temperatures were monitored and logged with the DAC system.

4.4 Mercury Vapor Source

A mercury vapor source was configured using Hg permeation devices from VICI Metronics. The permeation tubes are small, inert capsules that emit a stable concentration of Hg at a given temperature. The tubes were inserted in a glass reactor supplied with a constant flow of heated carrier gas. The glass reactor was maintained at a constant temperature (±1°C) in a convection oven. A nominal mercury concentration to the columns of 6.0 $\mu g/m^3$ was established during the weeks prior to the start of the test. The Hg concentration at the inlet to the columns was periodically checked throughout the test to ensure the mercury generation was stable.

4.5 Mercury Measurement System

A CMM was used to measure the mercury at the exit of the individual bed stages for the duration of the test. A conditioning/conversion system was used to remove moisture and acid gases before the analyzer. In order to speciate between elemental and oxidized forms of mercury in the sample gas stream, the conditioning/conversion system either reduced all forms of mercury to elemental mercury for a measurement of total mercury concentration or removed the oxidized forms of mercury from the sample gas stream for a measurement of elemental mercury concentration. It should be noted that the mercury analyzer was only capable of measuring elemental mercury in the gas stream. The oxidized mercury forms (Hg^{2+}) were reduced to elemental mercury (Hg^0) by passing the sample gas through an impinger containing a solution of 20% sodium hydroxide (NaOH) and 2% stannous chloride ($SnCl_2$). Each solution also removes the HCl in the sample gas, which can poison the gold traps used to capture and concentrate the mercury. The sample gas exiting each bubbler passed through a chilled gas–liquid separator to dry the sample gas. The difference between the total mercury concentration and the elemental mercury concentration was assumed to be an oxidized form of mercury.

A P S Analytical (PSA) Sir Galahad online mercury analyzer was used for outlet sampling. The analyzer measurement is based on atomic fluorescence using the 253.7-nm line from a mercury vapor lamp. The mercury from the sample stream was concentrated by amalgamation onto a gold trap. The gold trap collected mercury for 2 minutes, after which it was purged with nitrogen and heated. Valves directed the released mercury into the optical stage for analysis. The total analysis time for an individual datum point was approximately 5 minutes.

The CMM was employed to measure the mercury concentration at the exit of each bed intermittently as a function of time. The CMM was set to measure total mercury as the normal operating mode. The priority was to obtain stable total mercury measurements wherever breakthrough had been observed. The intent was to determine the elemental mercury concentration if breakthrough was observed at the exit of the column. There was no interest in determining the mercury speciation between the beds. Since there was no mercury breakthrough at the exit of either column, elemental mercury measurements were not taken. The CMM was calibrated to detect mercury concentrations as low as 0.01 $\mu g/m^3$.

4.6 Test Sorbents

The two test columns contained DARCO Hg-LH in Column 1 and MERSORB HT-1.5 in Column 2. Each column consisted of six beds loaded with a total of 18 inches of material.

Column 1 was loaded with a sorbent provided to the EERC by Norit Americas. A material safety data sheet (MSDS) was provided to the EERC. The beds contained a granular activated lignite-based brominated activated carbon adsorbent designated by DARCO Hg-LH 4×8 and referred to as DARCO in this report.

Column 2 consisted of beds loaded with MERSORB HT-1.5, a 1.5-mm pelletized bituminous coal-based SIC supplied by NUCON International. A MSDS was provided to the EERC.

5.0 TEST RUN PERIODS AND POSTRUN ANALYSES

5.1 Shakedown

Prior to the formal start of the model run, shakedown testing was conducted to meet the following criteria:

Mercury source mass delivery rate and stability would be demonstrated by continuous monitoring with an independently calibrated CMM. The target Hg permeation rate was 25 μg/hr, which is equivalent to a column inlet mercury concentration of 6 μg/dscm at process conditions with 18% O_2.

System leak integrity was determined by static pressure-testing the entire system. Any observed leaks were corrected so that no leaks were observed at a static pressure greater than the operating pressure of the columns.

Temperature control stability would be demonstrated within the following tolerances:

- Mercury source: ±2°F
- Heated lines: ±25°F
- Inlet temperature to columns: 180 ± 5°F
- Heated enclosures for columns: ±10°F

The final set points for the mercury source, heated lines, and column enclosures were determined during the shakedown period.

Gas flow accuracy was demonstrated by independent calibration of the individual mass flow controllers within 5% of individual set points. Balance of flow between the two columns was demonstrated to be within 5% of each other by comparing outlet orifice readings at the exit of each column. A back-pressure valve at the outlet of each column was used for fine adjustment of the balance of flow between the columns.

Performance of DAC data logging was proven by demonstrating successful logging of all system inputs for a 24-hour period.

DAC sampling control was demonstrated by showing that the DAC program could successfully sample at each of the stage outlets. The time required for obtaining steady measurements and for switching from one port to another port depended on the mercury concentrations at the outlet of individual bed segments, and this time changed throughout the run. At the beginning of the run, this time was expected to be approximately 1 hour.

5.2 Test Run Start-Up and Data Collection

Once the shakedown testing was complete, the columns were loaded with sorbent material, and the test run was started.

The DAC system was programmed to log all of the gas component flows, system temperatures, port selection, and continuous mercury data. The system was set up to operate overnight without operator assistance. However, the system was monitored by an operator during a normal 8-hour day shift as well as checked during the evening. In addition, each weekend day, the system was checked twice a day. The operator was responsible for performing, as a minimum, the following checks:

- Compressed gases – document the tank pressures
- Gas component flows – visually check all backup manual flowmeters
- Temperature controllers – verify readouts and set points
- Mercury source – temperature, pressure, and flows
- Mercury conversion system – visual check of bubblers, pumps, and solutions
- Carbon columns – visual check, temperatures, and flow balance
- Mercury analyzer – check data for last 24 hours
- DAC system – check sampling and system-logged data for last 24 hours

In addition to filling out a daily checklist and manually recording data for the system, a daily logbook was maintained and signed by the operator to note any system changes, problems, corrective action, or operational concerns. The following data were collected during the course of the test from the DAC and CMM:

- Cumulative run time (hr)
- Test column number (e.g., L = left = Column 1 filled DARCO Hg-LH lignite-based brominated activated carbon; R = right = Column 2 filled with MERSORB HT-1.5 pelletized bituminous coal-based SIC)
- Bed segment outlet number
- Cumulative bed depth (inches)
- Mercury concentration (µg/dscm)
- Total (full time)

At a minimum, the following Hg-sampling guidelines were observed:

- The primary focus of bed outlet data collection was on beds in both columns that were initially breaking through (e.g., >0.5 µg/dscm) as well as beds immediately before and after the beds that were initially breaking through.

- The secondary focus of data collection was to be on beds that were breaking through at higher mercury concentrations but not at the expense of collecting sufficient-quality data from the beds that were initially breaking through or were indicating nondetectable breakthrough.

- The sequence of bed sampling was from lower-concentration beds to higher-concentration beds except when switching from one column to another when this may not be possible. As a general guideline, when sampling from a given bed (e.g., Bed 2) indicated a total Hg concentration greater than 0.5 µg/dscm, the next sampling cycle in the column began two beds downstream of this bed (e.g., Bed 4) and then proceeded to the next bed upstream (e.g., Bed 3).

- Adequate time was to be allowed for sampling from a given bed to accommodate the time required to purge residual mercury from the sampling line and manifold system.

- Adequate gas sample residence time on the CMM's gold trap was to be provided to give accurate low-mercury-concentration measurements (e.g., <0.2 µg/dscm).

- The calibration of the CMM was checked daily by injecting a known quantity of mercury at the calibration port and determining the percent recovery based on the theoretical mass of mercury injected. If the recovery was greater than ±10%, the PSA was recalibrated. The zero of the analyzer was also checked daily.

- Mercury inlet concentration measurements were periodically checked with the CMM.

5.3 Test Run Periods

The test was initially planned as a long-term 1000-hour run. Except for noted minor upset events, operating conditions were held constant during this period. At Run Hour 1009, the Hg, steam, and acid gases were stopped. After a 5-minute purge with dry air and nitrogen, the remaining dry gases were turned off, and the systems were allowed to cool to room temperature before the carbon samples were removed.

5.4 Postrun Sample Recovery and Analyses

Following completion of the model run, the samples of carbon from each stage were recovered and stored in an appropriately sized glass sample container with a Teflon lid. The postrun sorbent mass of each stage was recorded. To obtain a representative sample for the analyses, each stage was physically mixed by gently tumbling the recovered sorbent material prior to submitting the sample to the EERC Analytical Research Laboratory (ARL).

The first two stages from each column were analyzed for total mercury by taking a representative sample for that stage. Laboratory analyses of the carbons were performed by digesting approximately 1-g samples with concentrated hydrochloric acid and nitric acid (6 and 4 mL, respectively) in a capped digestion vessel. The digestion samples were placed on a dry block heater at 90°C for 4 hours. After digestion, the samples were cooled to room temperature, and the solutions were each brought up to a 50-mL volume. Undiluted samples were analyzed by cold-vapor atomic absorption (CVAA). To fall within the calibration range of the CVAA (0–5 µg/L), the solutions were diluted using volumetric pipettes and flasks.

6.0 RESULTS AND DISCUSSION

6.1 Sorbent Mass

The columns were loaded with as-received carbon samples, and the test was begun April 20, 2009. Tables 3 and 4 show the sorbent mass loaded into each stage of the two columns. At the end of the test, the sorbent from each stage was recovered and weighed, reported in Tables 3 and 4, to determine the mass gain that occurred from exposure to the mercury, flue gas components, and moisture.

