



STABILITY OF MERCURY IN COAL COMBUSTION BY-PRODUCTS AND SORBENTS

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Project Description

This continuing project focused on three primary tasks with the goal of determining the mechanisms of mercury release from coal combustion by-products (CCBs). Each task was designed to address specific objectives.

The major focus this past year was in Task 1, where the research was directed toward understanding releases of mercury from microbial activity in CCBs under a disposal setting. Three ash samples were evaluated in triplicate under aerobic versus anaerobic and fed versus starved conditions. Elemental (Hg^0) and organomercury releases were evaluated.

Task 2 centered around working with the U.S. Environmental Protection Agency (EPA) to determine a standard leaching protocol for environmental characterization of CCBs for environmentally sensitive elements in addition to mercury.

Task 3 addressed thermal effects on mercury vapor release under the two subtasks of long-term ambient release and thermal desorption at elevated temperatures. The method for the determination of blank values for the long-term ambient release of mercury from six ash samples previously studied under CATM was improved. Thermal desorption curves were determined for a number of samples, and attempts were made to determine if speciation of mercury forms was possible.

Goal

The overall goal of the effort was to determine the amount of mercury released from CCBs under specific environmentally related conditions and to compile a database of information on mercury release from CCBs for use in regulating and managing disposal and beneficial reuse applications. Supporting objectives were to:

- Determine the amount and forms of mercury released from fly ash and other CCBs into air due to microbial action.
- Determine the leachability of mercury from conventional fly ash samples, ammoniated fly ash, high-carbon fly ash, and dry flue gas desulfurization (FGD) materials.

- Develop more reliable and reproducible data on mercury release through offgassing.
- Identify mercury compounds present on CCBs using thermal desorption.
- Obtain data for a wider range of CCBs.

Rationale

EPA announced on December 14, 2000, that it would regulate mercury emissions from coal- and oil-fired electric utility steam generating power plants [1]; propose regulations by December 15, 2003; and finalize regulations by December 15, 2004. On December 15, 2003, EPA proposed the Utility Mercury Reductions rule, which seeks comments on two approaches for reducing mercury by up to nearly 70%. As mercury emission control technologies are implemented, there is potential for CCB mercury levels to increase. The stability of mercury on CCBs, including primarily fly ash and FGD materials, under a variety of conditions presents a potential environmental problem. In developing an understanding of the potential for mercury release from CCBs, it is important to develop techniques to evaluate those potential releases.

Biological activity, leaching, and thermal offgassing are potential mechanisms for the release of mercury from CCBs into the environment. Initial tests were performed under CATM to determine the effect of biological activity on the release of mercury vapor from CCBs. It was found that a larger mass of mercury was released from CCBs with biological activity [2]. The apparatus was improved, and organomercury compound determinations were made in addition to Hg^0 .

Leaching of mercury from CCBs is always of concern in disposal and use scenarios. Discussions are under way with EPA, the U.S. Department of Energy National Energy Technology Laboratory (DOE NETL), the Energy & Environmental Research Center (EERC), and others to determine the most appropriate leaching method(s) for the evaluations of CCBs.

Two initial CATM tests on high-mercury CCBs showed releases of mercury at ambient temperature equivalent to 2.2×10^{-8} and 2.9×10^{-9} lb Hg/ton CCB/yr, which are very small masses of mercury. To put these into context, a coal-fired power plant with an annual production of 200,000 tons of ash per year would potentially release 0.0044 pounds (2.0 grams) or 0.0006 pounds (0.26 grams) of mercury, respectively, per year because of offgassing from ash [2–4]. An improved apparatus lowered the release of mercury. A method for determining blank values more efficiently was devised.

Early CATM tests on thermal desorption for a limited number of samples indicate that some mercury remains on the CCB at temperatures up to 600°C [5]. Temperature profiles for mercury release showed one to five peaks but could not be related to known mercury compounds. Thermal desorption of mercury and mercury compounds from CCBs, CCBs spiked with mercury compounds, and mercury compounds added to pulverized quartz powder were studied at temperatures from ambient to 700°C.

Approach

Effect of Biological Activity

Three CCBs were tested for the microbiologically mediated release of mercury and are listed in Table 1. One ash sample is from a full-scale demonstration of a mercury control technology.

