

CONTROL OF MERCURY EMISSIONS IN UTILITY POWER PLANTS

Joseph R. Morency
Thomai Panagiotou
Physical Sciences Inc.
20 New England Business Center
Andover, Massachusetts 01810

Raul F. Lobo
Dept. of Chemical Engineering
University of Delaware
Newark, Delaware

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Thomai Panagiotou
Physical Sciences Inc.
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Raul F. Lobo
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Abstract

In a program funded by the Department of Energy, Physical Sciences Inc. (PSI) has begun the development of a mercury sorbent using a zeolite material with a proprietary agent for improved capture of elemental mercury in utility power plants.

Previous research has shown the feasibility of using zeolites for mercury capture. Several sorbents were tested in the laboratory by flowing coal combustion flue gases with known concentrations of mercury through a heated filter containing a thin bed of each sorbent. Results showed that a zeolite treated with a proprietary agent performed as well as a treated activated carbon in removing elemental mercury from the gas stream.

This paper will present the latest work in this development program. It will focus on further laboratory work in which the zeolite sorbent with additive is injected into coal combustion flue gases that contain known concentrations of mercury. The objective of this effort is to evaluate sorbent efficiency under conditions that approximate full-scale utilities with electro-static precipitators. As in the previous fixed bed work, mercury concentration and speciation are measured using the modified Ontario Hydro Method. The test matrix will also include an activated carbon sorbent so that a comparison can be made with the zeolite sorbent. Future work will include pilot-scale testing at a utility power plant.

Introduction

Title III of the Clean Air Act Amendments (CAAA) of 1990 identified 189 organic and inorganic compounds, 11 of which are toxic metals, as hazardous air pollutants (HAPs). These regulations were aimed at reducing the public health risk from exposure to HAPs in ambient air and require regulation of routine and accidental emissions of each HAP from large industrial sources and from small commercial sources. Although coal-fired power plants are not yet specifically regulated for these toxic trace metals under the CAAA, the EPA is mandated to study emissions and their associated risks in these facilities and to implement regulations if necessary. Many studies have been undertaken in the United States and Europe to evaluate the extent of toxic

metal emissions from coal-fired power plants. In this country, the Electric Power Research Institute (EPRI) initiated the Power Plant Integrated Systems: Chemical Emission Studies (PISCES) in 1988 beginning with the compilation of data existing in current literature. Because there were significant gaps in this literature data, field studies were begun by both EPRI and the Department of Energy (DOE) in an effort to fill in those gaps. Data were gathered at plants that covered a range of coals, boiler types and pollution control technologies.

The results of these programs generally indicate that most of the toxic metals which are vaporized during coal combustion, to a large extent, condense on the fly ash and are captured in the particulate control device (PCD). However, the more volatile species, such as mercury and selenium, may to some extent remain in the vapor phase and escape to the atmosphere.¹⁻³ In the case of mercury, the fraction of total metal entering the system that does not condense after vaporization, and therefore escapes the PCD, can be significant. Even though the concentrations of Hg in coal are relatively low at 0.012 to 3.3 ppmw, releases to the environment can be considerable due to the large amounts of coal that are burned. Mercury concentrations in the flue gas streams of coal fired power plants have been reported at levels between 1 and 120 $\mu\text{g}/\text{m}^3$ with emissions from the plant of 0.5 to 6 kg/day.^{4,5} Typical values are in the range of 1 to 10 $\mu\text{g}/\text{m}^3$. It has been estimated that the global emissions of mercury from all sources is approximately 6,000 tons per year and that emissions from power generation account for almost 40% of that total.⁶

The forms of the mercury vapor in power plant flue gases are elemental Hg and oxidized Hg (probably HgCl_2 , and in some cases, HgO). Research has indicated that the form of the mercury has an effect on the percentage of the metal that is emitted from the stack. The presence of a Selective Catalytic Reduction (SCR) de- NO_x device at a pulverized coal-fired boiler in the Netherlands was found to oxidize elemental mercury to HgCl_2 . Because of the solubility of this form of mercury, emission reductions of 80% were achieved by a wet flue gas desulfurization (FGD) unit.⁷ Other studies have indicated that the percentage of oxidized mercury in the flue gas is affected by the fuel constituents. A comparison of laboratory data with equilibrium calculations demonstrated that the presence of chlorine in the fuel at sufficient levels will promote the oxidation of Hg to HgCl_2 .⁸ Pilot-scale tests comparing mercury concentrations in flue gases generated from two different coals also indicated that more oxidized mercury was found in the flue gas of the coal with the higher chlorine content.⁹ There are several factors then that affect the species of mercury that exist in the flue gas, and a technology for mercury control must be effective for all forms of mercury.