Table 3. Column 1 DARCO Hg-LH Sorbent Mass Before and after the Run

Stage	Stage Depth, in.	Cumulative Bed Depth, in.	Loaded Sorbent Mass, g	Postrun Sorbent Mass, g	Mass Gain, g	Mass Gain, %
1	1	1	95.2	149.6	54.4	57.1
2	2	3	190.1	246.6	56.5	29.7
3	3	6	276.5	319.3	42.8	15.5
4	3	9	278.7	293.7	15.0	5.4
5	3	12	262.4	258.1	−4.3	−1.6
6	6	18	525.0	519.7	−5.3	−1.0
Total	18		1627.9	1787.0	159.1	

Table 4. Column 2 MERSORB HT-1.5 Sorbent Mass Before and after the Run

Stage	Stage Depth, in.	Cumulative Bed Depth, in.	Loaded Sorbent Mass, g	Postrun Sorbent Mass, g	Mass Gain, g	Mass Gain, %
1	1	1	103.0	181.9	78.9	76.6
2	2	3	189.8	300.2	110.4	58.2
3	3	6	295.2	390.3	95.1	32.2
4	3	9	281.8	304.6	22.8	8.1
5	3	12	282.4	306.0	23.6	8.4
6	6	18	554.2	596.4	42.2	7.6
Total	18		1706.4	2079.4	373.0	

Given the mercury exposure time for each column, the target mass of added mercury was approximately 13.3 mg to each column. The additional mass gain to each column can be attributed to moisture and acid gases retained by the carbon during the test. The higher weight gain observed with the MERSORB may be caused by the formation of SO_4 from the sulfur already on the carbon.

6.2 Process Measurements

Process data logged by the DAC included gas flow measurements, sorbent bed temperatures, cabinet temperatures, flow balance, and mercury source temperatures. Other aspects of the process were manually recorded daily, including the column pressure drop. Only three minor upset conditions occurred during the 1000-hour run. The first was roughly 156 hours into the test; the SO_2 cylinder ran out, and no alarm was sent. There was no SO_2 gas flow for 9 hours. The second and third upset conditions occurred approximately 180 hours and 206 hours into the run when the HCl gas cylinder regulator failed. There was no HCl gas flow to the columns for 7 hours and 3 hours, respectively. It was determined that an alarm bypass for the SO_2 and HCl gas flows (used in the previous test program) had been triggered, and no alarms were sent out for any of the upset conditions. The alarm was reset, and there were no further problems. The loss of these gas flows for the short periods of time should not affect the overall performance of the activated carbons to capture mercury.

6.2.1 Gas Flow Measurement

The total and individual dry gas flow rates based on the individual mass flow measurements for the various inlet gases were steady throughout the model run. When a tank of one of the acid gases was changed, that flow was adjusted based on the certified gas concentration of the new tank. With each gas tank change, the nitrogen balance was adjusted to maintain a total constant flow.

The moisture level was controlled by injecting deionized water into a steam generator with a peristaltic pump. The water feed rate was automatically controlled by the DAC based on weight loss measurements from a water supply tank. The water feed rate was monitored manually every day with a variable-area flowmeter.

The individual dry gases were also monitored with rotameters in series with the mass flow controllers. During previous testing, a comparison of the mass flow controller and rotameter data showed that the total dry flow measurements for the mass flow controllers were within 5% of the total flow indicated by the rotameters.

The flow between the columns was balanced by monitoring a flow orifice at the exit of each of the columns. Identical orifices were designed with a 0.25-inch diameter to produce a ΔP of 1 to 2 inches W.C. at the desired flow conditions. The logged orifice ΔP readings for each column, shown in Figure 5, indicate that the orifice readings were well within 0.05 inches of

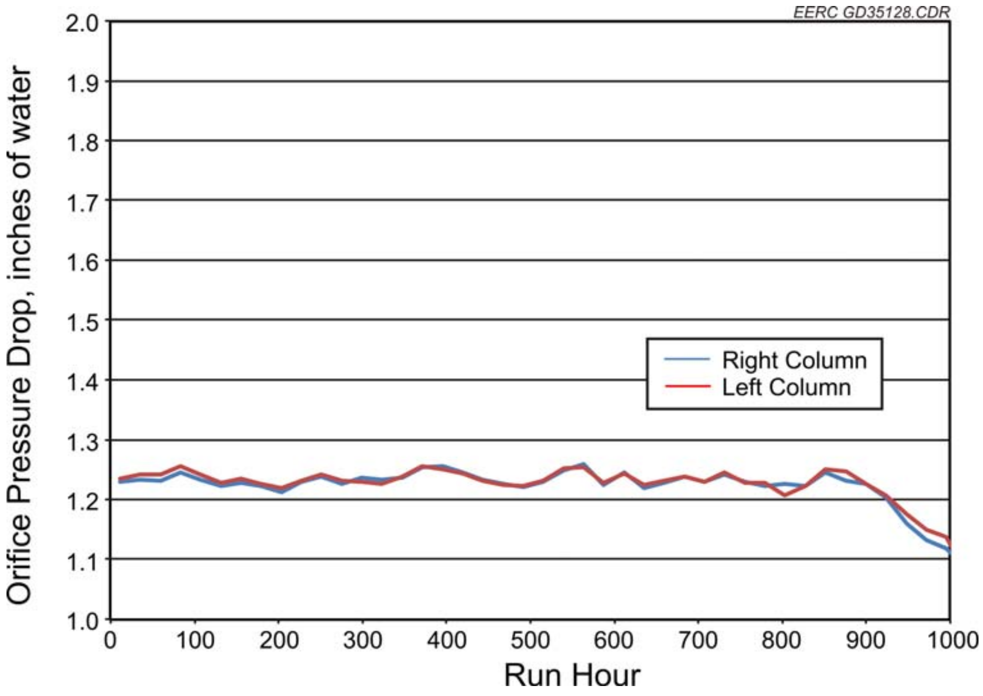


Figure 5. Flow balance indicated by orifice ΔP for each column.

each other for most of the run, which corresponds to the flows balanced to within 2% of each other. The deviation around 800 hours into the run was caused by condensation in one of the lines to the pressure transducers.

6.2.2 Bed Temperatures

The bed temperatures for each stage were logged continuously by the DAC. The temperature recorded for Bed 1 was the temperature of the inlet gas stream since the thermocouple for Bed 1 of each column was above the 1-inch-thick sorbent depth of this stage. For Beds 2–6, the thermocouples were within the carbon sorbent depth. Figure 6 shows the daily averaged temperature throughout the test for Column 1, which contained DARCO Hg-LH. Figure 7 shows the similar information for Column 2, containing MERSORB HT-1.5. The temperatures were within a few degrees of the target temperature of 180°F. The temperatures among the stages were within good general agreement. Initial temperature excursions at start-up

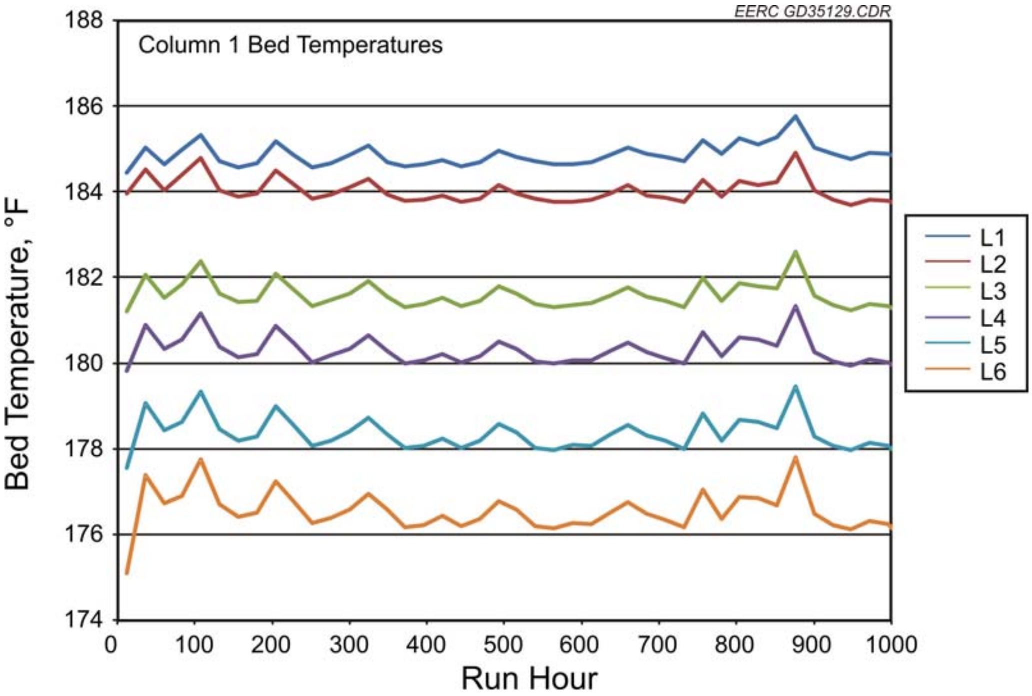


Figure 6. Daily averaged Column 1 bed temperatures.