Table 1. CCB Sample Description and Total Mercury Content

Sample	Coal/Ash Description	Additional Information	Hg, $\mu\text{g/g}$
99-188	PRB ^a subbituminous fly ash + FGD material	Neutralized	0.131
01-002	Eastern bituminous fly ash		0.234
03-060	Subbituminous fly ash	<i>Advanced Hybrid</i> TM Filter Technology	NT ^b

^a Powder River Basin.

^b Not tested.

The apparatus was constructed as shown in Figure 1. A 250-mL Erlenmeyer flask was fitted with an impinger inlet/outlet tube with the inlet center shortened to 6 cm below the standard taper. Cylinder gas was passed through several sets of gold-coated quartz traps for mercury removal and admitted to each of the flasks through a gas distribution manifold that routed the gas through 0.23-mm gas chromatography (GC) capillary tubing to each of the individual flasks. A GC capillary length of approximately 60 cm, when pressurized to between 1 and 2 psig through a gas distribution manifold, provided a convenient means of regulating gas flow to approximately 2 cm³/min. The gas passed mercury vapor from the headspace of the flasks to a mercury vapor collection system at the outlet of the flasks, consisting of two traps. The nearest trap contained Supelco CarbotrapTM, which collected organomercury compounds. This was followed by a gold-coated quartz trap, which collected Hg⁰.

The flasks were placed on a 16-flask wrist-action shaker. The experimental matrix consisted of eight flasks under anaerobic conditions (using argon) and eight flasks under aerobic conditions (using breathing-quality air). In each set of eight flasks, two contained only buffer, three contained the CCB with buffer (starved), and three contained the CCB with buffer and glucose (fed). A 50- or 80-gram aliquot of CCB was

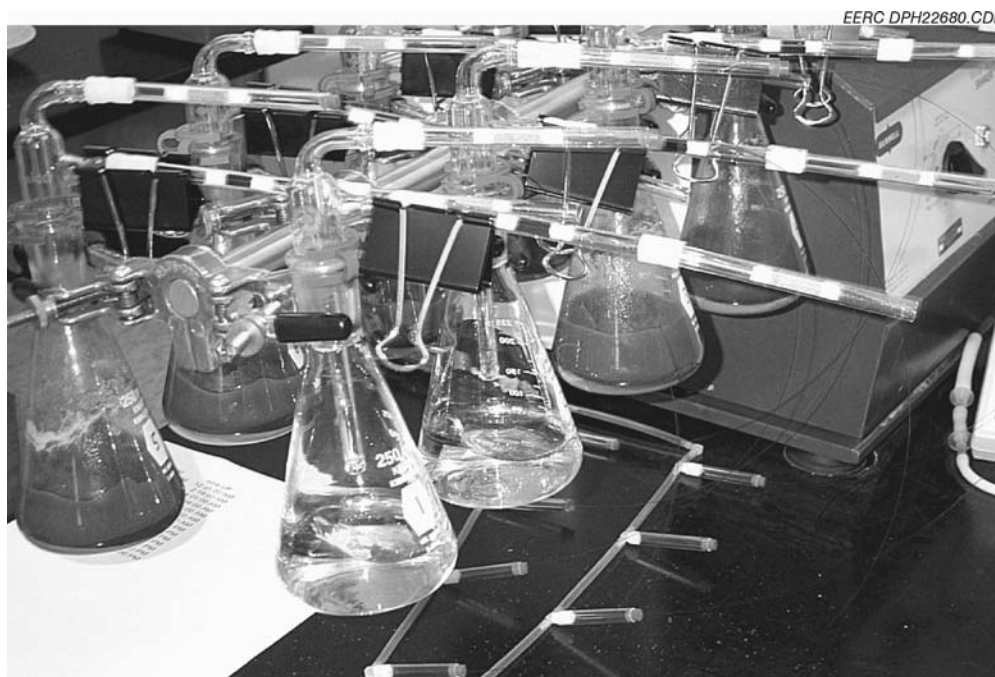


Figure 1. Microbiologically Mediated Mercury Vapor-Phase Collection Apparatus

placed in the flasks, and 94 or 100 mL of a phosphate buffer (with or without glucose as appropriate) was added to create a neutral pH. The CCB and buffer amounts varied because of the CCB density. The ash-containing flasks also had 100 μ L of mixed bacterial culture added. The source of bacteria was a mixed bacterial inoculum from a brackish wetland.

