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Project Title: **THE ADVANTAGE OF ILLINOIS COAL FOR FGD REMOVAL OF MERCURY**

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ABSTRACT

Research was conducted to characterize and modify mercury speciation in Illinois coal combustion flue gas so that a mercury control strategy can be implemented in conventional flue gas desulfurization (FGD) units. Mercury, present as traces in coal, is readily volatilized during coal combustion and leaves the high temperature zone as gas-phase elemental mercury (Hg^0). Previous results have shown that as the flue gas is cooled, a portion of Hg^0 is converted, primarily to mercuric chloride (HgCl_2), in the presence of catalytically active surfaces such as certain fly ashes. Unlike Hg^0 , HgCl_2 is highly soluble in water and has a high affinity for alkaline sorbents; it can therefore be easily removed in wet scrubbers and spray dryers (FGD units). This research specifically examined the effect of injection of Illinois coal combustion residues (ICCRs) on the conversion of Hg^0 to HgCl_2 in coal combustion flue gases. Tests were conducted in two tasks. Task I studied Hg^0 oxidation using a fixed-bed, bench-scale reactor in a simulated Illinois coal combustion environment. Various types of ICCRs were examined to determine active residues in the oxidation of Hg^0 . A scrubber sludge and two bottom ash samples were determined to be active Hg^0 catalysts. The most active ICCR (a bottom ash sample) was chosen for Task II. Task II was performed in two subtasks. In Subtask I, the active bottom ash sample was injected in a pilot-scale combustor while burning natural gas doped with appropriate amounts of Hg^0 , hydrogen chloride (HCl), nitrogen oxides (NO_x) and sulfur dioxide (SO_2). Injection of this bottom ash sample did not contribute to the background oxidation of Hg^0 (gas-phase oxidation), indicating that in-flight oxidation of Hg^0 in the pilot-scale combustor may be mass transfer limited. In Subtask II, a representative Illinois coal was combusted in the pilot-scale combustor. As is the case with most eastern bituminous coals, the combustion flue gas of this coal was dominated by oxidized forms of mercury (most probably HgCl_2). Additional Hg^0 was doped into this flue gas, and significant oxidation of Hg^0 was observed. It was determined that the fly ash generated from combustion of this coal is very active in oxidation of Hg^0 . Combustion of this Illinois coal in power plants equipped with FGD units should provide a better than 90% mercury emission control. Additional pilot-scale tests were performed to investigate the activity of the Illinois coal fly ash. It was determined that packed-bed flow modes, such as those observed in baghouses, are required to catalytically oxidize Hg^0 using this Illinois coal fly ash.

EXECUTIVE SUMMARY

The Air Pollution Prevention and Control Division (APPCD) of the United States Environmental Protection Agency (EPA) proposed co-funded research in conjunction with the Illinois Clean Coal Institute (ICCI) to evaluate the reduction of volatile mercury through speciation modification [i.e., oxidation of elemental mercury (Hg^0) to mercuric chloride (HgCl_2)] and subsequent removal by conventional flue gas desulfurization (FGD) units. This research studied whether speciation modification could be effected through injection of Illinois coal combustion residues (ICCRs). Title III of the Clean Air Act Amendments (CAAA) places limitations on emissions of various air toxics. Among these, Hg^0 poses a serious challenge to air pollution control technologies due to its high volatility, insolubility, and low adsorption affinity. For Illinois coal-fired utilities, reduction of this species is critical in complying with the emission standards set forth by the CAAA. Oxidation of Hg^0 to HgCl_2 prior to an FGD unit is a viable option for controlling mercury emissions since HgCl_2 is easily removed by wet scrubbers and spray dryers. Since mercury is present in Illinois coals, this research provides control strategies for reducing mercury air toxics emissions in utilities that are using Illinois coals.

Currently, there are no existing technologies for mercury speciation modification prior to an FGD unit. This study investigated the viability of such a technology in Illinois coal combustion processes. The objectives of this research were to conduct bench- and pilot-scale studies in order to identify conditions that favor formation of HgCl_2 . This objective was initially accomplished through a systematic, bench-scale study of the effect of gas-phase sulfur dioxide (SO_2), nitrogen oxides (NO_x), and hydrogen chloride (HCl) (at levels representative of Illinois coal combustion) on oxidation of Hg^0 to HgCl_2 in the presence of ICCRs. In addition, information gathered previously in the EPA laboratories on the effect of transition metals on catalytic oxidation of Hg^0 to HgCl_2 was used to determine and quantitate the catalytic activities of injected ICCRs on the oxidation of Hg^0 . An active ICCR was identified during the bench-scale tests and was subsequently injected in a pilot-scale natural gas or coal combustor to evaluate and establish the extent of Hg^0 oxidation capability of the selected ICCR.

This research consisted of two tasks. Task I involved bench-scale, fixed-bed testing of a variety of ICCRs (three fly ashes, two bottom ashes, and two scrubber sludges) to identify the most active residue in catalyzing oxidation of Hg^0 to HgCl_2 in the presence of simulated coal combustion flue gas containing oxygen (O_2), carbon dioxide (CO_2), water vapor (H_2O), SO_2 , NO_x , and HCl. Previous EPA investigations with synthetic and actual fly ashes have indicated that certain transition metals (iron and copper) are very active in catalyzing conversion of Hg^0 to HgCl_2 in the presence of HCl and NO_x . Out of seven tested ICCRs, three residues exhibited measurable and rather significant Hg^0 oxidation activities. These samples included a bottom ash (2PPB) and a scrubber sludge (2PPS) sample from power plant #2 (see the 1998 ICCI request for proposals for detailed descriptions) and a bottom ash (4PPB) sample from power plant #4. Fixed-beds of these residue samples (about 0.1 g mixed with 2 g sand) exhibited Hg^0 oxidation capabilities of 20-30% at a temperature of 250 °C and exposure times of 90 minutes. Based on the availability and Hg^0 oxidation capability, the bottom ash from power plant #2 (2PPB) was chosen for the pilot-scale combustion tests (Task II). X-ray fluorescence (XRF) analysis was also performed on this sample to determine its

elemental composition. The iron and copper content of this sample were determined to be 7.5 and 0.008 wt%, respectively. This bottom ash sample has a considerable amount of iron and a negligible amount of copper as compared to other coal bottom and fly ashes.