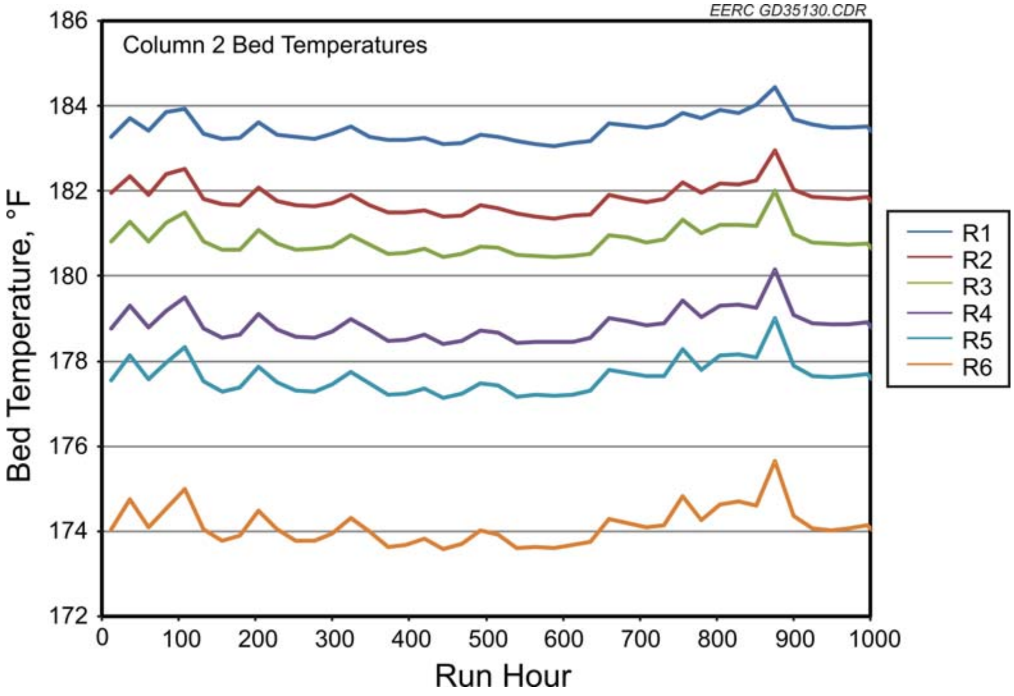


Figure 7. Daily averaged Column 2 bed temperatures.

do not show up on the daily average plot, but they have been seen in previous tests and are attributed to the temperature rise because of the heat of absorption of water when initially exposed to the flue gas.

6.2.3 Column Pressure Drop

Total pressure drop data across the entire column were recorded manually daily for the duration of the run. The total column pressure drop measurements included contributions from the flow resistance through the packed beds of sorbent, the restriction of the perforated flow distribution disks separating each column stage, and the wall friction from flow through the columns. Considering the relatively low gas velocity through the columns, this last component was considered to be negligible compared to the previous two contributors. As part of the previous test program, the pressure drop across the empty system was measured. The contribution of the seven disks to the overall column differential pressure measurements was determined and averaged 2.3 inches of water. The carbon differential pressure was estimated by subtracting the total disk ΔP from the measured total column ΔP measurements, and these data are presented in Figure 8. As indicated in the figure, the normalized differential pressure drop across both carbons increased slightly during the run. Near the end of the test, there was water condensed in the line to one of the pressure transducers used to measure the pressure drop across the flow orifice. After this was cleared, the flow pressure was lower and the pressure drop across the columns was lower. There were no obvious explanations for the changes. All other readings indicated the system was operating normally.

6.2.4 Mercury Source

The mercury source was initially calibrated with the PSA Sir Galahad to deliver 25 µg of mercury per hour (within ±10%). This was with a nitrogen sweep gas rate of 0.1 dslm and a mercury source oven temperature of 276°F. The mass flow controller as well as the backup variable-area flowmeter indicated that the sweep flow was constant. The daily averaged source oven temperatures (Figure 9) were also constant throughout the test.

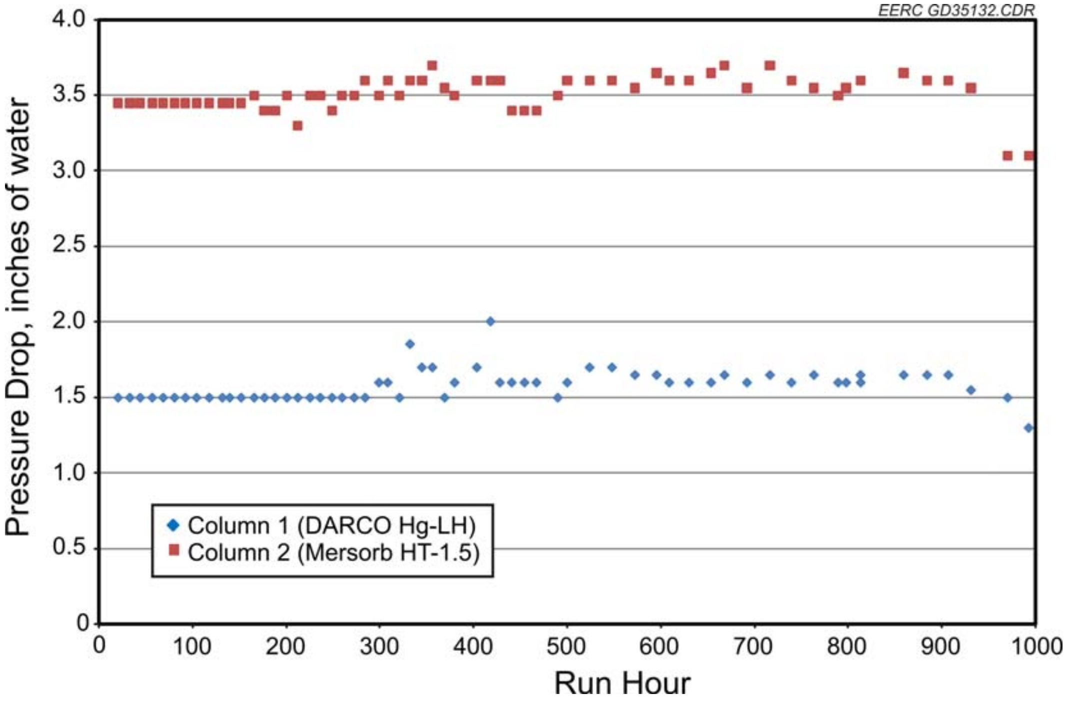


Figure 8. Derived carbon bed pressure drop.

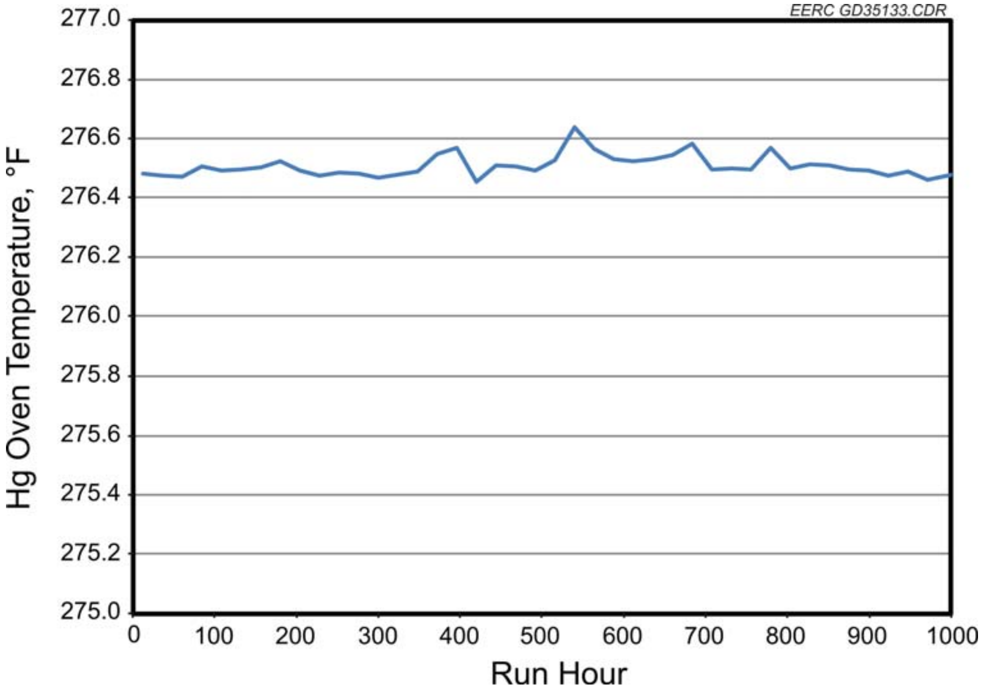


Figure 9. Daily averaged mercury source temperatures.

The averaged measured inlet CMM mercury concentrations to the columns are shown in Figure 10. The inlet concentration to the right column was periodically measured with the PSA CMM. The target inlet mercury rate was 25 µg/hr to achieve the target inlet mercury concentration of 6.0 µg/dscm at the process condition of 18% O_2.

6.3 Mercury Capture and Breakthrough Curves

A single CMM was used to sample mercury from the exit of selected beds. An individual sample was taken approximately every 5 minutes with the CMM. Data reduction was performed on the sample points obtained from the CMM to reduce noise within the instrument readings, subtract the nitrogen zero level, and apply calculations to present the data in the preferred units.