Due to the toxicity of mercury and prompted by the expected implementation of EPA regulations, much work has been done or is under way to develop more accurate methods to measure and processes to control mercury emissions in fossil fuel fired power plants. Bench-scale tests conducted with elemental mercury showed generally low removals using untreated activated carbon and increased removals with carbons treated with sulfur and other additives. This work also showed the potential for high mercury removals using mineral sorbents such as zeolites.^{10,11} All of this work also revealed that capture of mercury with these sorbents drops dramatically as the temperature increases above 200°C. Pilot-scale testing of mercury removal using activated carbon, both treated and untreated, achieved good results when the ratio of ionic

to elemental mercury in the flue gas was high. However, the presence of fly ash seemed to interfere with the capture in some cases.¹² Other tests with untreated activated carbon from lignite coal and a carbon impregnated with iodine showed mixed results for mercury capture under varied conditions.¹³ In general, a review of current work shows that, although much progress has been made in the area of mercury control in coal fired power plants, the results have been varied and more work needs to be done to identify an effective control technology.

One issue that may be raised concerning the use of activated carbon as a mercury sorbent is the increased carbon content in the fly ash due to the high levels of sorbent injection that may be necessary to meet expected mercury control limits. Coal fly ash has potential commercial value in the cement making process and in the plastics industry, but limits of carbon content are imposed to prevent degradation of the final product. For example, concrete used in areas with frequent periods of freezing weather is deliberately mixed with air bubbles to provide space for expansion and contraction of the other components. The excess carbon in the ash used in the cement actually absorbs the air, which produces a less durable material. The plastics industry requires filler materials for its products and would consider the use of coal fly ash that is low in carbon and of consistent quality. The issue of high carbon levels in fly ash is further complicated by the increasing use of low-NO_x burners by electric utilities to comply with other CAAA requirements. These burners, because of their operating characteristics, tend to increase the amount of unburned carbon contained in the fly ash. The use of carbon as a mercury control method coupled with low-NO_x burners can therefore lead to carbon concentrations that may render the ash commercially undesirable. The use of an alternative non-carbon based sorbent must be considered so that the sorbent does not adversely affect the fly ash and possibly turn a potential source of income for the utilities into an expense.

Physical Sciences Inc. (PSI) is investigating the use of a low cost additive to improve the capability of a high surface area sorbent such as a zeolite to remove **all** forms of mercury vapor from power plant flue gases. The success of a treated zeolite in preliminary research led to the conclusion that a zeolite material could achieve substantial mercury removal at a significant cost reduction over current mercury control technologies. In the current research program, PSI is pursuing full development of a zeolite sorbent for mercury control in utility power plants. The success of a zeolite sorbent has the potential to reduce overall costs of mercury removal and will eliminate the problem of increasing the carbon content of the ash. Achievement of high capture efficiencies for all mercury species will allow a sorbent injection system to be integrated into all types of power plant pollution control systems with a minimum addition of plant equipment and without effecting the commercial value of the ash.

Zeolite Structure

Zeolites are tectoaluminosilicates with crystalline structures constructed from TO₄ tetrahedra (T = Si, Al). In these tetrahedra, each apical oxygen is shared with an adjacent tetrahedron and therefore zeolites have framework structures with a metal to oxygen ratio of 2. The framework structures of zeolites are open and thus, zeolites reversibly adsorb water and other molecules, and contain non-framework cations that can be exchanged. There has been ample academic and industrial interest in these materials because, among other reasons, there is a very direct link

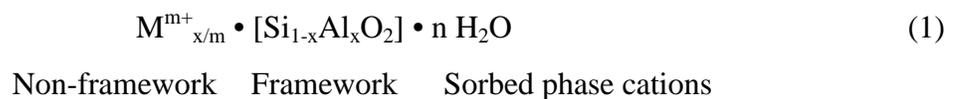
between their local structure and the bulk macroscopic properties. Zeolites are extensively used as catalysts in the petroleum and petrochemical industries; they are used as adsorbents for gas separation and purification, and they are also used as ion exchange media for water treatment and purification.