Mercury vapor was collected for approximately 30 days. The gold-coated quartz collection traps were desorbed for analysis by heating to approximately 500°C, and the mass of mercury released was determined using atomic fluorescence. The Carbotrap™ collection traps were analyzed for total mercury by heating the trap to approximately 300°C, passing the released organomercury through a tube at about 800°C, and collecting the mercury vapor on a gold-coated quartz trap, which was analyzed as described above.

Bacterial counts were performed upon completion of the 30-day period. A 1-mL aliquot of solution was taken from each flask. The aqueous supernate was serially diluted in 0.1% sodium pyrophosphate buffer (pH 7.0) and then used to inoculate a series of tubes containing 1% PTYG (peptone, tryptone, yeast extract, glucose) broth. The tubes were incubated at 30°C, and growth, as turbidity, was monitored over a 3-week period.

Improvements to the experimental setup are being made in the current CCB (03-060) investigation. An initial bacterial count is being performed to provide a baseline comparison value. The gold-coated quartz collection traps are being analyzed once a week in an attempt to see a trend of Hg⁰ release. The reusable traps are placed back on the system after analysis, and mercury is collected again.

Leaching

Leaching is the most likely mechanism of transport of constituents from disposed or utilized CCBs contacted by water. Leaching is typically performed on CCBs to characterize them for management purposes. Several issues have been raised by EPA's Office of Research and Development and Office of Solid Waste related to the best means of evaluating the leaching potential of CCBs. In fact, EPA held a meeting in early 2002 during which EPA representatives and others proposed an extensive series of leaching protocols which were suggested for CCB leaching characterization. EERC researchers reviewed the proposed methods and held discussions with scientists at DOE NETL. DOE NETL provided an alternate proposed method, and the EERC proposed that the synthetic groundwater leaching procedure (SGLP) with the long-term leaching option be used. Both the proposed DOE NETL and EERC methods included a long-term component, although the DOE NETL long-term component is a simulated long-term evaluation. The EERC agreed to participate in a testing program that will include the EERC SGLP with a long-term component, the DOE NETL leaching method, and the EPA-proposed leaching procedure.

Thermal Effects and Ambient Release of Mercury Vapor from Ash

Long-Term Ambient Release

Six ash samples were previously evaluated in duplicate for the release of mercury over 263- and 264-day periods in Tests 1 and 2, respectively, through CATM [2, 6]. However, blank values for the samples were an unresolved issue. In Test 1, separate empty bottles were used in an attempt to have a blank value to subtract from all sample results. An improved method was used in Test 2. Each sample bottle was emptied of ash, and gas was allowed to continue to flow, collecting mercury for an additional 180 days. These two 90-day periods were used to derive a separate blank for each sample container. The apparatus was similar to that described for the microbiologically mediated release of mercury and is described in detail elsewhere [7].

After mercury vapor was collected for the two 90-day time intervals, the tubes were desorbed by heating the analytical gold-coated quartz trap to approximately 500°C, and the mass of released mercury was determined using atomic fluorescence.

Thermal Desorption at Elevated Temperatures

A schematic for the controlled thermal desorption of mercury and mercury compounds was assembled and is shown schematically in Figure 2. The apparatus was constructed using an atomic absorption (AA) spectrophotometer for mercury detection and included a small tube furnace and temperature controller for thermal desorption. A Hewlett Packard 3395 integrator was used for data collection. Detection of thermally desorbed mercury and mercury compounds was done in an electrically heated quartz cell operated at 800°C. The use of a heated cell allowed detection of mercury compounds by thermally decomposing compounds to form Hg^0 , which can be detected by AA. Gas flow was 20 cm^3/min of nitrogen. The temperature controller was ramped from ambient temperature to 700°C at a rate of 25°C per minute. The AA was calibrated for the amount of mercury thermally desorbed from some of the CCBs tested. This was done by injecting a known amount of mercury, from air saturated with Hg^0 , onto a gold-coated quartz trap and thermally desorbing the mercury in the same manner as samples. A more complete description along with a description of the experimental protocol can be found elsewhere [8].

Thermal desorption curves were determined for various CCB samples (see Table 2). The ash samples included those from the combustion of lignite, subbituminous, and bituminous coals; pilot and full-scale samples; fly ash and fly ash–FGD mixtures; and with or without mercury control technologies in place. Some of the samples were spiked with mercuric oxide (HgO) or mercuric chloride (HgCl_2) powder. Experimental work was also done on determining the thermal curves for devolatilization of HgO and HgCl_2 compounds added to pulverized quartz powder to simulate an inert matrix.