The primary focus of bed outlet sampling was on beds in both columns that were initially breaking through (e.g., >0.5 µg/dscm) as well as beds immediately before and after the beds that were initially breaking through. The general sequence was to sample the nitrogen purge for a

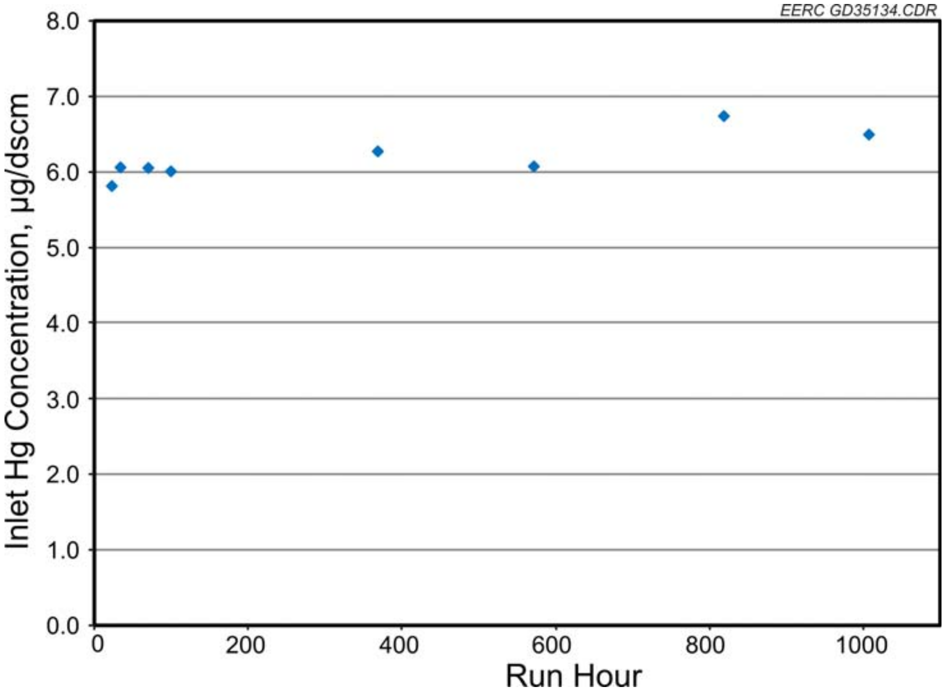


Figure 10. Averaged inlet mercury concentrations.

column and then to sample the outlet mercury from successive beds, including one to two beds past where breakthrough was seen. This procedure was used as an effort to minimize any effect of residual mercury in the sampling lines from a previous stage from biasing the measurement for the next sample. The breakthrough level of 0.5 µg/dscm was considered quantifiable whereas mercury in the 0.1–0.5-µg/dscm range was less certain. The secondary focus was on beds that were breaking through at higher mercury concentrations.

The CMM was calibrated to provide the most accurate readings over the entire measurement range, <0.10 to >6.0 µg/dscm, and additional calibration checks were completed daily to verify that the CMM calibration had not drifted. The CMM was recalibrated if a calibration check indicated it had drifted by more than 10%.

Breakthrough response curves and column-specific results are presented separately for Column 1 in Section 6.3.1 and for Column 2 in Section 6.3.2.

6.3.1 Column 1 Breakthrough Curves (DARCO Hg-LH)

Column 1 contained 18 inches of lignite-based granular brominated carbon (DARCO Hg-LH 4×8) from Norit Americas in six bed segments. Figure 11 shows a plot of all of the reduced Column 1 bed outlet mercury concentration data on the scale of the target inlet concentration. Breakthrough was measured from Bed 1 (1 inch in depth) and, possibly, Bed 2 (cumulative 3 inches in depth). No breakthrough was detected by the CMM for Beds 3 through 6.

Figure 12 plots the mercury concentration at the outlet of Bed 1 as a function of time. One curve represents the “raw” outlet concentration, and the other represents the raw outlet concentration minus the baseline concentration measured while sampling nitrogen through the sampling manifold. The baseline measures the residual mercury in the sampling system. Bed 1, 1-inch depth of DARCO Hg-LH, showed significant mercury breakthrough roughly 300 hours into the run. The breakthrough from Bed 1 then increased throughout the remainder of the test. As the data show, the correction baseline was small compared to the breakthrough levels. By

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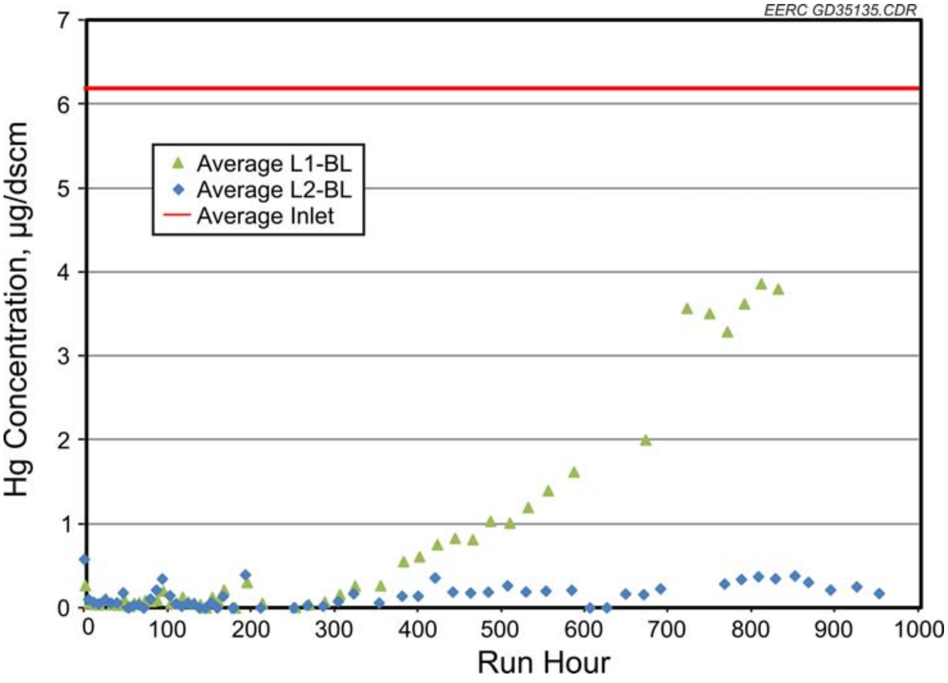

Figure 11. Mercury outlet data for Column 1 containing DARCO Hg-LH compared with target inlet value.

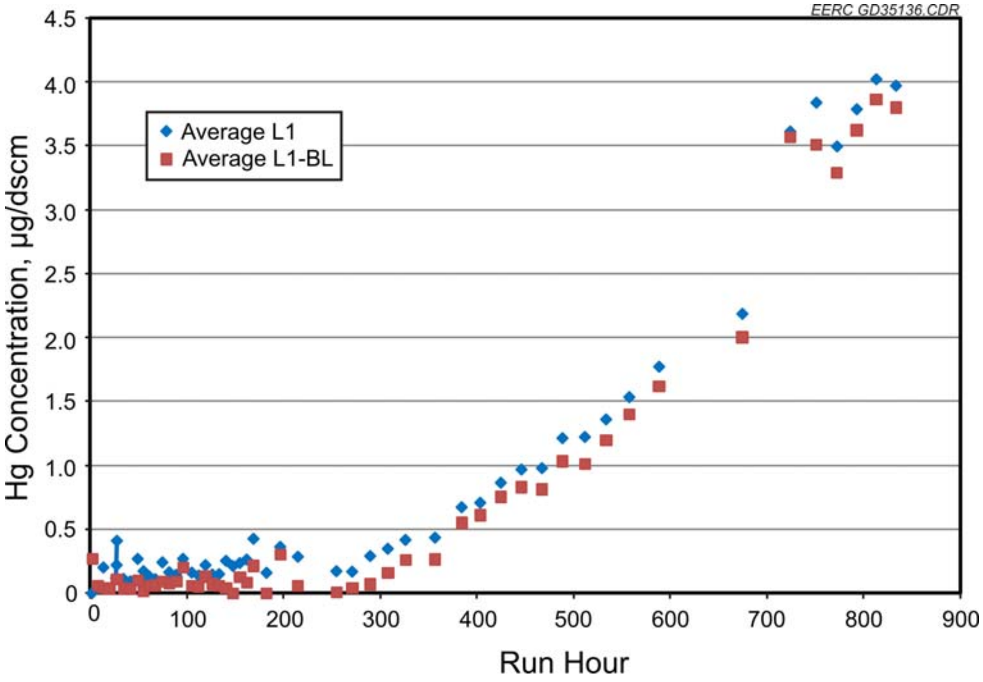

Figure 12. Bed 1 mercury outlet data for Column 1 containing 1 inch of DARCO Hg-LH. The cumulative bed depth is 1 inch.

Run Hour 833, the level of mercury measured at the outlet of Bed 1 was near 4.0 µg/dscm. Measuring mercury at this concentration caused high baseline readings that made it difficult to get an accurate measurement of the mercury concentrations at the exit of Beds 2–6. Therefore, it was decided to stop measuring the mercury concentration at the outlet of the first bed for each column and focus on the low mercury concentrations at the outlet of Beds 2–6. Figure 13 plots the mercury capture efficiency of Bed 1 as a function of time. After 833 hours of exposure, the Hg capture efficiency had dropped to less than 40%, but the remainder of the column was still capturing essentially 100% of the mercury.

All Bed 2 (2-inch depth with a cumulative depth of 3 inches) data are shown in Figure 14. Prior to Run Hour 833, it was not possible to reliably quantify the mercury concentration at the exit of Bed 2 because of the residual mercury in the sampling manifold from measuring the outlet of Bed 1. After discontinuing the measurement of the Bed 1 outlet, it was possible to purge the residual mercury and accurately measure the mercury concentration at the outlet of Bed 2. Figure 15 plots the cumulative capture efficiency at the outlet of Bed 2. For the last 50 hours of the test (when Bed 1 was no longer being measured) the mercury capture efficiency averaged over 97%.