Structure-Property Relations in Zeolites

Although the individual tetrahedra (TO₄) in zeolites are rather rigid and regular, the T-O-T bond angles are very flexible (~125° to 180°) and therefore, the tetrahedra can be combined to form a great variety of structures.¹⁴ These structures have pores that vary in shape, size and dimensionality and importantly, since they are crystalline, the pore size distribution is essentially a delta function. The aperture dimensions which control entry into the pores are determined by the number of T-atoms and oxygen atoms that bind the zeolite pores. These apertures are of the same length scale as small molecules. Zeolites with 8-ring pores (such as zeolite A, Figure 1) have an effective pore opening of ~4 Å, zeolites with 10-rings have effective pore openings of ~5.4 Å (such as zeolite ZSM-5 and clinoptilolite) and zeolites with 12-ring pores have pore openings of ~7.4 Å (zeolites X and Y, see Figure 2).¹⁵ Since the pore sizes are of the same order of magnitude as small molecules, zeolites show very high selectivities in sorption and catalytic processes. Moreover, most atoms comprising the three-dimensional structure are part of the internal surface. Effective surface areas of 300 to 700 m²g⁻¹ are common, with more than 95% of the total surface area being internal.

The adsorption capacity (in milliliters of adsorbate per gram of dry zeolite) is a function of the pore size and pore dimensionality. Materials with a three-dimensional pore system have larger adsorption capacities (e.g., up to 0.3 ml of n-hexane per gram in zeolite X). Some small-pore zeolites also have large adsorption capacities due to the presence of large cages within the structure.¹⁶ Zeolites with three-dimensional pore systems are always the ones used in adsorption applications due not only to their adsorption capacity, but also to the faster diffusion rates (in comparison with one-dimensional zeolites where diffusion rates can be often slow) for most adsorbates.

The general formula for an aluminosilicate zeolite can be written as:



Each of these three components play an important role in the determination of the zeolite properties. The framework is anionic with a negative charge equal to the number of aluminum atoms [AlO₂]⁻. The non-framework cations are necessary to balance the charge of the framework and are incorporated during synthesis. The framework cations are normally only loosely bound to the zeolite framework and can be exchanged by treatment with a salt solution. The sorbed phase, indicated here as water, can be removed from the zeolite at high temperatures and low pressures preserving the structural integrity of the zeolite. This desorption process is reversible.

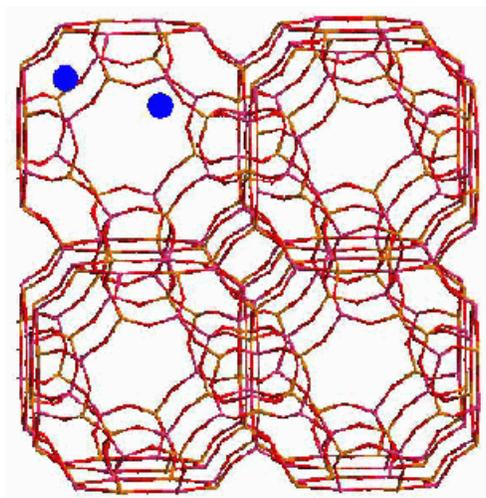


Figure 1.

Framework Structure Of Zeolite A. The Windows of this Zeolite Are Bounded by 8-Rings and Determine the Maximum Size of the Molecules that Can Enter into the Large Internal Cages (~11.5 Å). The Small Dots Represent Positions in Which Cations Are Typically Found Coordinated to the Framework.

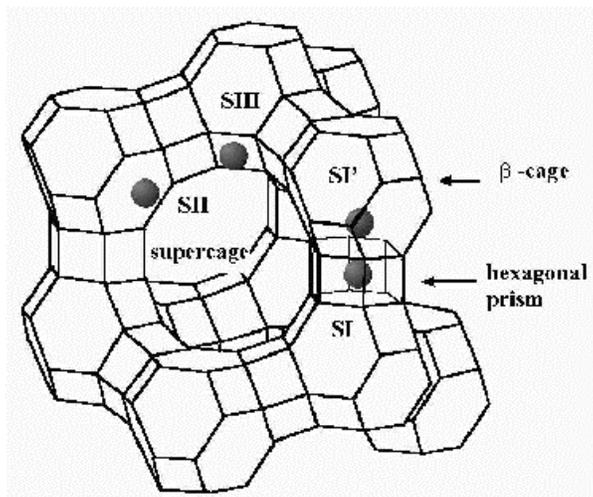


Figure 2.

