

Measurement of Mercury in Flue Gas Based on an Aluminum Matrix Sorbent

Juan Wang, Wei Xu, Xiaohao Wang, and Wenhua Wang

School of Environmental Science & Engineering, Shanghai Jiao Tong University, 800 Dongchuan Road, Shanghai 200240, China

Received 24 June 2011; Accepted 13 October 2011

Academic Editor: Richard Brown

The measurement of total mercury in flue gas based on an economical aluminum matrix sorbent was developed in this paper. A sorbent trap consisted of three tubes was employed to capture Hg from flue gas. Hg trapped on sorbent was transferred into solution by acid leaching and then detected by CVAAS. Hg adsorbed on sorbent was recovered completely by leaching process. The 87.7% recovery of Hg in flue gas by tube 1 and tube 2 was obtained on the equipment of coal combustion and sampling in lab. In order to evaluate the ability to recover and accurately quantify Hg⁰ on the sorbent media, the analytical bias test on tube 3 spiked with Hg⁰ was also performed and got the average recovery of 97.1%. Mercury measurements based on this method were conducted for three coal-fired power plants in China. The mercury in coal is distributed into bottom ash, electrostatic precipitator (ESP) ash, wet flue gas desulfurization (WFGD) reactant, and flue gas, and the relative distribution varied depending on factors such as the coal type and the operation conditions of plants. The mercury mass balances of three plants were also calculated which were 91.6%, 77.1%, and 118%, respectively. The reliability of this method was verified by the Ontario Hydro (OH) method either in lab or in field.

KEYWORDS: Mercury, flue gas, measurement method, sorbent trap, mass balance

1. INTRODUCTION

Mercury is considered as a true global pollutant due to its toxicity, volatility, persistence, and long-range transport in the atmosphere. Global Hg emissions were reviewed for the year 2000 in detail by Pacyna et al. [1]. And it was observed that two thirds (1.46 kt) of the total atmospheric emission (2.19 kt) originate from coal combustion. Increasing awareness of the mercury emissions from coal-fired flue gas has led to many efforts to develop strategies and technologies to control Hg emission. Hg measurement in flue gas in a more convenient way has been paid more attention to.

At present, many methods for sampling and quantifying Hg in flue gas are used. Several standard EPA sampling procedures (29, 101A, and OH methods) have been widely recommended for the determination of particulate and gaseous mercury emissions from stationary sources [2–4]. These EPA methods are based on isokinetic sampling of the flue gases at a suitable sampling point using a filter collection system for particulates firstly. The sampled gases then pass through a set configuration of impingers containing liquid absorbents such as KCl, HNO₃/H₂O₂, and KMnO₄/H₂SO₄ to trap any mercury present in the sample gases. Some other aqueous oxidative solutions were tested by Shendikar et al. [5], such as 10% H₂O₂, acidified 10% H₂O₂, 0.2 M (NH₄)₂S₂O₈, 0.2 M (NH₄)₂S₂O₈ + 0.025 M AgNO₃, and 1.5% KMnO₄ in 10% H₂SO₄. Good collection efficiencies for Hg⁰ by using more than one impinger were exclusively obtained by the last two mixed solutions. K₂Cr₂O₇ (4%) in 4 M HNO₃ solution was used to sample Hg in flue gas of a coal-fired power plant by Meij [6].

Furthermore, many solid mediums were used for the collection of Hg from flue gas. In Frontier Geosciences Mercury Speciation Method [7], flue gas passes through a set of two KCl/soda lime traps and two iodated carbon traps. Hg²⁺ and Hg⁰ are collected by the two different sorbents, respectively. In MIT's Solid Sorbent Method [8], flue gas passes through two activated charcoal sorbent traps connected in series. The traps capture Hg²⁺ as well as Hg⁰. In HEST Method [9], carbon-impregnated filters are used to adsorb gas phase Hg. And activated carbon treated with iodine or some other halogen has been applied to capture total Hg in flue gas in EPA method 30B [10] and method Appendix K [11].