Bed 3 (3-inch depth with a cumulative depth of 6 inches) data are shown in Figure 16. Again, the only reliable outlet mercury concentration data come from the time period after sampling at the outlet of Bed 1 was discontinued. From Run Hour 850 to the end of the test, the mercury concentration at the outlet of Bed 3 dropped. Within the limits of quantification, the data indicate no mercury breakthrough at the exit of Bed 3 by the end of the test. Figure 17 plots the cumulative mercury capture efficiency at the outlet of Bed 3. Over the last 50 hours, the average capture efficiency was 98.9% and increasing. The results show that 6 inches of the DARCO Hg-LH carbon was effective at capturing 100% of the inlet mercury for over 1000 hours.

The cumulative mercury loading for the first two beds was calculated based on the average breakthrough levels observed for each bed for the run and the average inlet mercury

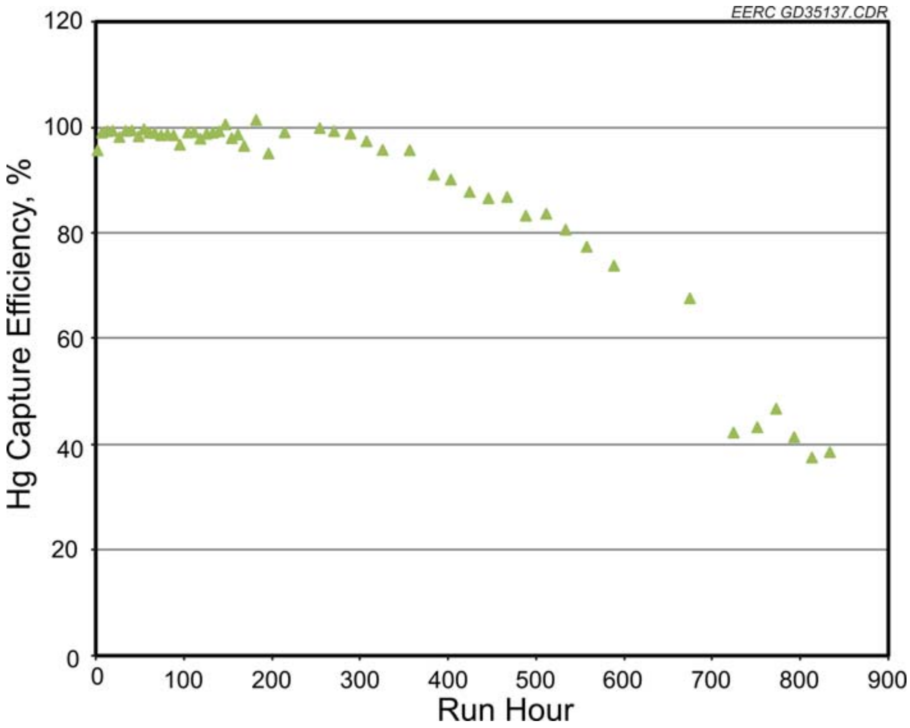

Figure 13. Column 1 Bed 1 cumulative mercury capture efficiency.

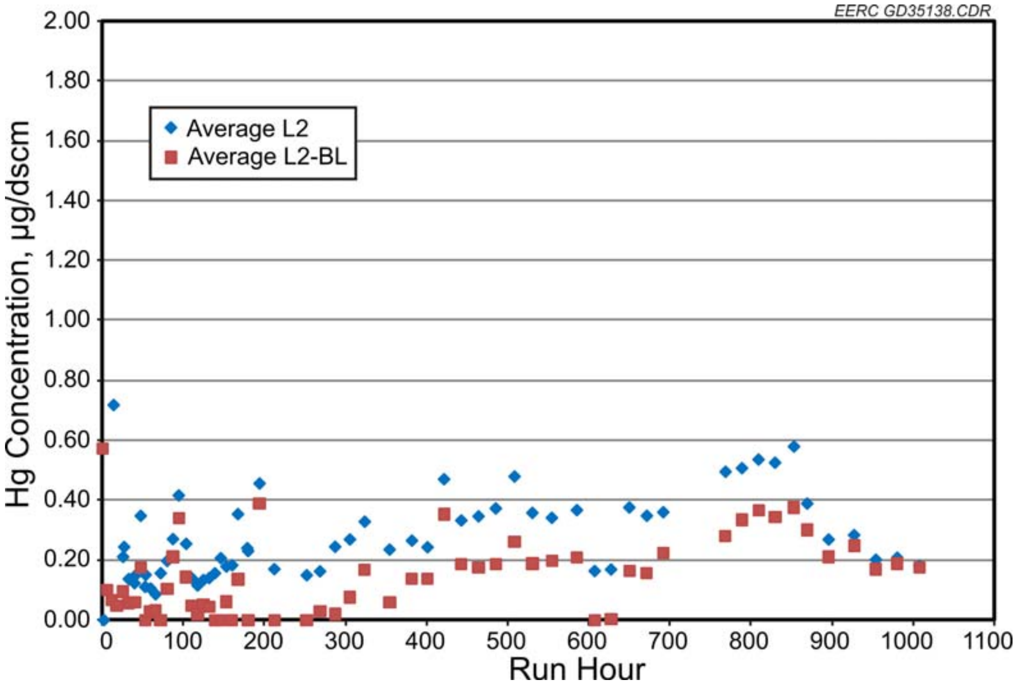

Figure 14. Bed 2 mercury outlet data for Column 1 containing 2 inches of DARCO Hg-LH. The cumulative bed depth is 3 inches.

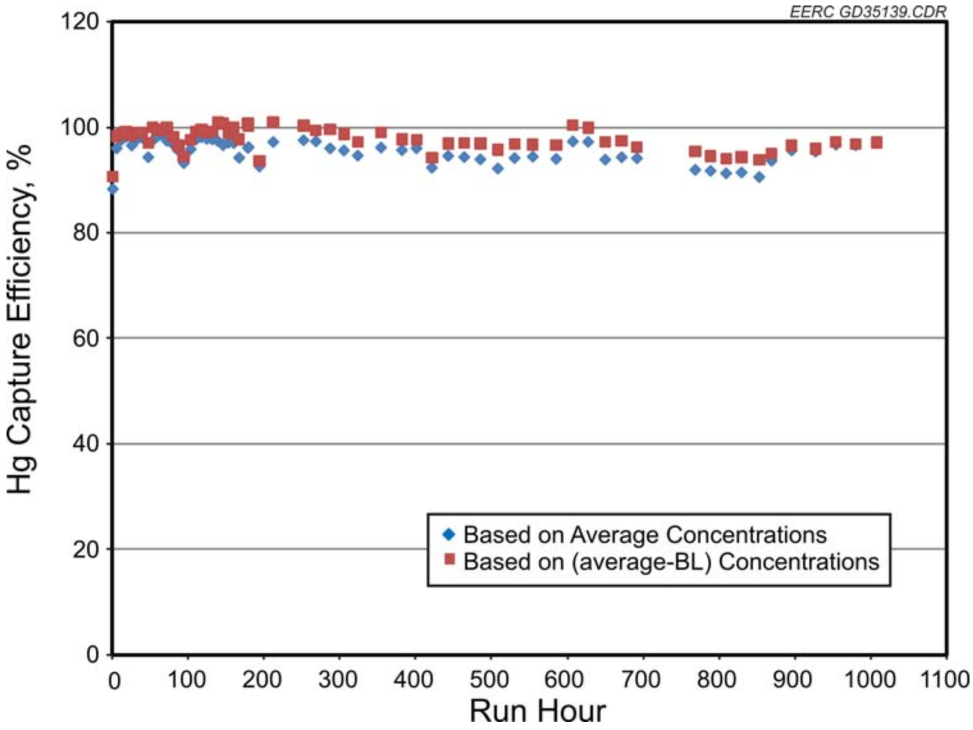

Figure 15. Column 1 Bed 2 cumulative mercury capture efficiency.

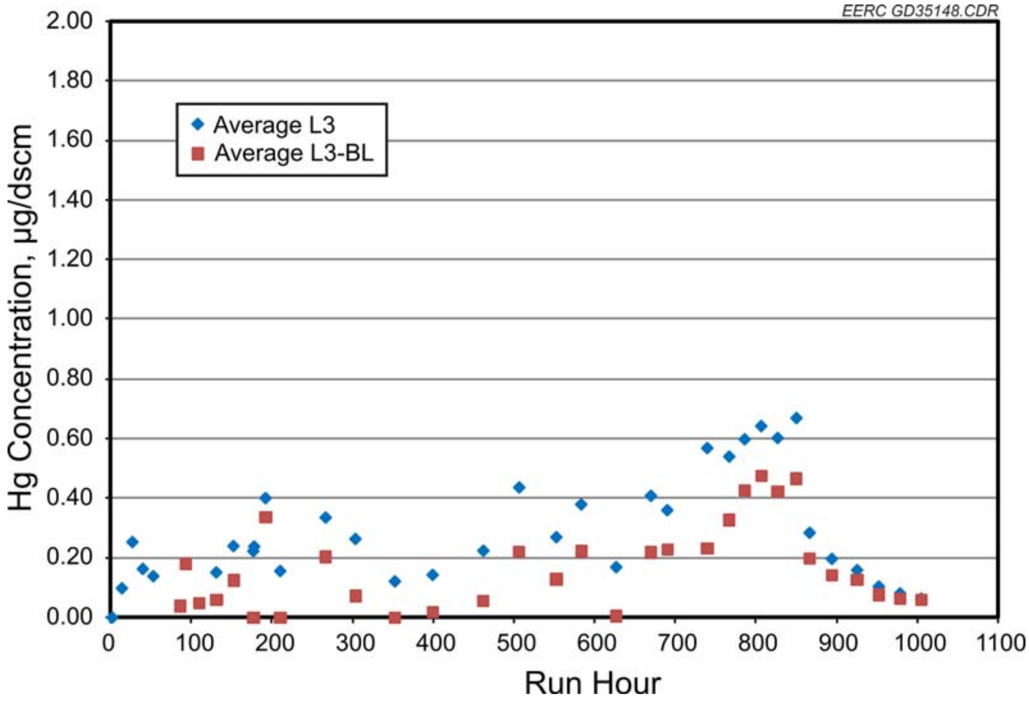

Figure 16. Bed 3 mercury outlet data for Column 1 containing 3 inches of DARCO Hg-LH. The cumulative bed depth is 6 inches.