Task II was performed in two subtasks. In Subtask I, the active ICCR (2PPB) was injected in a pilot-scale 18.6 kW (80,000 Btu/h), refractory-lined, down-fired cylindrical furnace fired with natural gas. This furnace, termed the “innovative furnace reactor” (IFR), also has the capability of firing pulverized coal. The natural gas combustion flue gas was doped with coal combustion levels of Hg^0 (20-25 $\mu\text{g}/\text{dscm}$), HCl (50 ppm), NO_x (250 ppm), and SO_2 (1500 ppm). These species were doped into the IFR immediately after the natural gas flame, at a temperature of about 800 °C. Mercury speciation of the flue gas (Hg^0 and HgCl_2 content) after 2 to 3 s of gas-solid residence time (a drop in temperature from 800 to 300 °C) was measured using a mercury speciation method known as the Ontario-Hydro (OH) method. Blank tests (in the absence of sample 2PPB) were also performed to assess the extent of gas-phase conversion of Hg^0 to HgCl_2 . Blank tests showed that about 36% of the injected Hg^0 was converted to oxidized forms of mercury even in the absence of particulate injection, indicating that the gas-phase reaction of Hg^0 , most probably with HCl, can occur at a residence time of 2-3 s and a temperature range of 800-300 °C. Following the blank tests, the active ICCR (sample 2PPB) was injected in the IFR at a rate of [2 lb/hr (0.9 kg/hr)] to generate a particulate matter concentration of about 2000 mg/dscm at the point of the OH measurements. Injection of this sample did not contribute to any additional oxidation of Hg^0 ; about 36% oxidized mercury was observed in the presence of the bottom ash sample. This suggests that the in-flight, short-time-scale (2-3 s) oxidation of Hg^0 in the presence of the active ICCR is unlikely to occur, thus duct injection of this sample can not be used as a speciation modification method.

In Subtask II, a representative Illinois coal sample (pulverized, sample #IBC-110) was combusted in the IFR [firing rate of about 80,000 Btu/hr (18.6 kW); coal feed rate of 6 lb/hr (2.7 kg/hr)]. The objective was to determine baseline mercury speciation in the flue gas and subsequently inject the active ICCR sample (2PPB) into the flue gas to implement a change in speciation from Hg^0 to HgCl_2 . Speciation of mercury in the Illinois coal combustion revealed mostly oxidized forms. A total mercury concentration of 7.7-9.7 $\mu\text{g}/\text{dscm}$ was obtained; about 90-95% of the mercury was either gas-phase oxidized or particulate bound mercury. It was therefore determined that the injection of 2PPB could not contribute to additional oxidation. As a result, 2PPB injection tests were not performed. As is the case with most eastern bituminous coals, combustion of this Illinois coal predominantly produced oxidized mercury (most probably HgCl_2). It was hypothesized that the fly ash and the flue gas generated during combustion of this coal is very active in the oxidation of native mercury in the coal. To prove this hypothesis, additional Hg^0 was injected into the combustion flue gas of this coal immediately after the flame. About 83% of the injected Hg^0 was oxidized in the flue gas of this coal and in the presence of its fly ash. Additional natural gas pilot-scale tests were performed to investigate the activity of this Illinois coal (IBC-110) fly ash. It was determined that packed-bed flow modes, such as those observed in baghouses, are required to catalytically oxidize Hg^0 in the presence of this coal's fly ash. In summary, combustion of this Illinois coal in power plants equipped with baghouses and FGD units should provide a better than 90% mercury emission control.

OBJECTIVES

The specific goals of the research were to study the potential conversion of Hg^0 to HgCl_2 in the presence of Illinois coal combustion residues (ICCRs). Control of HgCl_2 emission in coal burning power plants can be easily implemented using existing wet or dry FGDs. The initial goal was to screen a number of ICCRs in a well-controlled, bench-scale experimental system and to identify the most active one. A subsequent objective of this study was to inject the active ICCR in a pilot-scale natural gas and coal combustor to effect in-flight, short-time-scale conversion of Hg^0 to HgCl_2 prior to FGD units. These objectives were performed in two tasks:

- Task I: Perform bench-scale, fixed-bed tests to study oxidation of Hg^0 to HgCl_2 by seven different ICCRs. Illinois coal combustion flue gas conditions were simulated in the bench-scale system and fixed-bed reactors with ICCRs were exposed to Hg^0 laden simulated flue gases at a bed temperature of 250 °C. The amount of Hg^0 oxidation across each ICCR was quantified.
- Task II: Evaluate the potential Hg^0 oxidation capability of the most active ICCR selected during Task 1 in a pilot-scale combustor. This ICCR was injected in the duct of a pilot-scale combustor while burning natural gas (doped with Hg^0 , SO_2 , HCl , and NO_x) and an Illinois coal. The extent of Hg^0 conversion to HgCl_2 (mercury speciation modification) was evaluated during these tests.

INTRODUCTION AND BACKGROUND

The area of this research was of particular relevance and significance in that Title III of the Clean Air Act Amendments calls for control of various hazardous air pollutants (HAPs), including mercury. Although mercury is present as traces in various coals, including Illinois coals, even low concentrations in large volumes of flue gases may result in non-compliance. Preliminary results in the U.S. Environmental Protection Agency's (EPA's) laboratories have indicated the effectiveness of spray dryers and wet scrubbers (FGD units) in removing HgCl_2 (and not Hg^0) from flue gases (Ghorishi and Gullett, 1998). Previous investigations have also indicated that many flue gas and fly ash parameters determine the speciation of mercury ($\text{Hg}^0/\text{HgCl}_2$ ratio) in a simulated combustion environment (Ghorishi, 1998; Ghorishi et al., 1999).

Metallic air toxics in flue gases are found in elemental or various oxidized forms and exist in the solid, aerosol, or vapor state. These compounds originate from the raw coal and are typically enriched in the fine particles (Markowski and Filby, 1985). The form and size distribution of each metal is a function of metal- and system-specific properties. For example, various concentrations of chlorine or sulfur have been shown to significantly affect the expected equilibrium product distribution (Linak and Wendt, 1993). Ghorishi et al. (1999) have shown that in combustion flue gas, and in the presence of active simulated and actual fly ashes, Hg^0 is readily oxidized by HCl and NO_x at temperatures typical of air

pollution control systems (150-250 °C). The presence of these active fly ashes, NO_x, and HCl in the flue gas can therefore shift the ratio of Hg⁰ to an ionic form of mercury (specifically HgCl₂) and positively influence the performance of the FGD units. Quantitative information on speciation of mercury in Illinois coal combustion flue gases in the presence of NO_x/HCl and Illinois coal combustion residues is lacking at this time. Task I of this research was aimed at addressing these issues.

Without additional modifications, conventional FGD systems in municipal waste combustors (MWCs) are capable of removing mercury emissions to some extent (White et al., 1993). Limited tests performed by White and co-workers without specific control for mercury emissions showed a reduction in mercury emissions ranging from 24-76%. The control of mercury emissions has been found to be strongly dependent on the form, or species, of mercury. Pilot-scale data (Schager, 1991) where activated carbon injection technology was used, showed the oxidized form to be more easily captured than Hg⁰. Similar results were observed at the Electric Power Research Institute's High Sulfur Test Center's (HSTC) 4 MW_e pilot wet FGD system and at the MWC Sysav tests (Volland, 1991). The presence of HCl in the combustion flue gases shifts the total mercury concentration toward its oxidized form (Hall et al., 1991; Ghorishi, 1998). The type of coal has also been found to affect different levels of mercury capture (Felsvang, 1992; 1993). The chlorine content in coal, capable of forming the less volatile and more water-soluble mercuric chloride (HgCl₂), is thought to be the factor for differences in mercury capture (Felsvang et al., 1993; DeVito et al., 1993).

