



DEVELOPMENT OF MERCURY CONTROL TECHNOLOGIES

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Project Description

This project is intended to test promising sorbents and catalysts that have high reactivities to provide rapid in-duct mercury capture or oxidation when injected upstream of a particulate control device. In-duct capture sorbents would provide a useful method for both electrostatic precipitators (ESPs) and baghouses, while the ability to oxidize mercury would make scrubbers more effective in removing mercury. Success with these approaches will lead to cost-effective mercury control options that could be applied to most coal-fired applications.

New sorbent work included the evaluation of reactive Fe^{4+} compounds and electrical properties of ash that have demonstrated sorption properties. Catalysts were identified and tested for conversion of mercury to the oxidized form to facilitate capture by scrubbers. An entrained-flow sorbent screening system was developed to test sorbents and catalysts on flue gases from larger combustion systems.

Goal

The overall goal of the work is to develop cost-effective mercury control in an approach consistent with the research needs itemized in the U.S. Environmental Protection Agency *Mercury Study Report to Congress*.

Specific goals were:

- Further develop protocols to test the required sorbent reactivity and capacity necessary for effective mercury capture in ESPs.
 - Test new sorbents (based on previous successes at the EERC and elsewhere) that are likely candidates for rapid in-duct sorption of mercury and can overcome the SO_2 - NO_2 -induced breakthrough problem.
 - Further refine the understanding of flue gas concentrations on sorbent reactivity, capacity, and desorption.
 - Investigate the effect of carbon basicity on the activated carbon (AC) sorption mechanism.
 - Evaluate catalysts or additives that can be utilized in flue gas desulfurization (FGD) systems to consistently achieve 90% Hg capture.
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- Determine the feasibility of bioleaching as a viable method for mitigating the emission of vapor-phase mercury from coal combustion.

Rationale

Mercury emissions from combustion systems have become recognized as a significant source of anthropogenic mercury. Several approaches are suggested for mercury control from coal-fired boilers, including enhancing the ability of wet scrubbers to capture mercury. However, many coal-fired boilers are not equipped with wet scrubbers. Since almost all coal-fired boilers are equipped with either an ESP or a baghouse, sorbent injection upstream of either an ESP or a baghouse appears attractive, because it has the potential to control both elemental and oxidized mercury species, would appear to be easy to retrofit, and would be applicable to both industrial and utility boilers.

Since mercury in the gas stream from coal combustion is present in only trace quantities, usually in the range of 5 to 10 $\mu\text{g}/\text{m}^3$ (about 1 ppbv), only very small amounts of sorbent may be necessary. Assuming a mercury concentration of 10 $\mu\text{g}/\text{m}^3$ and a sorbent-to-mercury mass ratio of 1000:1, the required sorbent loading is 10 mg/m^3 , which is only 0.1% to 0.2% of a typical dust loading of 5–10 g/m^3 (2.2–4.4 grains/scf). This amount of sorbent material in the ash would appear to be negligible and would not be expected to have an impact on control device performance or ash utilization. The primary requirement is that the mercury be tightly bound in the sorbent, not desorbing upon exposure to ambient air or leaching under wet disposal conditions.

The rate of sorption may depend on a number of factors such as the diffusion within the particle, the rate of any heterogeneous reactions that may occur, and the ultimate capacity of the sorbent. Assuming the sorbent has the capacity and reactivity to trap the mercury that reaches the sorbent particles, the final critical step is the long-term stability of the sorbed mercury.

Previous testing established that ACs, such as fine powdered Norit FGD AC, are effective for mercury capture in full flue gas component mixtures, but mercury breaks through most sorbents in about 1 hour, mainly due to a NO_2 - SO_2 -water interaction that limits the capacity and results in release of oxidized mercury species [1]. The current model for explaining the capture of Hg(II) formed in the initial oxidation step describes the sorption of the Hg(II) at a basic site on the carbon to form a nonvolatile salt [2, 3]. This occurs until the basic sites are exhausted (by reaction with SO_2 , sulfate, or HCl). At this time, the Hg(II), which continues to form, reacts with nitrate (from NO_2) or with HCl to form volatile salts such as the nitrate hydrate or chloride, and breakthrough appears. In addition, the release of oxidized mercury from the basic binding sites on the sorbent occurs, resulting from the conversion of these nonvolatile basic salts, in which the Hg(II) is initially captured, into volatile Hg(II) salts.