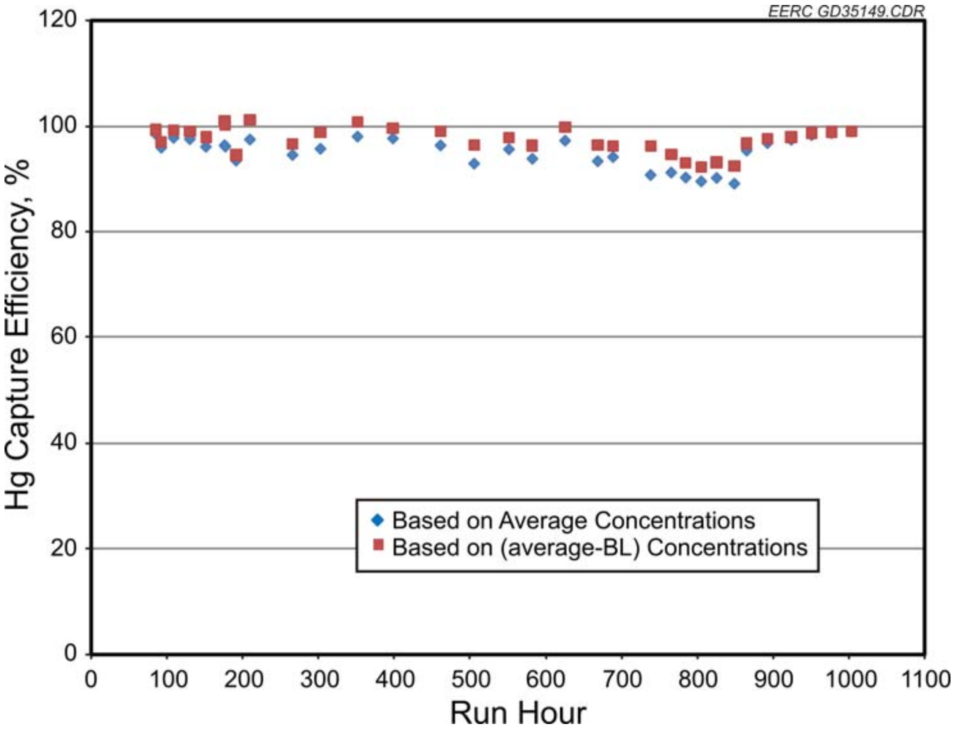

Figure 17. Column 1 Bed 3 mercury cumulative mercury capture efficiency.

concentration measured with the CMM. These data are based entirely on the inlet and outlet CMM data. The data are presented in Figure 18 as cumulative mercury loading in milligrams of mercury per 100 grams of starting carbon mass. The plot only goes up to Run Hour 850 because sampling at the outlet of Bed 1 was discontinued at that time. For comparison, the theoretical cumulative inlet loading to Bed 1 is shown.

6.3.2 Column 2 Breakthrough Curves (MERSORB HT-1.5)

Column 2 contained 18 inches of MERSORB HT-1.5 SIC in six bed segments. Figure 19 shows a plot of all of the Column 2 bed outlet mercury concentration data on the scale of the target inlet concentration. Breakthrough was detected only from Bed 1. Beds 2, 3, and 4 were also monitored.

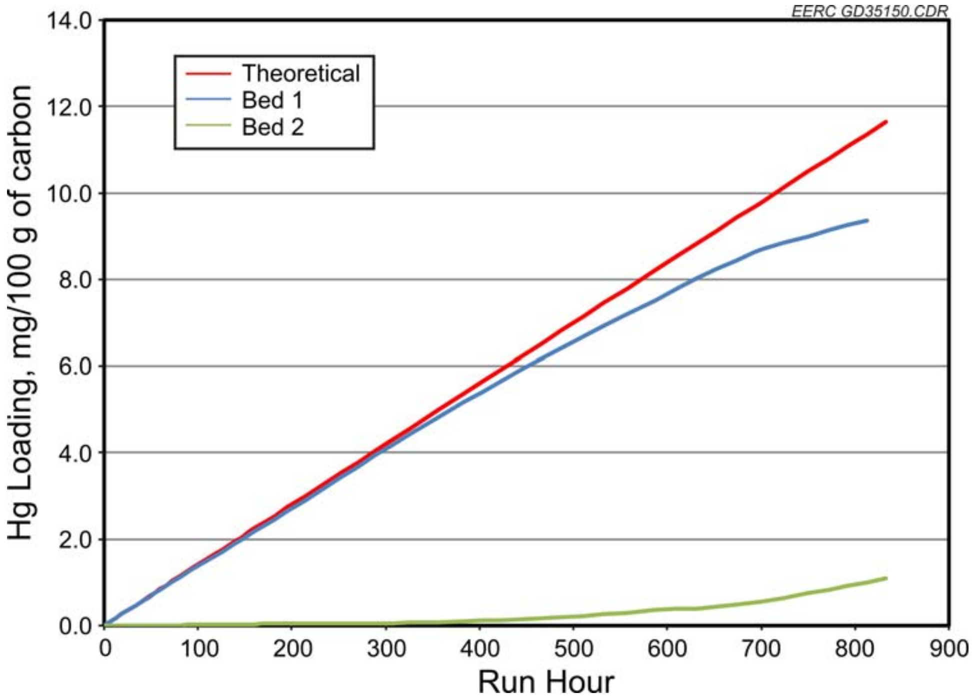

Figure 18. Cumulative Hg loading for Beds 1 and 2.

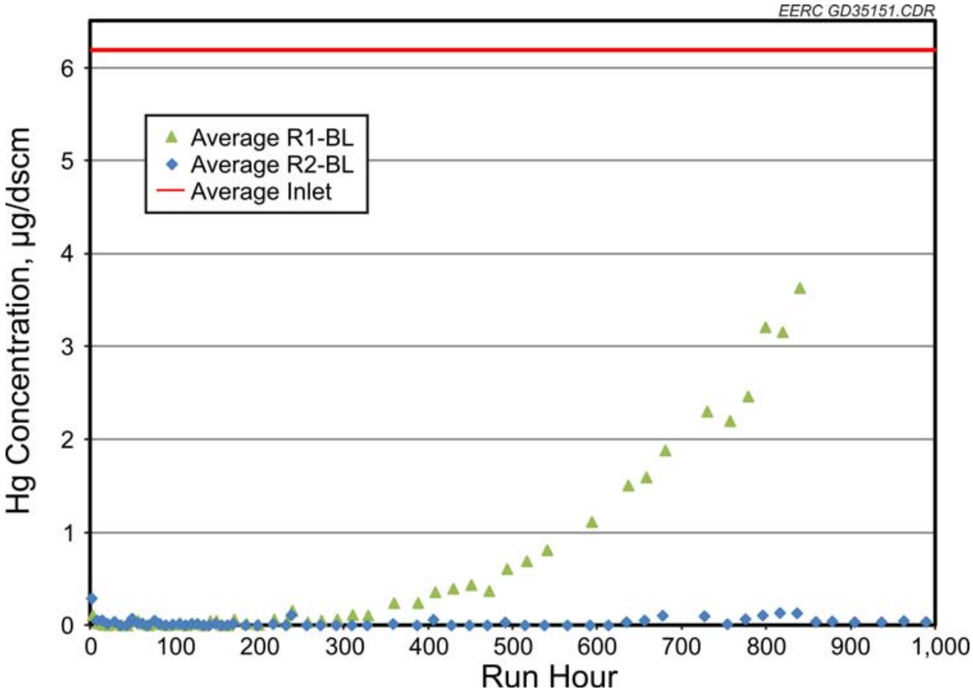

Figure 19. Mercury outlet data for Column 2 containing MERSORB HT-1.5 compared with target inlet value.

Figure 20 plots the mercury concentration at the outlet of Bed 1 as a function of time. Again, the “raw” concentration and the raw minus the baseline concentration are plotted and indicate that the background correction was minor. Bed 1 (1-inch depth of MERSORB HT-1.5) showed quantitative mercury breakthrough roughly 350 hours into the run. The breakthrough from Bed 1 then increased throughout the remainder of the test. By Run Hour 833, the level of mercury measured at the outlet of Bed 1 was near 3.5 μg/dscm. As mentioned above, sampling at the outlet of Bed 1 was discontinued at this time to focus on the low mercury concentrations at the outlet of Beds 2–6. Figure 21 plots the mercury capture efficiency of Bed 1 as a function of time. After 840 hours of exposure, the Hg capture efficiency had dropped to near 40%, but the remainder of the column was still capturing essentially 100% of the mercury.