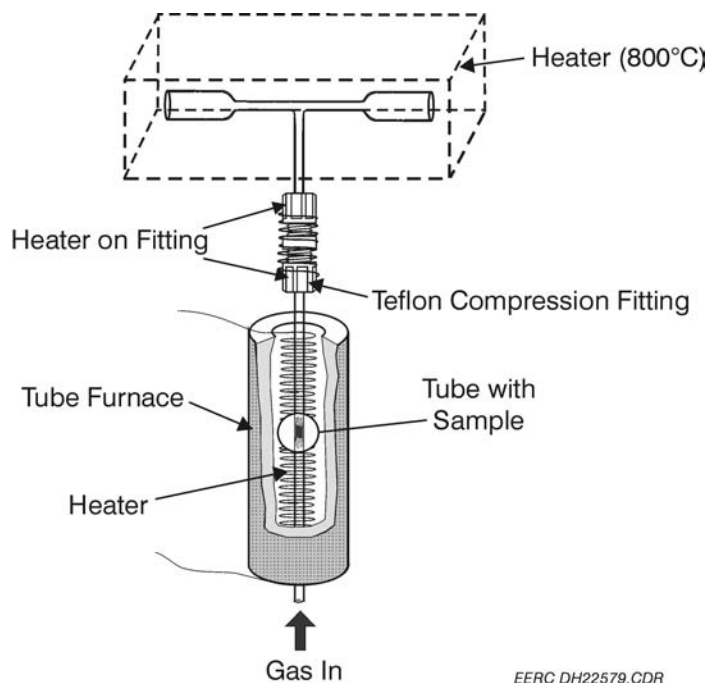


Figure 2. Mercury Thermal Desorption Apparatus

Table 2. CCB Sample Description and Mercury Compound Spikes

Sample	Coal/Ash Description	Spiked With
99-186	Subbituminous fly ash	HgO
99-456	Subbituminous fly ash	
00-002	Lignite fly ash	HgO, HgCl ₂
00-004	Lignite fly ash/FGD	
00-006	PRB subbituminous fly ash	
00-008	Lignite fly ash	HgO, HgCl ₂
00-054	PTC ^a ammoniated fly ash	
00-057	PTC ammoniated fly ash	
01-001	Subbituminous fly ash	HgO, HgCl ₂
01-004	Lignite fly ash	HgO, HgCl ₂
01-008	PRB subbituminous fly ash	
01-009	Lignite fly ash	HgO, HgCl ₂
01-011	Lignite fly ash/FGD	
02-056	Bituminous fly ash	
03-016	Lignite fly ash	HgO, HgCl ₂
03-017	Lignite fly ash/FGD	
03-018	Lignite fly ash/FGD	
03-019	Lignite fly ash/FGD	
03-060	Subbituminous fly ash with <i>Advanced Hybrid</i> TM filter technology	
03-063	Bituminous fly ash	

^a Particulate test combustor, EERC's pilot-scale furnace.

Progress

Effect of Biological Activity

The biological activity experiments were conducted in triplicate primarily because the mass of mercury collected on the gold-coated quartz and CarbotrapTM collection traps can only be tested one time. If the amount of mercury captured on the trap over the duration of the experiment was too high for the settings used when testing on the atomic fluorescence instrument, the value was incomplete or lost.

Results to date have been confusing; however, general trends have emerged. The mercury released from the CCB slurry was generally higher in the samples fed with glucose versus starved samples and in aerobic versus anaerobic conditions. The bacterial count has also generally followed that trend. The Hg⁰ vapor captured on the gold-coated quartz traps has been higher than that seen in the long-term ambient-temperature vapor-release experiments. The flasks containing buffer only have been treated as blanks.