Therefore, an important scientific issue that needs to be addressed is the chemical form of mercury in coal combustion flue gases. Previous EPA studies have shown the relative ease of controlling HgCl₂ as opposed to Hg⁰ (Ghorishi and Gullett, 1998; Gullett and Jozewicz, 1993) under conditions similar to those experienced in the FGD units. Knowledge on the transformation of Hg⁰ to HgCl₂ is absolutely crucial in this regard. Most researchers agree that mercury in coal vaporizes completely in the combustion zone of a boiler and leaves this zone in the form of Hg⁰ in the gas phase. Some oxidation of Hg⁰ may occur as the flue gas cools. At the economizer exit, where the flue gas typically enters the FGD units, mercury can be found as Hg⁰ or ionic form (most probably HgCl₂). Predicting emissions of mercury species has been a problem, since the transformation of Hg⁰ in the post-combustion region is not well understood. A detailed review on the state of knowledge on mercury speciation in coal-fired processes has been performed by Senior et al. (1997). Their review and another investigation (Krishnan et al., 1995) concluded that the assumption of gas-phase equilibrium for mercury species in coal-fired flue gases is not valid, and major reaction pathways for mercury oxidation in coal combustion flue gas need to be investigated. These investigations should include fly-ash-mediated surface reactions as well as gas-phase reactions (Senior et al. 1997). Therefore, the following two questions should be the primary focus of the on-going and future research:

1. What is the rate of oxidation of Hg⁰ in the post-combustion flue gas? Answering this question would lead to prediction of mercury speciation (Hg⁰/HgCl₂ ratio) at the inlet of the FGD units.

2. What properties (or which components) of coal fly ash affect the oxidation of Hg^0 ? In other words, what are the processes through which fly ash components mediate or catalyze the transformation of gaseous Hg^0 to HgCl_2 ?

A preliminary investigation was performed at the EPA's laboratories (Ghorishi, 1998) to provide some insights into these questions by studying mercury speciation in simulated coal combustion flue gases. Gas-phase studies indicated that the in-flight, post-combustion oxidation of Hg^0 in the presence of HCl is very slow and proceeds at measurable rates only at high temperatures ($>700\text{ }^\circ\text{C}$) and high HCl concentrations (200 ppm). The presence of SO_2 and H_2O in the simulated flue gas significantly inhibited the gas-phase oxidation of Hg^0 in the presence of HCl. These results indicate that conversion of Hg^0 to HgCl_2 needs to be mediated by the presence of active surface such as certain fly ashes. The effects of coal fly ash components and compositions were investigated using a fixed-bed of model or simulated fly ashes. The results are summarized in Figures 1 and 2. The primary focus was on evaluating the catalytic Hg^0 oxidation activity of major mineral constituents of coal fly ashes: alumina (Al_2O_3), silica (SiO_2), iron (III) oxide (Fe_2O_3), copper (II) oxide (CuO), and calcium oxide (CaO). Copper and iron oxides were the only two components that exhibited significant catalytic activity toward a surface-mediated oxidation of Hg^0 . The observed catalytic activities were hypothesized to be effected through the formation of a chlorinating agent (most probably Cl_2) from gas-phase HCl on the surface of metal oxides (the Deacon process reaction). Copper was a much more active catalyst than iron, and its catalytic activity was less influenced by the presence of oxidation inhibitors (SO_2 and water vapor) in the simulated flue gas. The presence of small quantities of CuO (0.1 wt%) in the model fly ash caused a 95% oxidation of Hg^0 in the temperature range of 150-250 $^\circ\text{C}$. The same extent of Hg^0 oxidation was obtained by adding 14 wt% Fe_2O_3 to the model fly ash.

EXPERIMENTAL PROCEDURES

Screening of the ICCRs for their Hg^0 oxidation capabilities was performed in a bench-scale, fixed-bed system. A schematic of the fixed-bed, Hg^0 oxidation reactor is shown in Figure 3. Pure Hg^0 (in liquid form) in a permeation tube (VICI Metronics, Inc.) generated the desired Hg^0 vapor. The concentration of Hg^0 (in the gas) was controlled by adjusting the water bath temperature. A detailed description for the calibration of the Hg^0 vapor generation system and its quality control checks have been reported elsewhere (Krishnan et al. 1994; 1996). The Hg^0 vapor generated (40 ppbv) was carried into the manifold by a nitrogen (N_2) stream, where it mixed with carbon dioxide (CO_2 , 5 mole%), oxygen (O_2 , 2 mole%), NO_x (250 ppmv), HCl (50 ppmv), SO_2 (500 ppmv) and water vapor (H_2O , 1.7 mole%) at a constant total system flowrate of 300 cm^3/min (at standard temperature of 20 $^\circ\text{C}$ and pressure of 101.4 kPa). A three-way valve placed before the manifold (Figure 4) diverted the Hg^0 -laden N_2 stream away from the manifold, when desired. The first three-way valve placed ahead of the oxidation reactor was used to direct flow to or away from the reactor. The reactor inlet Hg^0 concentration was measured when the valve was turned to the by-pass mode. It should be noted that the oxidation reactor is made of quartz, and the connecting lines are made of Teflon. Prior quality control checks (Krishnan et al., 1994; 1996) have shown that this system had no affinity for Hg^0 . The

gas-phase residence time (empty reactor) is 2 seconds at 20 °C. The tested ICCRs (0.1 g mixed with 2 g sand, bed length of approximately 2 cm) were placed in the oxidation reactor, which was maintained at a bed temperature of 250 °C by a temperature-controlled heating tape. At the beginning of each test, the concentration of Hg⁰ vapor generated by the permeation tube (hereafter, inlet concentration, Hg_{in}) was registered by an on-line ultraviolet (UV) Hg⁰ analyzer (Buck 400A, detection limit of 1 ppb Hg⁰), while the oxidation reactor was in the by-pass mode. It is important to note that the UV analyzer does not respond to oxidized forms of mercury. During each test with the reactor on-line, the post-reaction or outlet Hg⁰ vapor concentration (Hg_{out}) was measured continuously using the Hg⁰ UV analyzer (experimental variability of ±5%). Considering the fact that mercury in the flue gas exists in either the elemental or oxidized form, the difference between the inlet and the outlet Hg⁰ concentration was used to quantify the extent of oxidation of Hg⁰ in the reactor. Percent oxidation was obtained as:

$$\% \text{ Oxidation} = 100 * (\text{Hg}_{\text{in}} - \text{Hg}_{\text{out}}) / \text{Hg}_{\text{in}}$$