Framework Structure of Zeolite X. The Supercage of this Zeolite is Accessed through 12-Ring Pores of ~7.4 Å. The Oxygens Connecting the Si and Al Atoms Have Been Eliminated for Simplicity.

The nature and quantity of the non-framework cations is also a key factor in the determination of zeolite properties. Upon dehydration, the cations in the zeolite pores will be highly undercoordinated and as a result, the cations will interact strongly with any Lewis base (or polar) molecule in the pores. Water, ammonia, CO₂, SO₂, etc., bind strongly to cations coordinated to the zeolite framework, and this tendency is exploited industrially for drying gases and to remove hydrogen sulfide and similar molecules from hydrocarbon gases. Cations tend to coordinate to specific locations in the zeolite framework. For example, the two cation locations for Na⁺ in zeolite A are depicted in Figure 1 in the 6- and 8-rings. After exchange of the Na⁺ for Ca²⁺ ions, only the 6-rings sites remain occupied. Since there were cations partially blocking the pore windows, the ion exchange extends the effective pore size from ~4 Å to ~5 Å. Similarly, in Figure 2 it can be observed that cations in sites SI, SI' and SII' are occluded within the small building units of the zeolite framework (β-cages and hexagonal prisms) and are precluded from interaction with molecules in the supercage. Only cations in sites SII and SIII are exposed to molecules in the supercage.

Although in principle many types of natural and synthetic zeolites can be used in medium to large quantities for industrial adsorption, in practice, the search should be focused on materials with a three dimensional pore system and a large adsorption capacity. The main advantages of the synthetic materials is their high purity and the possibility to tailor the properties by ion exchange and other post-synthesis treatments. Also, the zeolite adsorbents with the highest adsorption capacities are synthetic. The main advantage of natural zeolites is the cost.

Previous Zeolite Research at PSI

Zeolites have been examined in earlier work at PSI. Several sorbents were tested in a fixed bed mode for their ability to remove mercury from a coal combustion flue gas at 150°C. These sorbents included carbon and non-carbon based materials that had been treated with an additive to enhance their ability to remove all forms of mercury from the gas stream. To determine the ability of each sorbent to capture mercury under realistic conditions, the mercury-containing gas stream was to be generated by the combustion of coal in PSI's electrically heated Entrained Flow Reactor (EFR), shown schematically in Figure 3.

This apparatus, which can achieve a maximum gas temperature of 1723 K, has been used extensively on many of PSI's combustion research projects. Fuel, air, and any other desired gases are injected into the reactor through an alumina venturi that serves as a mixing section on the top of the EFR. The combustion section is an externally heated ceramic tube that is 8.57 cm in internal diameter and 158 cm in length. Gas sampling can be performed at the bottom of this section through one of several types of sampling devices that are available to meet the requirements of the research program.

Coal was burned in the EFR to more closely approach the conditions found in power plant flue gases. For this program, a bituminous coal, Pittsburgh seam, was used. Table 1 shows the ultimate and proximate analyses and the ash composition of the coal. The mercury content of the coal as determined by Neutron Activation Analysis is 0.11 ppm.