The results from the experiment using CCB 99-188, a subbituminous fly ash neutralized prior to the experiment, are shown in Table 3. Atomic fluorescence instrument problems resulted in incomplete results for the Hg⁰ captured on some of the gold-coated quartz traps because of the unexpected high masses of mercury. The Hg⁰ release results appear to be higher from the anaerobic condition. No trend is apparent for the organomercury results. This derivation from the trend stated earlier may be because the order of the gold-coated quartz and CarbotrapTM collection traps was incorrect. The gold-coated quartz traps were placed

Table 3. Results of Microbiologically Released Mercury from CCB 99-188

Anaerobic	Gold Trap Hg, ng	Gold Trap Hg, ng/g	Carbotrap™ Hg, ng	Carbotrap™ Hg, ng/g	Bacterial Count/mL
Blank	3.92		0.3		930
Blank	3.98		0.19		ND ^a
Fed	63.1	1.26	0.72	0.014	>24,000,000
Fed	45.6 ^b	0.91 ^b	2.32	0.046	>24,000,000
Fed	69.2	1.38	4.14 ^c	0.083 ^c	>24,000,000
Starved	NA ^d	NA ^d	1.83	0.037	240,000
Starved	47.7 ^b	0.95 ^b	5.62	0.112	93,000
Starved	66.3	1.33	2.07	0.041	43,000
Aerobic					
Blank	2.37		0.08		ND
Blank	1.65		0.06		ND
Fed	31.5	0.63	1.06	0.021	>24,000,000
Fed	61.3	1.23	0.86	0.017	>24,000,000
Fed	33.2	0.66	1.93	0.039	>24,000,000
Starved	45	0.9	0.66	0.013	240,000
Starved	42.2	0.84	0.5	0.01	240,000
Starved	44.9	0.9	0.89	0.018	93,000

^a Not detected.

^b Value is believed to be low because of testing conditions.

^c Value may be high because of testing conditions.

^d Value was not obtained because mass of mercury was too high for testing conditions.

nearest the flask outlet during the experimentation; therefore, it is believed that some of the organomercury was trapped on the gold-coated quartz traps before the gas passed to the Carbotrap™ collection traps. The bacterial count shows more bacteria in the flasks in which the bacteria were fed glucose, in both the anaerobic and aerobic conditions.

The results from the experiment using CCB 01-002, an eastern bituminous fly ash, are shown in Table 4. The blank flasks, containing only buffer, yielded higher mercury release values than those containing the CCB slurry. The mercury release results show that more Hg⁰ was released from flasks in which the bacteria were fed with glucose versus starved. These values are much lower than those from CCB 99-188, pg/g versus ng/g. The bacterial count results show that the bacteria were able to survive in the aerobic environment and that within the aerobic environment, the bacterial count was higher at the end of the experiment in the flasks where the bacteria were fed with glucose.

Initial results from CCB 03-060, a subbituminous fly ash resulting from the full-scale use of *Advanced Hybrid™* filter technology, are shown in Table 5. These initial results show that Hg⁰ is being released from the CCB-containing flasks in the aerobic environment in which the bacteria are fed with glucose. The mass of mercury released from the blank flasks in the aerobic environment was much lower than in the anaerobic environment.

The high blank values from the buffer solutions still need to be resolved.

Table 4. Results of Microbiologically Released Mercury from CCB 01-002

Anaerobic	Gold Trap Hg, pg	Gold Trap Hg, pg/g	Carbotrap™ Hg, pg	Carbotrap™ Hg, pg/g	Bacterial Count/mL
Blank	1280		95.47		NT ^a
Blank	2303		203		NT
Fed	155	3.1	13.45	0.27	ND ^b
Fed	177.3	3.55	14.12	0.28	ND
Fed	376.9	7.54	12.66	0.25	ND
Starved	66.49	1.33	13.16	0.26	ND
Starved	45 ^c	0.9	22.29	0.45	ND
Starved	56.28	1.13	19.9	0.4	ND
Aerobic					
Blank	1096		36.16		NT
Blank	1160		44.52		NT
Fed	405.71	8.11	13.76	0.28	>24,000,000
Fed	1758	35.2	52 ^c	1.04	>24,000,000
Fed	136.1	2.72	13.16	0.26	>24,000,000
Starved	48 ^c	0.96	5.876	0.12	43000
Starved	79.39	1.59	13.59	0.27	24000
Starved	30.48	0.61	4.739	0.1	150000

^a Not tested.

^b Not detected.

^c Lower sensitivity.

Leaching

The EERC agreed to participate in a testing program that will include the EERC SGLP with a long-term component, the DOE NETL leaching method, and the EPA-proposed leaching procedure. This effort is under development with DOE NETL. At this time, EPA has not agreed to participate.

EERC researchers presented their thoughts on the requirements of an appropriate leaching characterization evaluation for CCBs in a paper titled “A Leaching of CCBs: Observations from over 25 Years of Research” at the 2003 International Ash Utilization Symposium held in October 2003 in Lexington, Kentucky [9]. The paper was presented with the intent to elevate awareness of the issues related to selection of leaching procedures.