These methods could be classified in two main groups: methods to employ impingers with oxidant solutions and methods based on solid sorbents. Although the latter is easier to handle than the former, it was found in most of the solid sorbents that the collection efficiency of Hg⁰ was decreased with the increasing temperature of flue gas and the increasing level of SO₂ [12–14]. Thus, a number of disadvantages in the methods based on solid sorbents have to be improved.

In the previous work [15, 16] in our lab, a series of aluminum-matrix-based sorbents were prepared to convert Hg⁰ to Hg²⁺ by catalytic oxidation and then absorb it. Some characteristics of the sorbents such as the size of the particles, crystal structures, binding energies, Brunauer-Emmett-Teller- (BET-) specific surface area (S_{BET}), and pore size distribution were analyzed, respectively. The catalytic and adsorption tests were carried out with a flow of simulated flue gas (consisted of N₂, O₂, SO₂, NO, and HCl). These sorbents have shown a good SO₂ antipoisoning ability and a stable oxidation and adsorption abilities during the change of temperatures. Furthermore, they can be regenerated by thermal decomposition above 500°C easily. This feature made the application of these sorbents more economical than carbon-based sorbents, especially in China.

Based on these materials, the sorbent AL-CCI (CuCoO₄ + NH₄Cl loaded on γ -Al₂O₃) was selected as the collection medium to capture Hg in flue gas. Sampling and detection approach was established with small system for coal combustion and flue gas sampling in this work in lab and was applied in three power plants in China. The overall mass balances have been calculated to evaluate the efficiency of this method and the reliability has been verified with OH method.

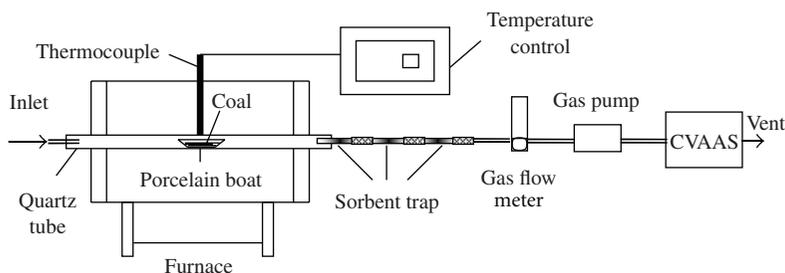
2. EXPERIMENT

2.1. Materials and Determine Method

CuCoO₄ + NH₄Cl loaded on γ -Al₂O₃ (AL-CCI) was prepared by impregnating AL-C (CuCoO₄ loaded on γ -Al₂O₃) with 30 wt% NH₄Cl solutions before the decomposition, dried at 60°C, and then calcined

TABLE 1: Major components of BCR-40 and coal sample (dry air basis).

Coal type	Moisture (%)	Ash (%)	Volatile (%)	N (%)	C (%)	S (%)	Cl (%)	[Hg] _{mean} (mg/kg)
BCR-40	1.20	16.1	13.0	1.20	70.6	1.28	0.230	0.354 ± 0.003
Coal	2.97	10.4	13.2	1.03	83.1	1.41	0.110	4.61 ± 0.04

**FIGURE 1:** Schematic diagram of the coal combustion and sampling system.

at 400°C for 3 h. AL-C was prepared by the thermal decomposition of Cu-nitrate/Co-nitrate mixtures loaded on γ -Al₂O₃. Both nitrates were mixed in Co/Cu atomic ratio of 1. The mixtures were dissolved in water to form the mixtures solution. γ -Al₂O₃ was impregnated by the mixtures solution in proportion to 20 wt% nitrate loading values and heated to 230°C, the temperature at which nitrogen oxides were evolved. The heterogeneous mass obtained was ground in a mortar and heated for 5 h at 400°C.

Coal sample from the south-west China was used in lab research after the pretreatment of grinding and sieving. They were digested in a V₂O₅-HNO₃-H₂SO₄ solution firstly and then the digestate solution was determined by a cold vapor atomic adsorption spectrometry mercury analyzer (CVAAS, SG-921, Jiangfen Ltd.). A standard reference material (BCR-40) with a certified value for mercury (0.35 ± 0.06 mg/kg) was introduced for quality control. All chemicals were of analytical grades. The major components of BCR-40 and coal are listed in Table 1.