Water vapor created interferences in the UV Hg⁰ analyzer. Prior to entering the Hg⁰ analyzer, water vapor was removed from the simulated flue gases. A NAFION[®] gas sample dryer (Perma Pure, Inc.) Was used to selectively remove water vapor from the effluent of the oxidation reactor. Repeated quality assurance checks have indicated that this system has no affinity toward adsorption of Hg⁰ and acid gases present in the flue gas. The UV Hg⁰ analyzer responded to SO₂ as well as Hg⁰. For instance, a gas stream consisting of 500 ppm SO₂ and 40 ppb Hg⁰ produced a SO₂/Hg⁰ signal ratio of 1/12. Contributions from SO₂ was corrected by placing a SO₂ analyzer (UV, model 721AT2, Bover Engineering, Inc.) on-line, downstream of the Hg⁰ analyzer. The SO₂ analyzer was incapable of responding to mercury in the concentration range used in this study. By subtracting the SO₂ signal measured by the SO₂ analyzer from the total response of the Hg⁰ analyzer, the outlet Hg⁰ concentration was obtained.

Task II tests were performed in a pilot-scale 18.6 kW (80,000 Btu/hr), refractory lined, down-fired cylindrical furnace fired with coal and natural gas (Figure 4). The furnace, termed the “innovative furnace reactor” (IFR), has an inner diameter of 15.2 cm and an overall vertical length of about 4 m. After the vertical section and a heat exchanger, a horizontal, stainless steel duct section (about 3 m) directs the flue gas to a baghouse and a scrubber. The furnace was used to simulate and generate a coal combustion environment and quench rate conditions anticipated upstream of FGD units in coal-fired utility boilers. View and injection/probe points traverse the length of the furnace for testing flexibility. Sampling ports along the 3-m horizontal duct section allow gas and particle monitoring. The IFR was equipped with a complete continuous emission monitoring (CEM) system for O₂, CO₂, NO_x and SO₂. The furnace was operated under normal conditions with tangential and axial air totaling 0.275 m³/min [standard temperature and pressure (STP)], including an excess air of 35%. The IFR is equipped with a baghouse and scrubber to treat the flue gas before releasing it into the environment. During the natural gas test, Hg⁰, HCl, SO₂, and NO_x were doped immediately after the flame (in the vertical section) at a temperature of about 800 °C. Mercury speciation measurements were performed in the horizontal duct of the IFR (upstream of the baghouse) using the Ontario-Hydro (OH) method. A detailed description of this method is outlined elsewhere (Linak et al., 2001

and the Standard Method outlined in the reference section). OH method measurement was performed in a high particulate matter (PM) environment (PM concentrations of 200-2000 mg/dscm). The filter of the OH method, under this high PM environment, introduces a fixed bed in the sampling system, thus creating biases in the duct mercury speciation measurement. The speciation and capture of mercury across the filter of the OH method may be affected. To assess and correct for these biases, simultaneous isokinetic and non-isokinetic OH sampling were performed in the horizontal duct section of the IFR. The isokinetic OH train used a 1" probe with a sampling rate of 0.6 cfm while facing the flow. The non-isokinetic OH train used an oversized 2.4" probe with a sampling flow rate of 0.2 cfm while faced away from the flow. The non-isokinetic train was located about 3 feet downstream of the isokinetic train. The non-isokinetic sampling train was designed to capture a minimum amount of PM from the duct flue gas. The purpose was to assess the extent of mercury speciation change across the OH filter. The non-isokinetic OH train was a measure of gas-phase mercury speciation in the IFR duct, while the isokinetic OH train indicated the total amount of mercury in the duct (gas and particulate mercury). It was anticipated that the comparison between these two trains will reveal the change in mercury speciation achieved through injection of the active ICCRs or the coal's native fly ash in the duct of the IFR only, and not across the OH filter.

RESULTS AND DISCUSSION

The results of each task are discussed separately in this section.

Task I: Screening of the ICCRs with regard to their Hg⁰ oxidation activities

The bench-scale, fixed-bed setup (Figure 3) was used for these screening tests. In this task, Hg⁰ oxidation capabilities of seven different ICCR samples were evaluated. The residue samples were obtained from the Illinois Basin Coal Combustion Residues Sample Program (CRSP). Table 1 summarizes the sample designations. Power plant #2 is located near the Illinois-Indiana border. It burns Springfield (No. 5) and Herrin (No. 6) coals combusted in PCC boilers. This plant has two wet limestone scrubber units; one utilizes an inhibited oxygen cycle and the other employs a forced-oxidation cycle. Power plant #3 is located in central Illinois and burns Herrin (No. 6) and Springfield (No. 5) coals, which are mined in southern and central Illinois, respectively. This plant uses a fluidized-bed combustion technology. Power plant #4 is located in central Illinois and burns Springfield (No. 5) coal. The coal is combusted in one PCC boiler and two cyclone boilers. A wet limestone scrubber utilizing a forced-oxidation cycle has been installed on the PCC unit.

Table 1. Illinois coal combustion residue designations

Sample ID #	Power Plant #	Sample Type
2PPF	2	Fly Ash

2PPB	2	Bottom Ash
2PPS	2	Scrubber Sludge (inhibited oxygen cycle)
3PF	3	Fly Ash
4PPF	4	Fly Ash
4PPB	4	Bottom Ash

Hg⁰ oxidation activities of the Illinois coal combustion residue samples are included in Table 2. A number of tests were replicated. Some of the samples were not homogenous, thus reproducible data could not be easily obtained. Best efforts were made to obtain a representative sample (0.1 g) for fixed-bed oxidation tests. Samples 2PPS, 4PPB, and 2PPB appeared to be the most active residues. Sample 2PPS was not a homogeneous sample. It is believed to be a mixture of scrubber sludge and fly ash. Apparently, the wet scrubber in this unit also acts as a particulate control device. Samples 4PPB and 2PPB are bottom ashes and require grinding and/or screening prior to the IFR tests. Based on these data and availability of the samples, the bottom ash sample 2PPB was selected for Task II studies. Injection of this sample into the IFR requires fine powder. A sieve shaker (Swaco CO.) was used to size-segregate the bottom ash sample. This sample was screened using a sieve mesh size of 87 (particle sizes of less than 163 μm). About 37% of this sample was collected using this sieve. This portion was used for the combustion studies in the IFR (Task II).

Table 2. Hg⁰ oxidation activity (%) of Illinois coal combustion residues

Sample	2PPF	4PPF	4PPIS	4PPB	2PPS	3PF	2PPB
Hg ⁰ oxidation, %	8.8	2.4	0.0	25±8	26±11	4.3	26±6

An attempt was also made to correlate the chemical composition of the residue samples to their Hg⁰ oxidation capabilities. The residue samples were analyzed for their elemental composition using a Siemens 303 X-Ray Fluorescence (XRF) analyzer. Table 3 outlines the elemental compositions of the residue samples. No direct correlation between the residues' chemical composition and their Hg⁰ oxidation activities was observed.