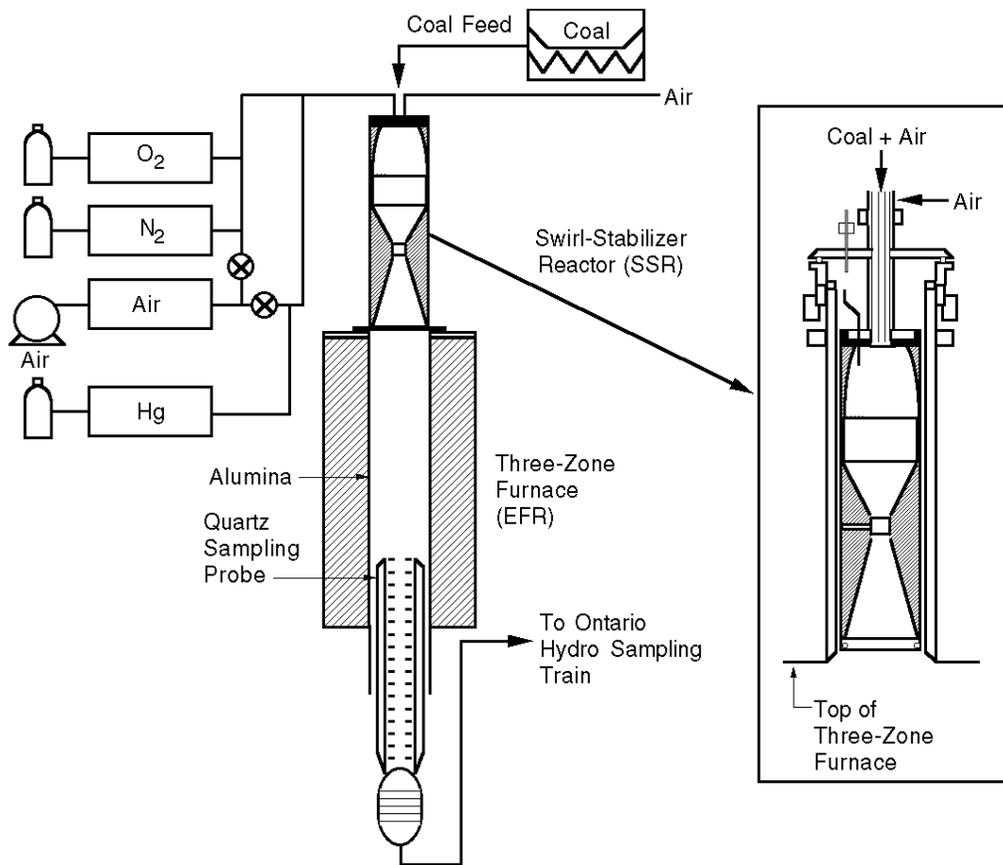


Figure 3
PSI Entrained Flow Reactor

Table 1. Properties of Pittsburgh Coal

Proximate (wt%, as received)		Ash Composition (wt %)	
Fixed Carbon	61.99	SiO ₂	42.92
Volatile Matter	30.22	Al ₂ O ₃	22.87
Moisture	1.44	Fe ₂ O ₃	19.18
Ash	7.01	TiO ₂	1.08
Ultimate (wt %, as received)		CaO	4.51
Carbon	76.62	MgO	0.97
Hydrogen	4.80	Na ₂ O	1.28
Nitrogen	1.48	K ₂ O	1.21
Sulfur	1.64	SO ₃	5.40
Oxygen	6.91	P ₂ O ₅	0.57
Chlorine	0.098		
Moisture	1.44		
Ash	7.01		

Table 2. Flue Gas Composition from the Combustion of Pittsburgh coal in the EFR

Constituent	Volume %
CO ₂	16.14
H ₂ O	6.07
O ₂	3.73
N ₂	74.6
SO ₂ (ppm)	1293
HCl (ppm)	70
Total Hg*	41 µg/m ³

*With addition of elemental mercury

A device consisting of a temperature controlled permeation tube with a carrier gas flow of oxygen regulated by a mass flow controller was employed to inject additional mercury into the axial air stream at the top of the EFR. Using this technique, the presence of mercury was assured at the exit of the reactor while still utilizing the combustion of coal to ensure the presence of any flue gas constituents, for example sulfur or chlorine, that may interfere with or accelerate the reaction of the mercury with the sorbent in the fixed bed. Table 2 shows the approximate composition of the gas stream exiting the reactor.

Once the flue gas exited the reactor, it was drawn through an unheated section of the reactor to reduce the temperature to approximately 250°C. It then passed through a glass cyclone which removed most of the ash particles from the stream, thereby minimizing particle contact with the mercury vapor and precluding capture of the mercury by any ash constituents. The remaining finer ash particles were removed by a Whatman 934 AH Glass Microfiber Filter in a 100-mm-diameter glass holder placed in a temperature-controlled box adjusted to 150°C to simulate baghouse temperatures. For the sorbent testing runs, the sorbent sample was placed in a 2- to 5-mm-thick bed on the heated filter holder and the fly ash was allowed to accumulate on top of it. The sample gas flow rate was adjusted to 13.9 slpm and its velocity through the bed, or face velocity, was calculated to be 0.09 m/s. This yields gas/sorbent contact times of approximately 500 ms.