FGD systems have shown promise for capture of mercury. Early studies reported by EPRI show mercury capture as high as 80%, but also report relatively low capture efficiencies. Further research demonstrates that oxidized mercury species are quite effectively removed during FGD, while essentially all elemental mercury passes through the wet scrubber. Efforts of several researchers have focused on oxidizing elemental mercury either upstream to or in the scrubber as a method to enhance mercury removal. Researchers at McDermott have reported an FGD reagent that removes 86% of the total mercury including nearly all of the oxidized mercury and some of the elemental mercury [4]. Mendelsohn and Livengood at Argonne National Laboratory have reported a 40% reduction in elemental mercury utilizing chloric acid in a laboratory-scale FGD simulator [5].

An alternate strategy to end-of-pipe emission abatement is to remove the pollutant prior to combustion. Extensive work on these “coal-cleaning” technologies has been previously completed for a number of

pollutants by previous researchers [6–8]. Unfortunately, this strategy has not typically been found to be attractive compared to end-of-pipe technologies. However, vapor-phase mercury removal has proven to be a challenging problem. For this reason, pretreatment may be a feasible alternative for small and mid-sized pulverized combustors and gasifiers.

A growing field for the removal of target compounds from solid materials is bioleaching. In many cases, bioleaching has been found to represent the best practical technology for applications involving the removal of trace amounts of toxic metals from wastes, including coal mine tailing piles [9–11]. Previous studies have shown that a number of different biological resources (e.g., bacteria, fungi, plants, dead biomass, algae) have shown promise for the uptake of mercury [12–13].

Approach

The approach to determine reactivity and capacity of promising sorbents was to perform fixed-bed tests designed to measure the maximum reactivity under realistic simulated flue gas conditions.

Since previous work has shown that very high sorbent reactivity and capacity can be achieved under some conditions (e.g., HCl present without SO₂ and NO_x), there is evidence that a sorbent can be designed to provide effective mercury capture when injected into a duct upstream of an ESP. To further elucidate sorption mechanisms, the work included select tests to better understand the relative effects of SO₂, NO, NO₂, HCl, CO, Cl₂, and H₂O on sorbent performance.

A series of sorbents was prepared consisting of basic complexing agents impregnated on a reactive carbon support. These offer the potential to increase sorbent capacity owing to their high affinity for mercury(II) ions formed at the oxidation sites on the carbon surface. The stability of the ligands in the flue gas is not known. The ligands utilized for impregnation of the Norit FGD carbon included dithiophosphinic acid, sulfamic acid, thiosulfate, sulfanilamide, thiocyanate, thiosalicylic acid, thiocarbanilide, dimethylhydantoin, anthranilic acid, bisanil, bis(salicylidene)ethylenediamine, and terasulfide. These were compared with hard bases magnesium oxide and sodium hydroxide.

By using a sorbent containing the highly reactive Fe(IV)(DTC)₃ complex, the rapid oxidation of adsorbed Hg⁰ by the direct transfer of electrons from the complex is expected. This direct oxidation would not necessitate the concomitant reduction of NO₂ to nitrite that eventually forms nitrate. The reduction of the iron from Fe(IV) to Fe(II) destabilizes the iron dithiocarbamate complex allowing DTC ligands to transfer to Hg(II). Owing to the character of the sulfur ligand, DTC complexes of heavy metals are extremely stable. Formation of Hg(II)(DTC)₂ on the sorbent in this binding step could prevent the Hg(II) from reacting with nitrate or chloride and thus retard the desorption of mercury as the volatile nitrate or chloride. The stability of the Hg(II)(DTC)₂ to air containing NO₂ is not known, however, and oxidation of the DTC ligands could result in their loss and thus eventual breakthrough of volatile Hg(II) compounds.