Table 3. Elemental composition of Illinois coal combustion residues (wt%)

Element	Sample Identification (see Table 1)					
	2PPS	4PPF	2PPF	4PPB	2PPB	3P-F
Na	0.043	0.812	1.01	0.536	0.430	0.457
Mg	0.544	0.499	0.634	0.402	0.496	3.54

Al	0.174	8.22	11.2	6.55	8.4	4.35
Si	1.190	30.8	25.1	26.2	21.4	12.8
P	0.013	0.061	0.057	0.043	0.061	0.048
S	18.4	0.696	1.04	0.142	0.330	6.4
K	0.070	1.42	1.86	1.12	1.58	1.01
Ca	24.9	3.19	2.46	3.81	3.34	19.5
Ti	0.024	0.348	0.451	0.278	0.346	0.114
Mn	0.010	0.041	0.033	0.046	0.037	0.043
Fe	0.151	8.34	8.35	7.24	7.5	4.71
Cu	0.001	0.006	0.016	0.004	0.008	0.005
Sr	0.025	0.018	0.027	0.012	0.021	0.031

Task II (Subtask I): In-flight Hg⁰ oxidation in a natural gas combustion doped with active ICCR, Hg⁰, HCl, SO₂, and NO_x

The objective of natural gas tests was to evaluate the activity of the bottom ash sample (2PPB) during in-flight oxidation of Hg⁰ under a well-controlled combustion condition. The coal combustion flue gas was simulated by injecting Hg⁰, HCl, SO₂, and NO_x, immediately after the flame of the natural gas (temperature of 800 °C). Initially, a duplicate set of blank tests was performed to assess the speciation of injected Hg⁰ in the absence of the bottom ash sample. During the blank experiments, a single isokinetic OH measurement was performed at the last port of the horizontal section of the IFR (200 °C). CO₂, O₂, NO_x, CO, and SO₂ CEM measurements were performed at the first port of the horizontal section (300 °C). CEM measurements are included in Table 4, and mercury speciation measurements are included in Table 5. Mercury concentrations were not corrected for O₂. During the blank tests, consistent O₂, CO₂, NO_x, and SO₂ concentrations were observed. Experimental variability was rather high during the second blank test (see Table 4). Duplicate blank tests indicated an excellent precision (±3.6%) in injecting Hg⁰ into the IFR (see Table 5). Total mercury concentration (27.4±1.0) was in the range of those coal-fired processes with high mercury emissions. About 36% of injected Hg⁰ was converted to oxidized forms (Hg²⁺), even in the absence of the bottom ash sample. A portion of this conversion is believed to be due to the high temperature reactions of HCl and Hg⁰ (800-700 °C; injection location of Hg⁰). Moreover, heterogeneous reactions on the interior surface of the IFR (refractory in the vertical section and stainless steel duct in the horizontal section) may play a role in the conversion of Hg⁰ to Hg²⁺.

Table 4. CEM measurements during the natural gas combustion tests (Subtask I of Task II)

	O ₂ (%)	CO ₂ (%)	NO _x (ppm)	SO ₂ (ppm)	CO (ppm)
1 st blank test	12.9±0.0	4.7±0.0	216±9	1501±101	>5
2 nd blank test	12.8±0.6	4.8±0.1	204±17	1384±440	>5
2PPB injection (1 st test)	13.5±0.1	4.3±0.0	201±16	1517±51	29±2
2PPB injection (2 nd test)	13.6±0.0	4.4±0.0	204±2	1524±28	22±1

Table 5. Mercury speciation (in µg/dscm) and particulate matter (PM, in mg/dscm) measurement during the natural gas tests (Subtask I of Task II).

	blank test	2PPB injection (1 st test)		2PPB injection (2 nd test)	
	isokinetic	isokinetic	non-isokinetic	isokinetic	non-isokinetic
PM	0	420	245	524	313
Hg ⁰	18.0±0.9	13.2	15.2	19.1	21.7
Hg ²⁺	9.43±0.09	6.46	4.11	9.1	8.45
Hg _p	0.02	0.08	0.07	0.12	0.25
Hg _T	27.4±1.0	19.7	19.4	28.3	30.4
% Hg ⁰	65±1	67	79	68	71

Hg⁰: elemental mercury, Hg²⁺: ionic mercury, Hg_p: particulate mercury, Hg_T: total mercury

Following the blank tests, the bottom ash sample (2PPB) was injected into the IFR to assess potential mercury speciation changes. Bottom ash injection location was immediately downstream from the natural gas flame (800 °C). The 2PPB injection rate was set at about 1.8 lb/hr (0.8 kg/hr) to generate a target PM concentration of about 500 mg/dscm in the horizontal section of the IFR. It should be noted that about 20% of injected 2PPB reached the horizontal section, and the rest was removed in the collection bin connecting the vertical and the horizontal section. Isokinetic and non-isokinetic OH sampling (see the previous section) was performed to assess mercury speciation in the presence of an injected bottom ash sample (Table 5). Non-isokinetic sampling suppressed the capture of PM by only a factor of 2, indicating that this sampling technique was only partially successful in eliminating particles from the filter of the OH method.

Comparison between isokinetic and non-isokinetic sampling indicated that the mercury capture by PM (Hg_p) in the horizontal section does not contribute to the total mercury. This bottom ash sample is not a good mercury sorbent. The first set of the test results showed that

the speciation of mercury may have changed across the filter of the isokinetic train, since more Hg^{2+} was observed in the isokinetic train. The change in mercury speciation, however, was not observed during the second set of tests (duplicates). During each test set, the total mercury concentration was relatively consistent for both trains. The variability in total mercury between the two duplicate test sets was significant (19.5 vs 29.4 $\mu\text{g}/\text{dscm}$) but in the range of the blank test total mercury concentration (27.4 $\mu\text{g}/\text{dscm}$). This variation could be due to daily changes in Hg^0 injection rate. Comparison between 2PPB injection and blank tests showed no significant change in mercury speciation as the result of injection of the bottom ash sample, 2PPB. This sample was not active for in-flight oxidation of Hg^0 to Hg^{2+} . Task I results, discussed above, showed that 2PPB oxidized about 26% of Hg^0 in a fixed-bed reactor. This indicates that this bottom ash sample is active in the oxidation of Hg^0 only in a fixed-bed flow mode.

Task II (Subtask II): Mercury speciation modification in the combustion of an Illinois coal

The initial objective of this subtask was to investigate the effect of injection of the active ICCRs on the change in mercury speciation in the combustion of an Illinois coal that mostly produces Hg^0 in its flue gas. It should be noted that combustion of most bituminous coals (such as Illinois coals) produces oxidized mercury in the flue gas. A literature survey was performed to identify an appropriate Illinois coal for pilot-scale combustion studies in this subtask. Coal sample IBC-110 (see the Illinois Basin Coal Sample program; www.icci.org) was chosen for this task. This coal has a heating value of 13,076 Btu/lb and a moisture, volatile matter, fixed carbon, and ash content of 10.6, 39.5, 50.7, and 9.7 wt%, respectively. Its total sulfur content is 4.5% and it contains 2.12 ppmw of mercury.