All Bed 2 (2-inch depth with a cumulative depth of 3 inches) data are shown in Figure 22. Again, as with Column 1, Bed 2 outlet mercury concentrations could not be quantified below 0.2 μg/dscm until sampling at the outlet of Bed 1 had stopped. By the end of the test, the outlet concentration at the outlet of Bed 2 reached less than 0.05 μg/dscm which corresponds to greater than 99% capture efficiency. Figure 23 plots the cumulative capture efficiency at the outlet of Bed 2. For the last 100 hours of the test (when Bed 1 was no longer being measured), the mercury capture efficiency averaged 99.4%. Overall, a total of 3 inches of the MERSORB HT-1.5 captured 100% of the inlet mercury for over 1000 hours.

Similar to Column 1, the cumulative mercury loading was calculated for each bed based on the average breakthrough levels observed for each bed for the run and the average inlet mercury concentration measured with the CMM. The data are presented in Figure 24 as cumulative mercury loading in grams of mercury per 100 grams of starting carbon mass, and the cumulative inlet loading to Bed 1 is shown for comparison.

6.4 Postrun Laboratory Analyses and Mass Balance

The results of the mercury content analyses of the individual stages are shown in Tables 5 and 6. Because of the limited number of analyses that could be performed and the difficulty in

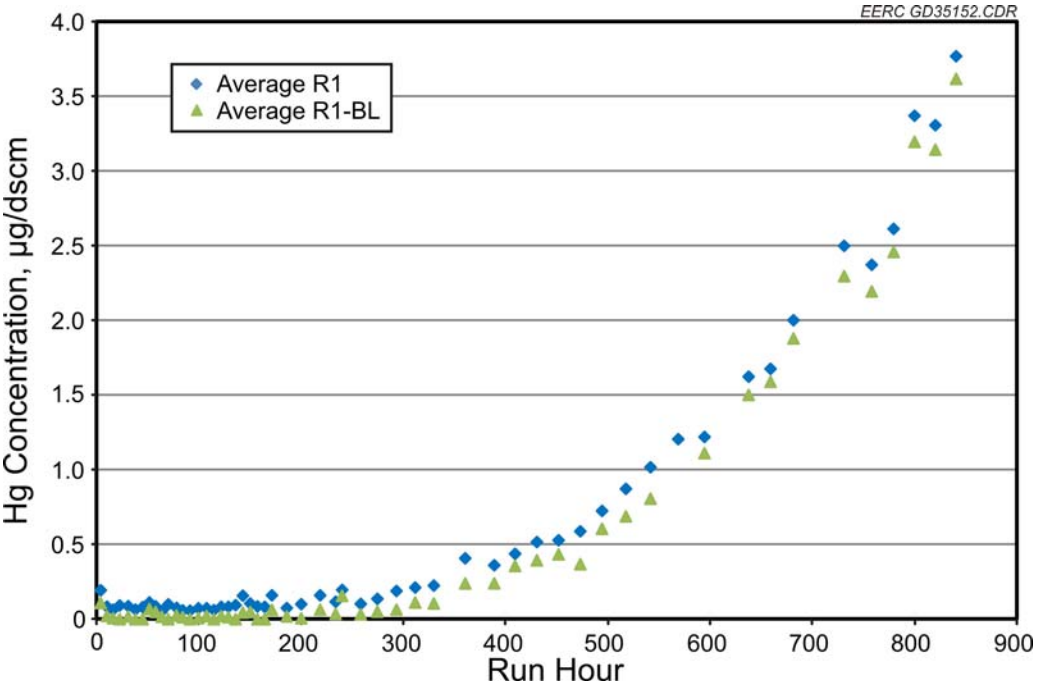

Figure 20. Bed 1 mercury outlet data for Column 2 containing 1 inch of MERSORB HT-1.5. The cumulative bed depth is 1 inch.

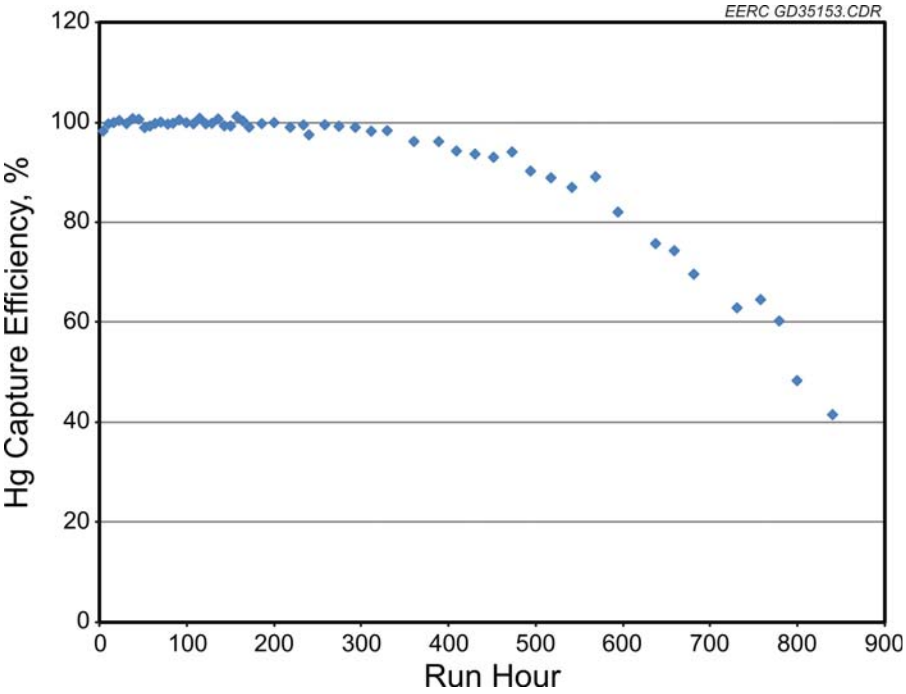

Figure 21. Bed 1 cumulative mercury capture efficiency.

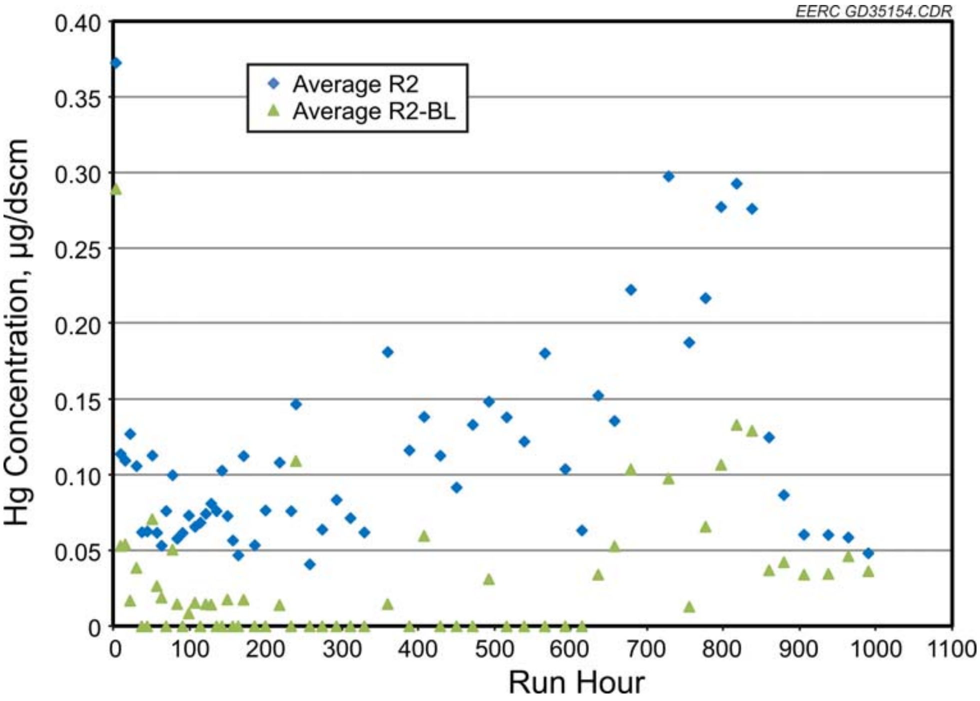

Figure 22. Bed 2 mercury outlet data for Column 2 containing 2 inches of MERSORB HT-1.5. The cumulative bed depth is 3 inches.

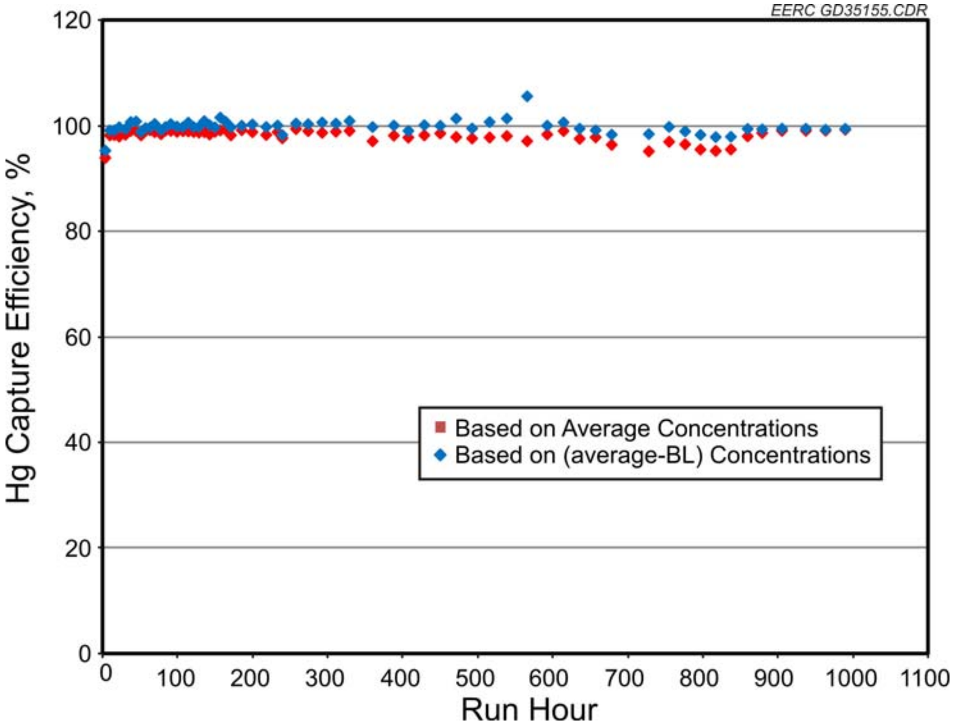

Figure 23. Column 2 Bed 2 cumulative mercury capture efficiency.