The variability in the components of coal fly ash makes it difficult to predict its more subtle properties such as adsorptive properties, redox potential, free radical content, conductivity, resistivity, and polarizability on the basis of chemical analysis. The effect of contact of the ash with metal vapor or aerosol is currently not a standard prediction that can be made on the basis of commonly measured ash characteristics. Since adsorption and oxidation of metals such as Hg must be dependent on electrical charge characteristics of the ash and metal, a correlation between electrical properties of the ash and the metal should define effective ash–metal couple formation.

Progress

Entrained-Flow Reactor

An entrained-flow reactor was fabricated to enable sorbent evaluations to include in-flight performance and to allow evaluation of mercury transformations as a function of residence time and temperature. Figure 1 provides a schematic of the lower portion of the reactor. The original plan was to build a portable fixed-bed system, but after further review, the project team concluded that an entrained-flow system would be more useful for determining residence time effects. The reactor was fabricated from stainless steel and is 5 feet in height, including the concentric reducer at the outlet. Ten ports allow sampling at multiple residence times for a given gas flow rate. Current plans are to test at residence times as high as 10 seconds. The reaction vessel has been completed and will initially be utilized in Project 1 for mercury transformation evaluations. Following these experiments, modifications to the design will be made as needed and a single-wire ESP will be added to the system to evaluate entrained-flow sorbent performance. The goal of the reactor is to provide a way to test sorbent reactivity and effectiveness by controlling the in-duct contact time, while minimizing sampling effects. The ESP will remove the sorbent and fly ash particles without exposing the gas to a fixed bed.

Sorbent Development

Thin-bed test results with sorbents impregnated with dithiophosphinic acid, sulfamic acid, thiosulfate, sulfanilamide, and thiosalicylic acid showed breakthrough times in full flue gas composition that were somewhat shorter than that of the unimpregnated FGD carbon. Tetrasulfide, thiocabanilide, and thiocyanate showed basically no effect. Thus the negative effect in adding the ligands must be explained by the acidic character of the thio compounds in poisoning the basic sites on the carbon sorbent responsible for initial Hg(II) binding.

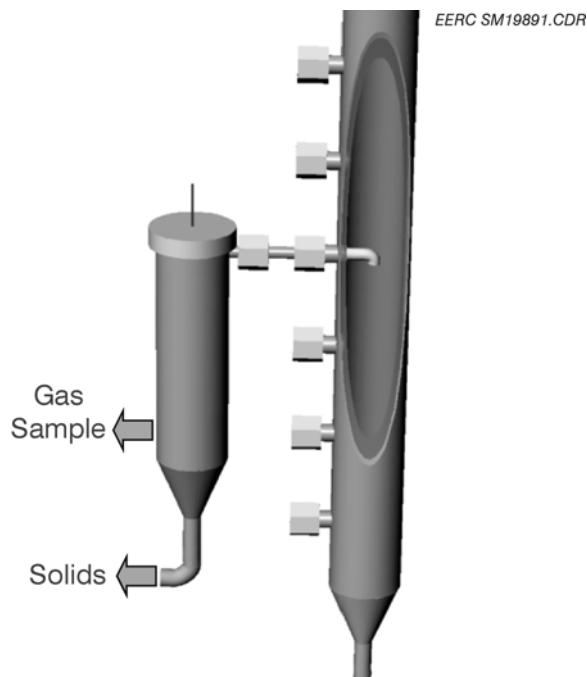


Figure 1. Entrained-Flow Reactor Schematic

The breakthrough curves for these ligands showed increases to 90% (dithiophosphinic and thiosulfate) to 100% (thiosalicylate and sulfamic) of inlet Hg^0 concentration after breakthrough, compared with 120% emission of the unimpregnated carbon. Thus the ligands may be binding and stabilizing the mercury absorbed prior to breakthrough.

The mercury emitted after breakthrough is essentially 100% oxidized, consistent with the effluent mercury from unimpregnated carbon. Thus the ligands have no effect on the oxidizing site on the carbon.