This coal had to be further pulverized to sizes below 90 μm before combustion in the IFR. IBC-110 was combusted in the IFR at a firing rate of about about 80,000 Btu/hr (18.6 kW) to determine its flue gas mercury speciation and to obtain CEM data. Temperature profiles across the IFR during the coal combustion tests were identical to those observed during the natural gas combustion tests. Combustion of this coal was performed in duplicate. CEM data are listed in Table 6. A lower O_2 concentration was recorded during the second coal combustion test; this resulted in higher CO_2 and SO_2 concentrations. Isokinetic and non-isokinetic OH sampling was performed to determine the mercury speciation. These data are included in Table 7. A number of problems occurred during the first coal combustion test's OH sampling. Sampling trains' filters slid out of their place and induced contamination of the impinger solutions with fugitive coal fly ash particles. Table 7 contains the OH sampling results from the second set of coal combustion tests.

Table 6. CEM measurements during the Illinois coal (IBC-110) combustion tests (Subtask II of Task II)

	O_2 (%)	CO_2 (%)	NO_x (ppm)	SO_2 (ppm)	CO (ppm)
1 st coal test	11.9±1.3	8.1±1.0	378±58	1764±248	19.5±8.5

2 nd coal test	9.7±1.3	10.3±1.0	NP	2464±420	18.8±6.9
Hg ⁰ injection coal (1 st test)	NP	NP	NP	2633±450	NP
Hg ⁰ injection coal (2 nd test)	12.4±0.8	7.9±0.7	NP	2450±226	NP

NP: not performed, CEM was not operational

Once again, the non-isokinetic sampling technique was only partially capable of suppressing the capture of the PM across the filter of the method. Other gas-particulate separation techniques need to be developed for sampling in a high PM environment, upstream of PM control devices. The total mercury concentrations from both trains were in good agreement. The majority of mercury was in the oxidized form (either gas-phase or captured by PM). The combination of a PM control device [electrostatic precipitator (ESP) or baghouse] and a wet or dry FGD will exhibit better than 90% mercury removal in the combustion of this Illinois coal (IBC-110). Very little Hg⁰ was found in this coal flue gas. It was determined that the injection of an active residue in this flue gas would not offer any additional benefits in terms of increasing the baseline oxidized mercury content, thus it was decided not to proceed with the injection of 2PPB in the furnace.

It can be hypothesized that the native fly ash of this coal may be very active in oxidizing Hg⁰. To test this hypothesis, the flue gas of this coal was spiked with additional Hg⁰ to assess the extent of oxidation of doped Hg⁰. These tests were run in duplicate and the CEM and mercury results are summarized in Tables 6 and 7, respectively. Most of the CEMs were not operational during these two test days. Measured SO₂ concentrations, however, indicated identical combustion conditions as previous coal combustion test days. The total mercury concentrations obtained with these two trains (during the first Hg⁰ injection test) did not agree with each other (Table 7). About 20-25 µg/dscm of additional Hg⁰ was injected into the furnace. According to the results (isokinetic train), only about 5 µg/dscm remained as Hg⁰. Thus, 83% of the injected Hg⁰ was oxidized by the native fly ash of this coal. These results once again showed the activity of this fly ash in oxidizing Hg⁰. During the second Hg⁰ injection tests, consistent total mercury concentrations were obtained. These total mercury concentrations agree with the isokinetic train performed on the first set of the Hg⁰ injection tests. The non-isokinetic train on the first set of the tests remains unexplained. In summary, the majority of the injected Hg⁰ (more than 80%) was converted to oxidized forms, indicating the activity of this fly ash. Particulate mercury concentrations were quite variable during these tests, demonstrating the difficulty in mercury sampling in a high PM environment (inlet of baghouse or ESP). The gas-phase speciation could have been affected by the presence of the filter, but since both isokinetic and non-isokinetic sampling indicated the same order-of-magnitude of speciation, one can draw the above conclusion on the activity of this fly ash.

Table 7. Mercury speciation (in µg/dscm) and particulate matter (PM, in mg/dscm)

measurement during the Illinois coal (IBC-110) combustion tests (Subtask II of Task II).

	2 nd coal test		Hg ⁰ injection coal (1 st test)		Hg ⁰ injection coal (2 nd test)	
	isokinetic	non-isokinetic	isokinetic	non-isokinetic	isokinetic	non-isokinetic
PM	5205	3181	5365	2560	4931	3080
Hg ⁰	0.49	0.78	4.52	5.89	5.51	7.65
Hg ²⁺	7.62	4.54	27.5	20.4	34.5	26.7
Hg _p	1.66	2.39	11.2	4.99	1.9	5.8
Hg _T	9.77	7.71	43.2	31.3	41.9	40.2
% Hg ⁰	5	10	10	19	13	19

Hg⁰: elemental mercury, Hg²⁺: ionic mercury, Hg_p: particulate mercury, Hg_T: total mercury

It was decided to further confirm the activity of the IBC-110 coal fly ash. Additional experiments (beyond the original scope of work) were performed to assess the activity of this fly ash during duct injection and also in a packed bed mode (filter of the OH method; baghouse simulation). The Illinois coal was combusted for more than 20 hours and approximately 3 lbs of the fly ash was collected from the IFR's baghouse. Natural gas combustion tests (doped with Hg⁰/HCl/NO_x/SO₂) were performed to assess the activity of this fly ash. Two different techniques were used to estimate the activity of IBC-110 fly ash in the oxidation of injected Hg⁰ into the natural gas combustion flue gas. In the first technique, the filter of the mercury speciation sampling method (OH) was pre-coated with an amount of fly ash that would normally be collected during an isokinetic sampling (10 g). In the second technique, the IBC-110 fly ash was injected into the vertical section of the furnace and the isokinetic/non-isokinetic OH sampling was performed in the horizontal section of the IFR. The purpose was to distinguish between fixed-bed (first technique) and in-flight oxidation of Hg⁰ by IBC-110 fly ash.

The results of these sets of tests are included in Tables 8 (CEM results) and 9 (mercury speciation results). The preload filter tests were performed in duplicate using one isokinetic sampling train, and the IBC-110 coal fly ash injection test was performed as one single test. Hg⁰ was doped at 20-25 µg/dscm levels. As can be seen from Table 9, most of the injected Hg⁰ was oxidized when the filter of the OH method was preloaded with the IBC-110 fly ash. Injection of this fly ash did not oxidize the doped Hg⁰ in the vertical and the horizontal duct section of the IFR. It appears that this fly ash was active in catalyzing the oxidation of Hg⁰ only in a fixed-bed flow mode (filters and baghouses) and not when injected into the duct. This indicates that Hg⁰ oxidation in the duct section of the IFR is an external mass transfer controlled process. The technology of injecting IBC-110 fly ash will be successful if the

FGD unit of a power plant is preceded by a baghouse and not an ESP. Changing the duct injection system to minimize mass transfer processes can also be considered as a viable technology.