A series of runs was also conducted at a filter box temperature of 110°C using the sorbent that demonstrated the most potential for further development in the high temperature test series. This filter temperature was chosen to cover the range of temperatures in which the mercury sorbent would be expected to perform (110 to 150°C). All other test conditions for these runs remained the same as the high temperature series. These runs consisted of a baseline test with no sorbent and a sorbent test in the fixed bed.

After leaving the filter system, the gas stream passed into a series of impingers designed to collect and determine the species of the mercury in the vapor. For this setup we used the Ontario Hydro Method which in recent testing has achieved good results in measuring total mercury and has shown the most promise for the determination of mercury species. Figure 4 shows a schematic of this system.

Before testing the sorbents for mercury capture ability, characterization runs were performed without sorbents to determine the total mercury concentration in the gas stream and to estimate the split between oxidized and elemental mercury in the flue gas.

All of the sorbents tested in this project exhibited some capture of mercury at 150°C with the higher surface area sorbents generally exhibiting higher mercury capture per gram of sorbent. It has been shown that addition of the PSI additive improved the ability of a zeolite to remove both oxidized and elemental mercury at both high and low temperatures and showed the potential of this material for further development as a mercury sorbent. This sorbent can be utilized for mercury control without impacting the quality of the fly ash thus preserving the commercial value of the ash. Leachability tests on this material showed that the sorbent can be safely disposed of in a landfill if sale of the ash is not an option.

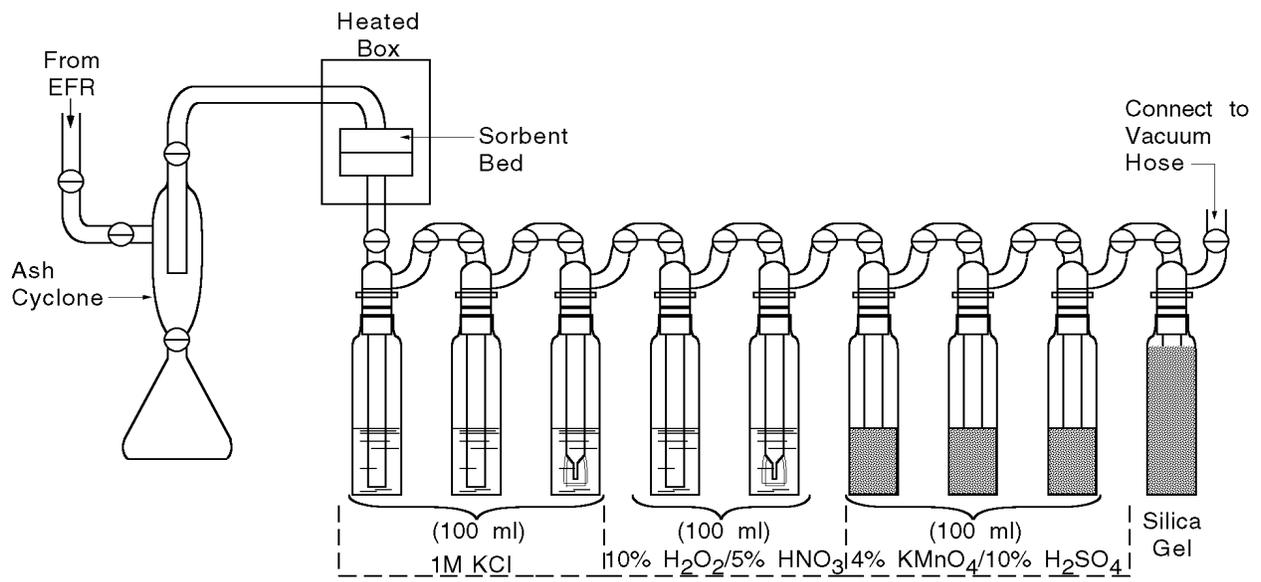


Figure 4
 Modified Ontario Hydro Method for Capture of Mercury Species in the
 Sample Gas Stream of the EFR

Experimental Apparatus for the Current Tests

For the current work, sorbents were tested by injecting them into the flue gas to determine their ability to remove vapor phase mercury in-flight. The sorbents chosen for this work were a high-surface-area activated carbon, CL-213 from Barneby and Sutcliffe, natural zeolite which had been size-classified to diameters of 1 to 5 μm , and the same zeolite treated with the proprietary agent to improve mercury capture. The apparatus for these experiments was identical to the fixed bed work with the addition of some key components. The first of these was a 5 cm diameter by 50 cm long quartz tube placed at the exit of the quartz sampling tube at the bottom of the EFR. This device was intended to simulate the power plant ductwork prior to an electrostatic precipitator (ESP). The sorbents were injected into the coal combustion flue gas stream at the top of the tube and the residence time in the tube was approximately 2 seconds. Immediately after the quartz tube were the particulate capture devices. The first of these was a simple impinger, similar to the liquid impingers, but with a shortened inlet tube. Following this device were the two glass cyclones in series to further reduce gas stream particle concentration.