The 5% iron(III)(DTC)₃-impregnated sorbent was evaluated in the thin bed with full flue gas composition. The breakthrough curve (Figure 2) for the iron(III)(DTC)₃ sorbent showed typical steep breakthrough beginning at 1.3 hr and rising to 110% of inlet Hg^0 (then dropping to 100%), giving 100% oxidized Hg in the effluent after breakthrough. Thus the impregnation of the iron(III)(DTC)₃ had no effect on the mercury capture.

The 5% iron(IV)(DTC)₃-impregnated sorbent showed much earlier (0.6 hr) breakthrough to 100% compared with the FGD and Fe(III)(DTC)₃ sorbent. It is clear that the sorption capacity of this carbon was impaired rather than enhanced.

An explanation for the iron(IV) results was developed based on the Lewis acidity of the ligand and its poisoning of the basic sites on the carbon which prevents the binding of Hg(II) formed at these sites.

Catalyst Testing

Three materials were evaluated to determine their effect on mercury oxidation in a real flue gas stream at the University of North Dakota (UND) steam plant. Gold and a vanadium/titanium catalyst were evaluated to determine the amount of oxidation of elemental mercury that could be achieved. A Ryton material was

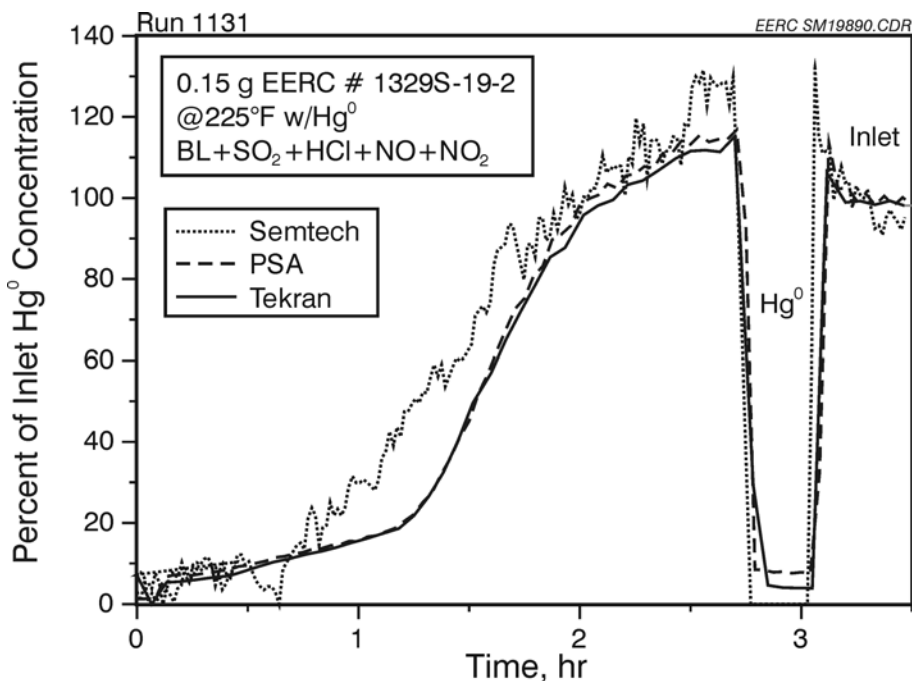


Figure 2. Breakthrough Curve Iron(III)(DTC)₃ Sorbent

evaluated to determine its ability to either reduce oxidized mercury to elemental mercury or possibly oxidize elemental mercury.

The steam plant burns a Powder River Basin subbituminous coal. An ECOM portable gas analyzer was used to measure the flue gas concentrations of O₂, CO₂, CO, NO, and SO₂ (Table 1). Flue gas was extracted from the exit of Baghouse 7, where the flue gas temperature was nominally 325°F. A quartz probe was used to extract the flue gas sample, and flue gas was transferred through a heated line to a heated oven which was housed in a sampling trailer. Figure 3 is a schematic of the sample oven and the elemental mercury spiking system. The catalyst samples were maintained at 300°F in the oven. Initial wet chemistry sampling at the baghouse outlet indicated a mercury concentration lower than desired. Therefore, an elemental mercury spiking system was constructed. The source of mercury was a permeation tube placed in a quartz filter holder and maintained at 194°F and constantly purged with 1.0 scfh of air. Based on results from a Semtech mercury analyzer, the temperature was adjusted to achieve an added mercury concentration of roughly 30 µg/m³.