Table 8. CEM measurements during the natural gas combustion and IBC-110 coal fly ash injection/preload filter tests

	O ₂ (%)	CO ₂ (%)	NO _x (ppm)	SO ₂ (ppm)	CO (ppm)
1 st preload test	7.4±0.5	7.9±0.3	221±12	2016±117	<5
2 nd preload test	6.8±0.2	8.0±0.1	27415	1551±64	<5
IBC-110 fly ash injection test	8.7±0.2	6.9±0.1	234±9	1564±51	<5

Table 9. Mercury speciation (in µg/dscm) and particulate matter (PM, in mg/dscm) measurement during the natural gas combustion and IBC-110 fly ash injection/preload filter tests.

	preload tests (in duplicates)	IBC-110 fly ash injection test	
	isokinetic	isokinetic	non-isokinetic
PM	NA	123	107
Hg ⁰	7.0±1.6	19.0	21.4
Hg ²⁺	14.0±3.9	3.5	4.7
Hg _p	1.0±0.0	>0.01	>0.01
Hg _T	22±2.3	22.5	26.1
% Hg ⁰	33±6	84	82

Hg⁰: elemental mercury, Hg²⁺: ionic mercury, Hg_p: particulate mercury, Hg_T: total mercury
NA: Not Applicable

CONCLUSION AND RECOMMENDATIONS

Research was conducted to characterize and modify mercury speciation (through conversion of Hg⁰ to HgCl₂) in Illinois coal combustion flue gas so that a mercury control strategy can be implemented in conventional FGD units. A number of ICCRs were found to be rather active in the conversion of Hg⁰ to HgCl₂ in a fixed-bed system. The active residues were a bottom ash and a scrubber sludge sample from one power plant and a bottom ash from

another power plant. Injection of these residues upstream of a baghouse in a coal-fired utility boiler may enhance the formation of oxidized mercury, which can be easily removed by wet or dry FGD systems downstream of the baghouse. One of the active bottom ash samples was injected in a pilot-scale combustor while burning natural gas doped with appropriate amounts of Hg^0 , HCl , NO_x and SO_2 . Injection of this bottom ash sample did not contribute to the background oxidation of Hg^0 (gas-phase oxidation), indicating that in-flight oxidation of Hg^0 in the pilot-scale combustor may be mass transfer limited. Subsequently, a representative Illinois coal was combusted in the pilot-scale combustor. As is the case with most eastern bituminous coals, the combustion flue gas of this coal was dominated by oxidized forms of mercury (most probably HgCl_2). Additional Hg^0 was doped into this flue gas and significant oxidation of Hg^0 was observed. It was determined that the fly ash generated from combustion of this coal is very active in oxidation of Hg^0 . Combustion of this coal in power plants that are equipped with FGD units will provide a better than 90% mercury emission control. Additional pilot-scale tests were performed to investigate the activity of the Illinois coal fly ash. It was determined that packed-bed flow modes, such as those observed in baghouses, are required to catalytically oxidize Hg^0 using this coal's fly ash. As was the case with other ICCRs, injection of this fly ash in those power plants equipped with baghouse and FGD units will offer an enhanced mercury control technology. Based on these observations the following recommendations can be made:

- Injection of selected Illinois coal combustion bottom and fly ashes upstream of a baghouse can significantly change the speciation of mercury from elemental to oxidized forms. Oxidized forms can be easily removed using wet or dry FGDs located downstream of a baghouse.
- Combustion of selected Illinois coals will result in a flue gas that is dominated with oxidized forms of mercury. Installation of a FGD unit in these power plants should provide a better than 90% mercury emission control.
- It is likely that blending of western subbituminous coals (mostly Hg^0 in the flue gas) with selected Illinois coals can result in a change in mercury speciation from the elemental to oxidized form, thereby easing removal using an FGD unit.
- The ability of co-firing Illinois coals with western coal to effect Hg oxidation and subsequent removal should be further developed and demonstrated.

DISCLAIMER STATEMENTS

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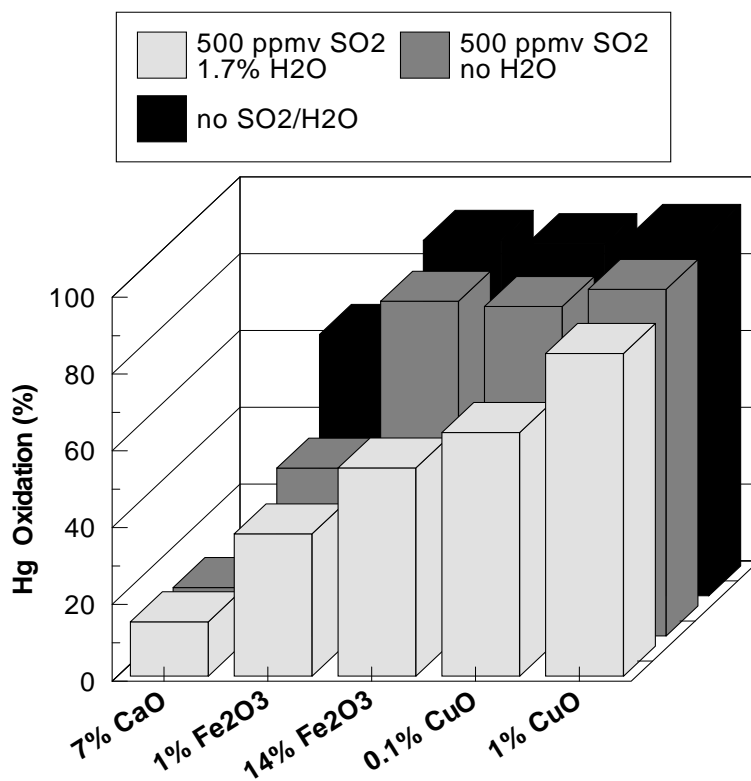


Figure 1. Effects of SO₂ and H₂O on the steady-state oxidation of fly ashes at the presence of 50 ppm HCl. Indicated amounts of additives were added to the alumina/silica inert base.