Thermal Effects and Ambient Release of Mercury Vapor from Ash

Long-Term Ambient Release

The measured mercury released from blank bottles was higher than from the sample bottles in Test 1 and confirmation of this was desired before it was suggested that the ash samples appeared to be sorbing mercury rather than emitting mercury. Higher blank values were also seen in the method used for Test 2. If one calculates and compares the emission rate in pg/day for the blank of each sample container and for the containers with ash in them from Test 2, it becomes apparent that the ash samples appear to be sorbing mercury. These values are presented in Table 6 as averages of the duplicate samples.

Table 5. Initial Gold-Coated Quartz Collection Trap Results of Microbiologically Released Mercury from CCB 03-060 after 28 Days

Anaerobic	Hg, pg	Hg, pg/g
Blank	581.4	
Blank	257	
Fed	143.7	1.8
Fed	206.4	2.58
Fed	48.98	0.612
Starved	55.74	0.697
Starved	41.99	0.525
Starved	117.2	1.47
Aerobic		
Blank	102.8	
Blank	69.39	
Fed	3262	40.8
Fed	1557	19.5
Fed	3132	13.2
Starved	50.86	0.636
Starved	43.22	0.54
Starved	65.07	0.813

Table 6. Comparison of Emission Rates Between the Empty Bottles and the Bottles Containing Ash in Test 2, pg/day

Sample	Bottles with Ash	Bottles Without Ash	Difference
99-188	2.237	2.161	0.076
99-189	0.077	1.127	-1.050
99-692	0.081	2.454	-2.373
99-693	0.077	4.328	-4.251
99-722	0.696	7.165	-6.469
99-724	0.411	4.436	-4.025

Long-term ambient temperature desorption experiments have indicated that five of the six CCBs analyzed acted as mercury sinks, although these samples were previously reported as having released small amounts of mercury vapor. The previously reported value of a maximum of 0.26 grams of mercury release from 200,000 tons of ash [2, 4] actually becomes a negative number in light of these new data. This does not necessarily mean that no mercury was being released from the CCBs, but that in our experiments, eleven of the containers appeared to release more mercury after emptied of ash than while containing ash. The total mercury content of these ashes has not correlated to the apparent amount of mercury released or sorbed.

Thermal Desorption at Elevated Temperatures

A large variety of CCBs have been analyzed for the thermal release of mercury. Most of the thermal curves generated were straightforward, containing only one or two major desorption peaks. Examples are shown in Figures 3 and 4. However, background noise or other interferences were encountered occasionally. For example, high-carbon ash samples seemed to generate more background noise, which the deuterium background correction lamp did not mitigate. High-sulfur CCBs also produce an irregular baseline and unidentifiable peaks; however, the use of deuterium lamp background correction reduced the interfering background.

Mercury compounds were also analyzed. HgO and HgCl₂ were added directly to pulverized quartz powder to simulate an inert matrix. Upon thermal desorption, HgO produced a sharp, symmetric peak that desorbed at 250°–325°C. The peak from HgCl₂ desorption was less symmetric and desorbed at temperatures of 200°–250°C.

Upon spiking ash samples with HgO and HgCl₂, it was found that the complex chemistry and/or matrix of the ash sample altered the decomposition of the mercury compounds. All spiked samples had peak desorption temperatures higher than that of the spike component alone. In all but Sample 99-186, the CCBs