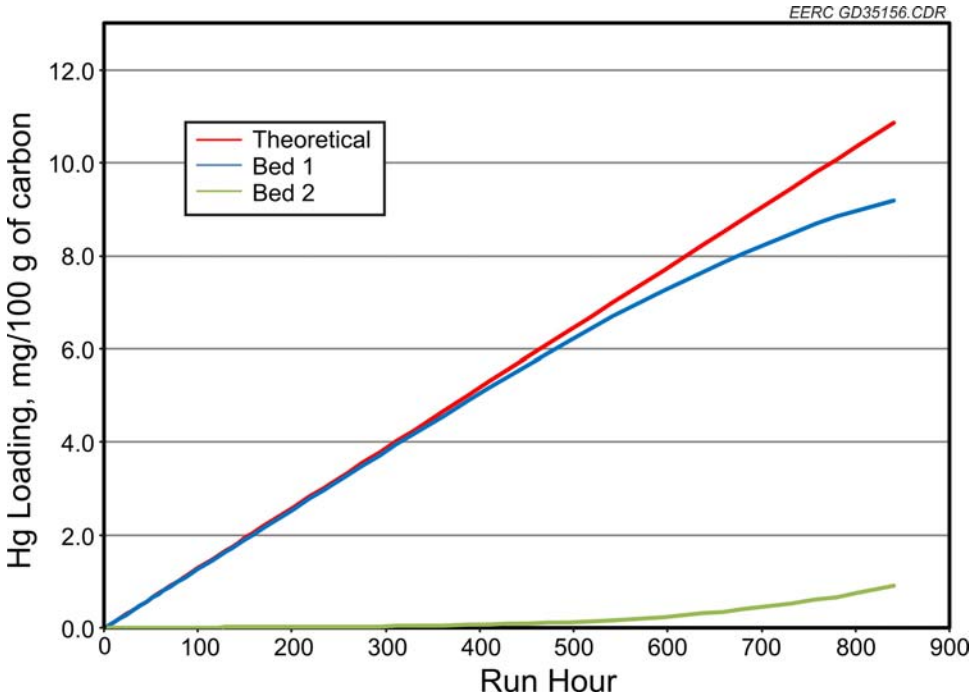

Figure 24. Column 2 MERSORB HT-1.5 projected stage loading throughout the run.

Table 5. Column 1 DARCO Hg-LH Sorbent Laboratory Mercury Analyses

Bed	Stage Depth, in.	Cum. Depth, in.	Prerun Sorbent Mass, g	Postrun Sorbent Mass, g	Mass Gain, %	Hg Conc., µg/g	Total Hg Collected, mg
1	1	1	95.2	149.6	57.1	43.2	6.46
2	2	3	190.1	246.6	29.7	35.5	8.75
Total							15.2

Table 6. Column 2 MERSORB HT-1.5 Sorbent Laboratory Mercury Analyses

Bed	Stage Depth, in.	Cum. Depth, in.	Prerun Sorbent Mass, g	Postrun Sorbent Mass, g	Mass Gain, %	Hg Conc., µg/g	Total Hg Collected, mg
1	1	1	103.0	181.9	76.6	46.0	8.37
2	2	3	189.8	300.2	58.2	26.0	7.81
As-Received Carbon						0.044	
Total							16.2

getting a representative sample from a granular material, the intent of the analysis was not to perform detailed mass balance, but to verify that the mercury was captured by the activated carbon. Calculations of the mercury collected by each stage based on the analytical results and the removal efficiency of each stage are included in the tables. These data are based only on the bed analyses completed after the run and are completely independent from the projected stage loading charts based on the CMM data presented in Figures 18 and 24. The remaining additional mass gain to each column can be attributed to moisture and acid gases retained by the carbon during the run and, possibly, the formation of SO_4 from the existing sulfur on the MERSORB HT-1.5 SIC. It appears more mercury than expected was captured in the second bed of each column. Since sampling at the outlet of Bed 1 was suspended before the end of the test, it is unknown what level of breakthrough was reached or if mercury started to offgas from the first bed and was captured by the second bed. What these analyses do show is that the mercury was captured by the activated carbon in the first two beds.

A comparison that helps to verify the results is the mass balance closure when the total amount of mercury generated based on the inlet CMM data is compared with the amount of mercury recovered based on the laboratory data. Table 7 shows a 118% closure for the bed analysis/CMM inlet average. This level of closure is as good as can be expected considering the limited number of analyses performed.

7.0 DISCUSSION AND CONCLUSIONS

7.1 Discussion

The objective of the test program was to evaluate the ability of a fixed bed of sorbent material to capture mercury in a flue gas stream simulating that generated during the taconite-manufacturing process. The flue gas composition including acid gas and mercury concentrations, the superficial gas velocity, the gas temperature, the carbon bulk density, and the residence time per inch of bed in both Columns 1 and 2 were all selected to be representative of the design for a full-scale system downstream of a venturi scrubber. The CMM results showed that for Column 1,

Table 7. Mass Balance Closure

	CMM Inlet Average, mg	Laboratory Bed Analyses, mg	Bed Analyses/CMM Inlet Average
Column 1	13.3	15.2	114.3%
Column 2	13.3	16.2	121.8%
Total	26.6	31.4	118.1%

which contained an 18-inch depth of DARCO Hg-LH activated carbon, mercury was first detected (>0.2 µg/dscm) at the outlet of the 1-inch bed depth at 300 hours and reached a maximum of 3.9 µg/dscm by 833 hours into the run. At the 3-inch bed depth, no clear mercury breakthrough >0.2 µg/dscm was seen by the end of the 1000 hours. No mercury was detected at the 6-inch depth or any depth past 6 inches by the end of the run. For Column 2, which contained an 18-inch depth of MERSORB HT-1.5 SIC, mercury >0.2 µg/dscm was first detected at the outlet of the 1-inch bed depth at 300 hours and reached a maximum of 3.6 µg/dscm by 840 hours into the run. No mercury was detected by the end of the run at the 3-inch depth or any other depth past 3 inches.

The results from mercury analyses of the individual bed sections at the end of the run verified that the inlet mercury was captured by the first two beds. The mercury analyses of the beds in Column 1 indicated that all of the mercury was collected in the first 3 inches of the bed. Both the mercury analyses data after the run and the CMM data show that 6 inches of DARCO Hg-LH provided essentially 100% mercury capture (to less than the mercury quantification limit) over the entire run. For Column 2, the mercury analyses indicate that all of the mercury was collected in the first 3 inches of the bed. Both mercury analyses data after the run and the CMM data show that 6 inches of MERSORB HT-1.5 SIC also provided essentially 100% mercury capture (to less than the mercury quantification limit) over the run.

The mercury mass balance closure for Column 1 based on the inlet CMM data was 114%. For Column 2, the mercury mass balance was 122% from the CMM data. The overall mass balance closure for both columns was 118% based on the CMM data. Considering the challenges of accurate mercury measurement for gases and solids, the complexity and duration of the run, and the uncertainty of getting a representative sample from each bed section, these mass balance closures are within expectations. From these results, a primary conclusion is that the commercially available carbons investigated in these tests would be effective for high degrees of mercury abatement, i.e., to levels below the quantification limit, using only six (6) inches of bed depth for the conditions evaluated, which are representative of the expected full-scale operation.

7.2 Conclusions

The following major conclusions are drawn from the 1000-hour test of fixed-bed mercury adsorption performance:

- Based on results from the current test program at the EERC, fixed-bed adsorption using activated carbon as the adsorbent is an extremely effective method of removing mercury from flue gas at process conditions typical of those anticipated for taconite processing.

- Two commercial adsorbents demonstrated good mercury abatement performance in the test: DARCO Hg-LH 4×8 (Norit Americas) and MERSORB HT-1.5 (Nucon International).

- After 1000 hours of run time for both adsorbents, no measurable breakthrough of mercury occurred past the 3-inch bed depth. More run time would be required to reach mercury breakthrough at the 6-inch bed depth.

The data produced in the testing clearly demonstrate the high degree of mercury abatement possible with minimal amounts of commercially available activated carbons. A fixed bed of activated carbon used for mercury control could provide 100% mercury capture for extended periods of time with minimal pressure drop. It should be noted, however, that full-scale mercury abatement in a site fixed bed would not necessarily follow the exact capacity and capture efficiencies demonstrated during this test. Variations in temperature, velocity, mercury concentration as well as other flue gas constituents could lead to different quantitative performance (e.g., bed life and total capacity). In order to develop the most economical design for a fixed bed, further evaluation of the effects of actual operating conditions such as thermal cycling and gas velocity through the fixed bed will be needed.

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