Table 1. Steam Plant Flue Gas Concentrations

Flue Gas Component	36935	36936	36942	36956	36969
O ₂	11.7	10.5	10.9	14.4	14.2
CO ₂	8.4	9.5	9	3.8	6.1
CO	80	100	75	65	330
NO	215	190	228	77	156
SO ₂	240	184	279	120	220

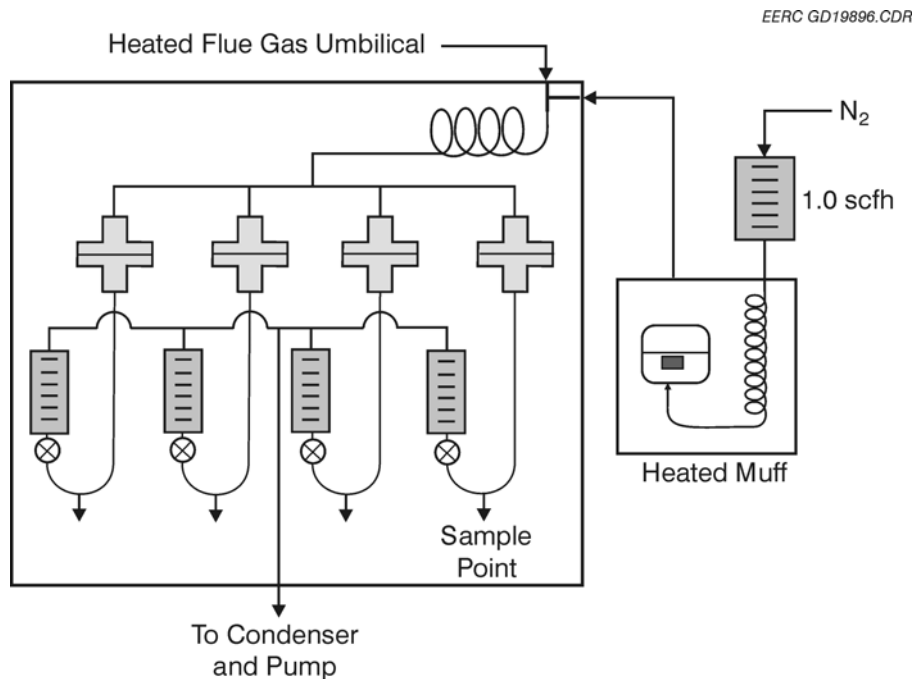


Figure 3. Schematic of the Sample Oven and the Elemental Mercury-Spiking System

Four quartz filter holders were used to hold the catalyst samples in the oven. A manifold connected the inlets of the filter holders to the heated inlet line. The outlets were also connected to a manifold which was equipped with flowmeters and control valves to ensure equal and constant flow through all of the samples. A sample pump was used to pull flue gas through the catalyst samples. A condenser, used to remove moisture from the flue gas, was placed upstream of the sample pump. The total flow through the filters was maintained at 12 scfh (3.0 scfh per filter). Ontario Hydro (OH) samples were taken at the filter holder outlets (upstream of the valves and flowmeters) to determine the outlet concentrations and speciation of mercury.

A blank quartz filter was placed in one filter holder to ensure no oxidation or reduction of mercury was taking place in the flue gas or across the filter holders. To evaluate gold as a catalyst, a quartz filter was coated using a sputtering technique. The vanadium/titanium catalyst was coated on a woven-glass filter material using a sol-gel process. The Ryton material was cut from a full-sized felted pulse-jet filter bag.

The OH results are presented in Table 2. Flue gas and elemental mercury flow through the catalyst samples were initiated late on February 13. The first set of OH samples was taken the morning of February 14. The results from the tests with the blank filter show there was no increase in oxidation across it, which indicates any ash bypassing the baghouse and collecting on the samples was not causing any additional oxidation of elemental mercury. The total mercury measured at the outlet of the blank filter varied from 28 to 69 $\mu\text{g}/\text{m}^3$. The 69 $\mu\text{g}/\text{m}^3$ may have been the result of the mercury permeation source not yet reaching steady state conditions. The blank results show that 80% to 92% of the total mercury at the inlet of the samples was elemental mercury. Some variability of the background mercury from the boiler as well as some minimal oxidation across the sampling system accounts for the fraction of oxidized mercury.