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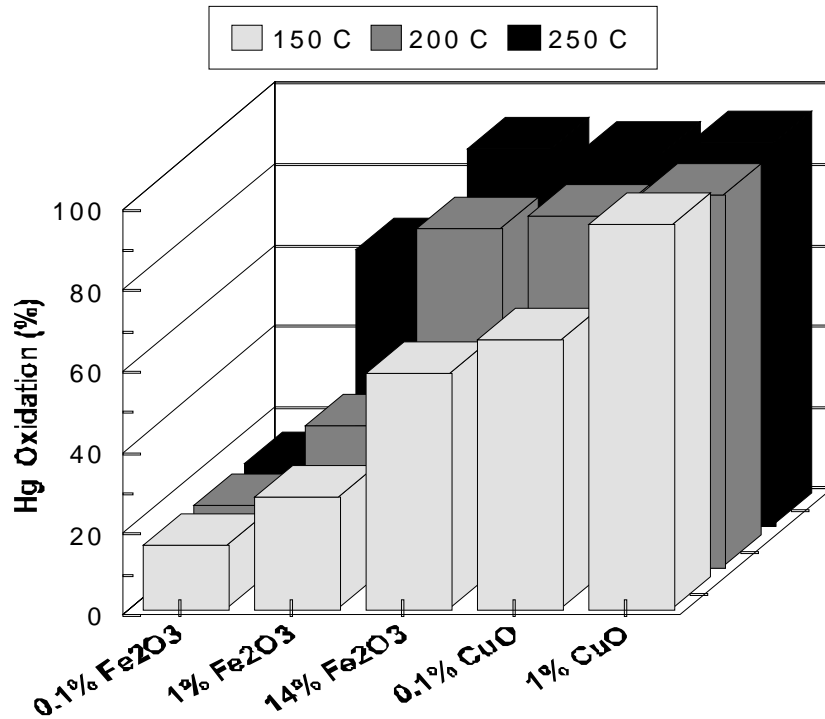


Figure 2. Effects of coal fly ash components/compositions and temperature on the steady-state heterogeneous oxidation of Hg^0 across model fly ashes in the presence of 50 ppm HCl and the absence of SO_2 and H_2O . Compounds were added to alumina/silica inert base.

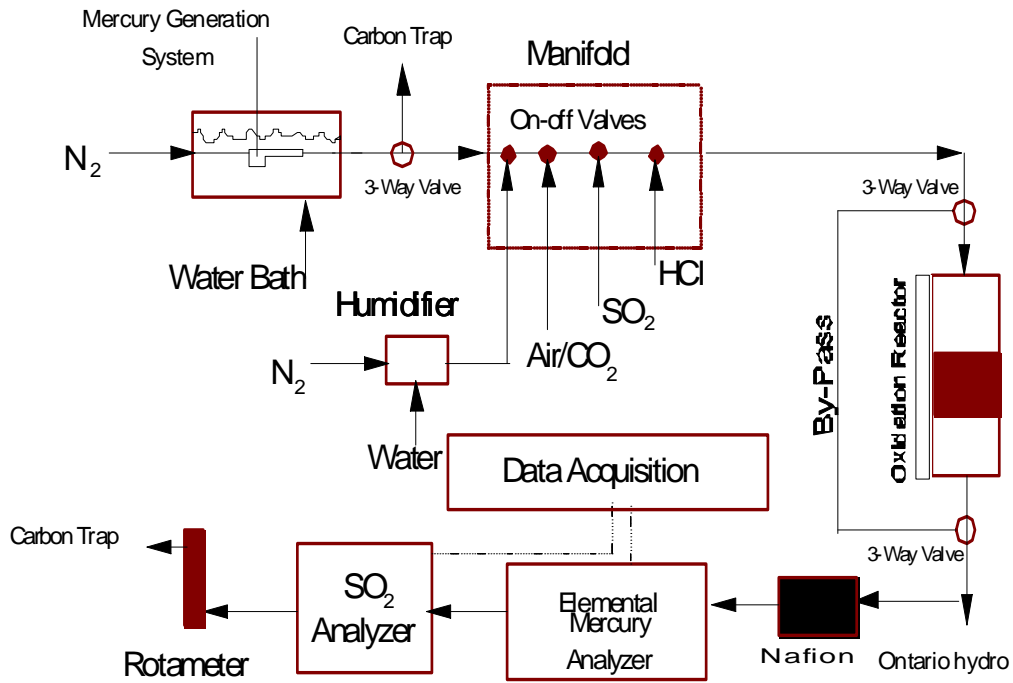


Fig
3. Schematic of the elemental mercury (Hg⁰) oxidation reactor system.

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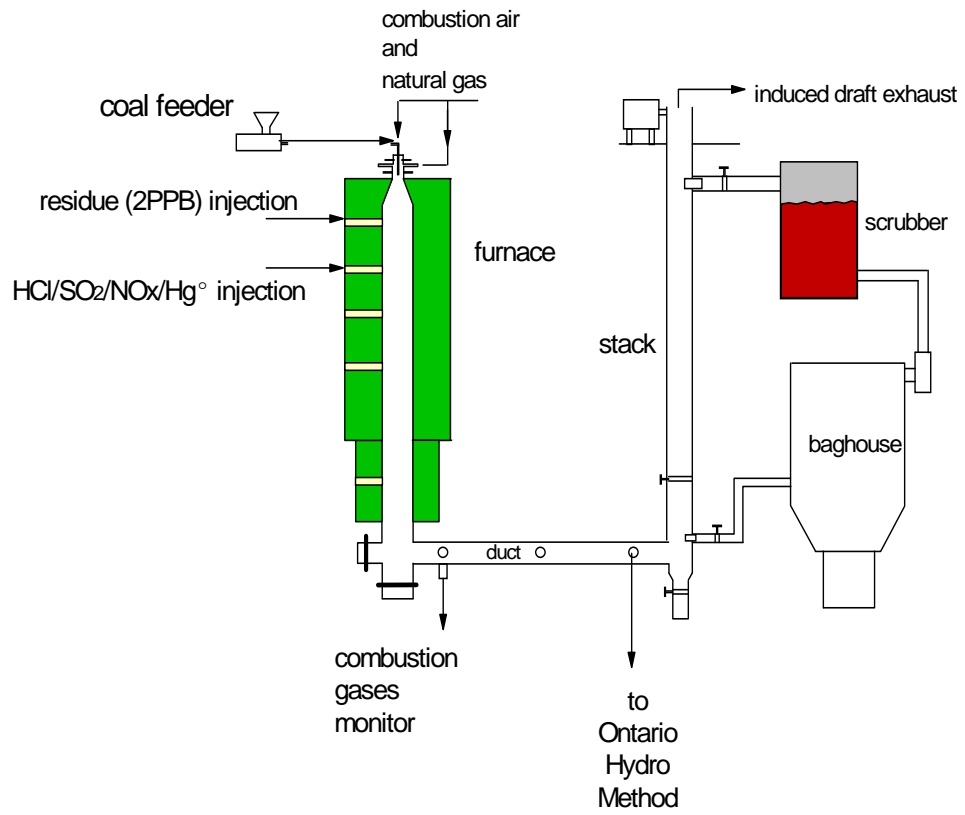


Figure 4. Schematic of the Innovative Furnace reactor (IFR).