The reason for so many particulate removal devices was to separate as much of the ash and sorbent particles as possible from the gas stream so that an accurate assessment could be made of the sorbent's ability to remove mercury in-flight. Any sorbent collected on the filter would have a much more intimate contact with mercury in the gas stream which would approximate conditions found in a baghouse type particulate collection device, but would not be accurate for a full size ESP. The particulate captured in the cyclones or collected on the walls of connectors leading to and from these devices was assumed to have no further contact with the gas stream, and thus any mercury captured by this particulate was removed while the sorbent was in-flight. All of this particulate was recovered and analyzed as one sample while the filter particulate was analyzed separately.

All of these devices were wrapped with heating tape or a heat mantle to maintain gas temperature and prevent mercury condensation on cold spots on the glass surfaces. From here the gas stream entered the heated filter box, which now served to remove any remaining particulate from the gas stream, and then into the Ontario Hydro train as in the previous work.

As in the previous work, additional mercury was added to the flue gas stream before combustion of the coal. The primary reason for doing this was to accommodate the lowest sorbent feeder setting and thus allow achievement of very low sorbent/Hg ratios. The final mercury concentration in the flue gas was approximately $40 \mu\text{g}/\text{Nm}^3$ which is within the range of values to be found in utility power plants.

Sample Analysis

For each run, there were two solid samples: one designated as cyclone ash which consisted of all ash from the particulate capture devices and connecting tubes, and the second designated as filter ash. These samples were first digested using an HF microwave digestion procedure and then analyzed by AA. The analytical laboratory was also sent a sample of Standard Reference

Material #1633b, Coal Fly Ash from National Institute of Standards and Technology (NIST). Their analysis of this material was 82% of the certified value for mercury.

The liquid samples for the runs consisted of four samples: three impinger samples from the Ontario Hydro train: KCl, H₂O₂/HNO₃, and KMnO₄, and one sample consisting of the nitric acid rinse of all the glassware prior to the heated filter box. These samples were analyzed according to the procedures for Ontario Hydro method. In addition to these samples, blanks and spiked samples of each impinger solution were analyzed by the laboratory.

Results

Mercury mass balance closure was determined for all runs by adding the mercury from each of the analyzed samples and dividing by the sum of the theoretical mercury from the combustion of the coal and the added mercury from the permeation tube as determined by the Jerome analyzer. Using this method, mercury recovery for these experiments ranged from 75% to 126%. Mercury was captured either in the ash samples or in the impingers. Little or no mercury was recovered from the glassware rinses which indicates that the mercury was not adhering to the walls anywhere in the system.

Only a small portion of the test matrix has been completed to date; however, initial results are promising. Analysis of the cyclone ash and the filter samples shows that most of the mercury is being captured in-flight while very little is collected on the filter. This seems to indicate that the sorbent is being removed from the gas stream by the cyclones and not the filter and that the apparatus can be used as a test bed for sorbent injection work. Preliminary analysis of the data indicates that the treated zeolite sorbent performs well when compared to the activated carbon even at low sorbent injection rates. Although much work needs to be done, results show that the zeolite sorbent may remove up to 4 times the amount of mercury per gram of sorbent as the activated carbon. Sorbent/Hg ratios of as low as 5000:1 have been achieved using the syringe pump feeder, which will enable us to perform tests under a wide range of conditions.

Future testing will include additional sorbent injection ratios for the treated zeolite and activated carbon as well as testing of the zeolite material with another proprietary agent. At the conclusion of the laboratory tests, pilot-scale work will begin at Public Service Electric and Gas Mercer Station in New Jersey. At that location, sorbent injection of the treated zeolite material will be conducted on a slipstream of Unit #2 to determine its performance in a power plant flue gas under various conditions. The results of both the laboratory and the pilot work will be used to develop a cost study to assess the performance of the sorbent and determine its viability in the marketplace in the coming regulatory climate.

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