Results from the first test with the gold sample showed minimal oxidation. This suggests the gold was still capturing mercury and had not reached its saturation point. Six days later, 98% of the total mercury in the sample was oxidized. For the third test, 99% of the mercury in the sample was oxidized, and by the fourth test, nearly 100% of the mercury in the sample was oxidized. These results suggest that for the given flue gas environment, a small amount of gold on an appropriate contactor can effectively oxidize elemental mercury.

The significantly lower total mercury values for the last three vanadium/titanium catalyst tests compared to the blanks suggest that either the sample captured the mercury or it was oxidized to a form that is not detected by the OH sample method. Of the mercury measured by the OH method, 72% was oxidized by the fourth test. This shows some potential for the vanadium/titanium catalyst to oxidize elemental mercury even though it does not appear to work as well as gold.

Results from the initial test with the Ryton sample showed 86% oxidation of the elemental mercury; however, since the total was low, the sample may have been capturing a significant amount of mercury. The results from the remaining tests with the Ryton fabric indicate that the Ryton fabric has a very limited ability to oxidize mercury, at least for the given test conditions.

Bioleaching Pretreatment

A pot-scale study was conducted using two well-characterized coals, Blacksville and ND lignite. Coal samples were leached for 45 days in a mineral solution and bacterial suspension. Coal samples were extracted periodically to monitor the progress of the bacteria. The samples were washed with either water or hot methanol, and all samples were analyzed for their mercury, sulfur, and moisture content.

The bacteria chosen for this study was *Thiobacillus ferrooxidans*. This bacterium operates on iron compounds and will break down the pyrite grains in the coal, thus releasing the mercury sulfide and other sulfur compounds contained in the pyrite. Once released, the mercury sulfide and sulfur were flushed from the coal with the water or hot methanol solvents.

Table 2. Mercury Concentrations

Catalyst	Hg ⁺⁺ in KCl solution, µg/m ³	Hg ⁺⁺ in H ₂ O ₂ solution, µg/m ³	Hg ⁰ in KMnO ₄ solution, µg/m ³	Total Hg, µg/m ³	Oxidation, %
36935					
Blank	7.36	0.49	60.89	68.75	11.42
Gold	1.08	0.09	2.89	4.06	28.89
VTi	4.75	0.11	23.21	28.07	17.32
Ryton	8.12	0.07	1.32	9.5	86.16
36941					
Blank	5.17	0.58	22.04	27.79	20.67
Gold	21.61	0.05	0.51	22.17	97.71
VTi	7.18	0.01	3.62	10.72	66.19
Ryton	1.15	0.04	12.15	13.34	8.95
36956					
Blank	2.65	0.24	34.14	37.03	7.79
Gold	32.5	0.04	0.34	32.89	98.95
VTi	12.02	0.11	9.1	21.23	57.14
Ryton	1.27	0.15	22.09	23.51	6.06
36969					
Blank	3.91	2.58	38.47	44.96	14.43
Gold	89.27	0.03	0.27	89.57	99.7
VTi	18.32	0.32	7.27	25.9	71.94
Ryton	2.69	0.53	23.99	27.22	11.84

The current results show little or no mercury or sulfur removal using *Thiobacillus ferrooxidans*. This is most likely because the bacteria was unable to substantially break down and oxidize the pyrite grains within the coal matrix.

Electrical Properties of Ash

The Hg-fly ash samples to be tested were obtained from commercial coal-burning plants and were thoroughly analyzed for elemental content. The samples were chosen to have chemistries as similar as possible except for significantly different levels of Hg. Ash resistivity was determined on three chemically similar ash samples collected from flue gas and having different concentrations of Hg. The same samples were submitted for ESR (electron spin resonance) analysis.