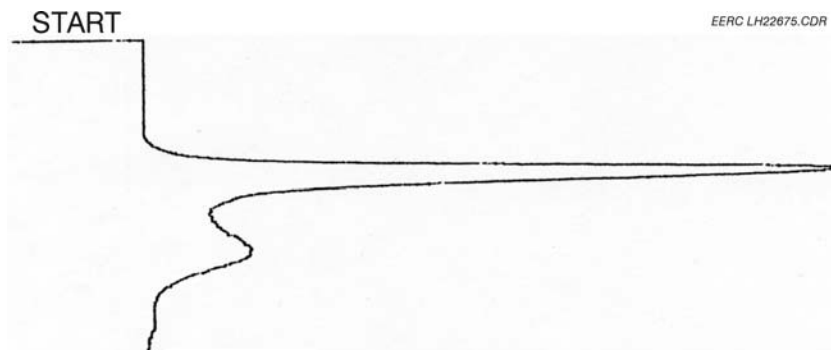


Figure 3. Thermal Desorption Curve from a CCB Yielding Two Major Mercury Peaks

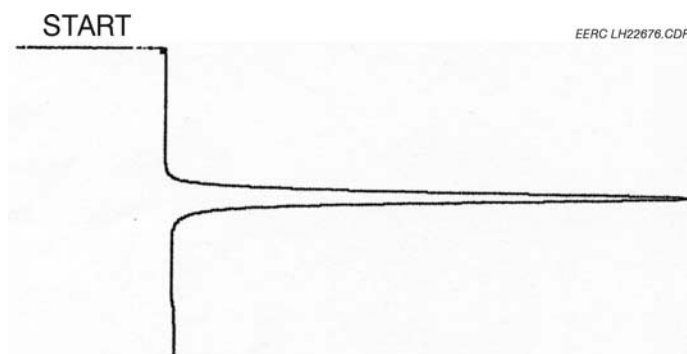


Figure 4. Thermal Desorption Curve from a CCB Yielding One Major Mercury Peak

spiked with HgCl₂ eluted peaks at temperatures lower than the plain CCB. Spiking samples with HgO yielded variable decomposition temperatures. Thermal desorption curves were rather difficult to interpret since there is no way, at present, using this apparatus to determine exactly what is happening during the thermal treatment. There are several possible scenarios:

- Mercury and mercury compounds, as sorbed, are being released unchanged during the thermal desorption procedure.
- Mercury compounds are being desorbed by a mechanism of thermal decomposition whereby sorbed compounds such as HgO are thermally decomposed to mercury and oxygen during the thermal desorption.
- Mercury or mercury compounds are chemically reacting with the CCB components then thermally desorbed according to the first or second scenario as described above.

Unfortunately, this delayed desorption was not completely reproducible making “peak matching” with desorption curves of the mercury compounds without ash difficult and problematic.

Calibration of the AA with Hg⁰ sorbed onto gold-coated quartz produced a peak that was very symmetric and well-defined, decomposing at an average temperature of 350°–375°C. Calibration allows one to quantify the amount of mercury desorbed from the CCBs. However, concentration results from the CCBs were not reproducible.

Status

The experimental portion of this project has been completed with exception of the last microbiologically mediated mercury vapor-release experiment. Additional activities to conclude this project include preparation of the final report.

Quality Assurance/Quality Control

Quality Objectives

The overall quality objective was to develop and conduct experiments such that the resulting data were repeatable and would allow an estimation of the mercury desorption of CCBs.

Measurement/Data Acquisition

Because this was a laboratory project to evaluate mercury stability in CCBs, most of the analyses of the samples were done using standard EPA-approved laboratory methods. Other laboratory techniques that did not have specific EPA-approved methods were performed in accordance with standard EERC laboratory practice. The Analytical Research Laboratory, where most of the work was done, is EPA-approved through the state of North Dakota. As part of the test plan, both replicate and blank experiments were performed to ensure the quality of the results.

Assessment and Validation

The standard analysis techniques used in the project indicate acceptable performance criteria. The repeatability of the data were within the expected $\pm 20\%$ with the exception of the data produced in the microbiological experiments. In this case, the experiments are still a work in progress, with much improvement yet needed on the technique. This is nearing completion. Despite more variability than had been hoped for, the data still proved useful.

The thermal desorption curves of HgO and HgCl₂ released peaks at temperatures repeatable to within 10%. Again this is a technique in development, and further improvements are expected.

Potential Users/Technology Transfer

The environmental fate and transport of mercury from CCBs is of great concern to coal-fired utilities, regulators, CCB vendors and users, and environmental groups, especially with the EPA announcement of upcoming mercury regulations for coal-fired utilities. For this reason, this project is highly relevant and has relatively high visibility. Because of the scientific outcome, this project is extremely important. To date, the results appear to be promising, with the releases of mercury at very low levels or the CCBs acting as mercury sinks. The information from this and other similar studies will be of great importance to users of products containing CCBs such as FGD wallboard and fly ash-containing concrete as well as regulatory agencies, municipalities, landfill owners and operators, and mercury sorbent vendors and users.

This project resulted in an award by DOE NETL in this same subject area looking at CCBs resulting from the use of mercury control technologies. That project will benefit from the method development and results obtained over the past year.

The results of these tasks were reported to various audiences [7, 10, 11].

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