ESR analysis of the ash samples resulted in excessively large, broad signals which could not be interpreted to give useful data. A second approach to acquire the ESR data is being attempted. This method involves an indirect measurement of ash radicals by reacting an organic reagent with radicals, quenching the radicals in the ash while leaving an organic radical that gives an interpretable signal, which is useful in determining radical content of the ash.

Resistivity was determined in duplicate as a function of temperature. The resistivity increased with temperature to a resistivity of 10^{11} ohm-cm for ash containing $< 0.002 \mu\text{g Hg/g}$. The maximum resistivity was measured at 138°C . Ash containing $1.25 \mu\text{g Hg/g}$ increased to 10^{12} ohm-cm over the same temperature range, reaching its maximum at 149°C . The curves intersect at 180°C .

Status

Entrained-Flow Reactor Testing

Results from the entrained-flow reactor system will be used to build on previous sorbent studies. Testing will be performed with the entrained-flow system at the bench scale on sorbents that have shown promise in the fixed-bed system, and results between the two sorbent evaluation methods will be compared. A variety of sorbents will be investigated as well as the effect of sorbent size and the impact of process conditions, including temperature, residence time, and simulated flue gas composition. Results will provide comparative reactivity measurements for various sorbents and evaluation of the impacts of process variables.

Testing on the entrained-flow reactor system will continue using actual flue gas from coal combustion facilities internal to the EERC. These tests will demonstrate the commercial potential of the most promising sorbents and provide added confidence in the readiness of those sorbents. Also as part of this effort, the sampling protocol will be evaluated for use in future entrained-flow reactor field tests on slipstreams at utility plants.

Sorbent Development

The sorbents with 1% loading have not yet been tested. No difference in activity is expected for the 1% iron(III)(DTC)₃, since the 5% showed no change. However, the 1% iron(IV)(DTC)₃ may show some intermediate activity, since fewer of the binding sites will have been poisoned.

Catalyst Testing

Additional tests are planned for the spring of 2002 at the UND steam plant. The evaluation of the gold and vanadium/titanium samples will continue. The Ryton will not be evaluated further, but a fixed bed of activated carbon will be evaluated. Bench- and pilot-scale fixed-bed tests with the FGD carbon have shown elemental mercury is oxidized by the carbon after an initial period of mercury capture.

Bioleaching Pretreatment

A smaller pot-scale study will be performed using *Thiobacillus thiooxidans* operating on a sample of the Blacksville coal. *Thiobacillus thiooxidans* directly reduces sulfides. If the bacteria is successful at accessing the sulfide fractions within the pyrite grains, it should be possible to release the sulfides, including the mercury sulfide. These released sulfides will then be removed from the coal by a hot methanol flush.

Mercury Capture in a Wet Scrubber

The first step of the work proposed under the current year is establishing a reliable baseline for mercury scrubbing. The proposed plan includes analyzing the impacts of various flue gas constituents on the capture of oxidized mercury species and the conversion of elemental to oxidized mercury (or visa versa). It was also the intent of the baseline work to investigate the impact of scrubber operational parameters, such as pH and L/G, and determine liquid- and mass-transfer characteristics for the oxidized mercury in the scrubber.

Problems associated with oxidized mercury, and especially mercuric chloride, were recognized. That is, mercuric chloride tends to stick to surfaces of piping, vessels (scrubber), and sampling lines. The hypothesis at the beginning of this initial stage was that there would be some adsorption/desorption that

would occur within the system and that the system would reach some stable equilibrium. If this equilibrium point could be established, it could be used as a baseline for future work. However, based on the work performed to date, accurate and reproducible mercuric chloride data cannot be obtained from the system. Alternatives that are being pursued include basing results only on elemental mercury (i.e., focus the program on oxidation of elemental mercury and evaluate performance solely on the basis of removal of elemental mercury in the scrubber); determine the feasibility of Teflon coating the scrubber and effluent holding tank; and perform preliminary work at a more fundamental level in batch equipment and address scaleup issues in a larger system.

Electrical Properties of Ash

Data acquisition and reduction are nearly complete. An alternative method of ESR determination will be attempted to complete the work.

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