The mercury concentration of BCR-40 was detected in an average value of 0.354 ± 0.003 mg/kg ($n = 5$) with the relative error <2% compared to certified value. Therefore, the determination course was reliable.

2.2. Sample Collection

Mercury in flue gas was collected using a sorbent trap with three tubes connected by Teflon sleeves. All the tubes were made by the quartz tube (50 mm in length with an outer diameter of 10 mm and an inner diameter of 7.5 mm) containing approximately 1 g sorbent which was sandwiched and fixed by the quartz cotton. Among them, the tube 1 was designated for primary capture of gaseous Hg. The tube 2 was designated as a backup to check vapor phase Hg⁰ breakthrough. For the third tube, a known mass of Hg⁰ was spiked to perform the analytical bias test and spiked Hg⁰ recovery study. Each sorbent trap was marked with an identified number for tracking.

In lab research, mercury collection experiment was carried out on the equipment of coal combustion and sampling shown in Figure 1. A porcelain boat with the coal was put into a quartz tube (650 mm in length with an outer diameter of 36 mm and an inner diameter of 32 mm), which crossed through a clam-shell furnace. Figure 2 presents that Hg emission was started from 150°C in furnace and the maximum emission was got during 240–250°C. In Figure 3, it can be seen that most all of volatile mercury in coal was released into flue gas at 600°C. Therefore, the temperature in furnace was maintained at 600°C. The gas pump was turned on before trapping Hg and adjusted the gas flow meter at an appropriate flow rate (0.3–0.5 L/min). Then, coal-fired flue gases were extracted through the sorbent trap. Sampling time was

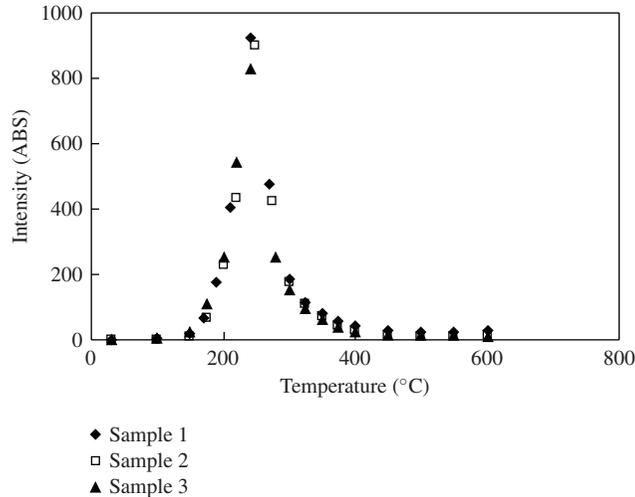


FIGURE 2: Hg emission from coal with the increased temperature in furnace.

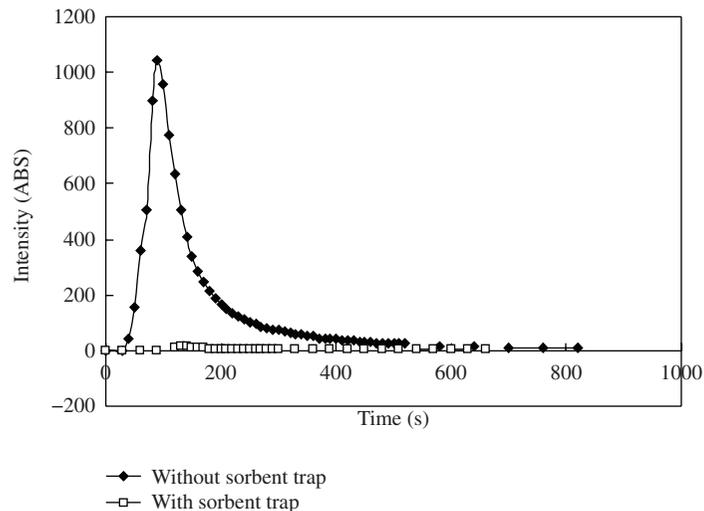


FIGURE 3: Hg emission from coal with and without sorbent trap at 600°C.

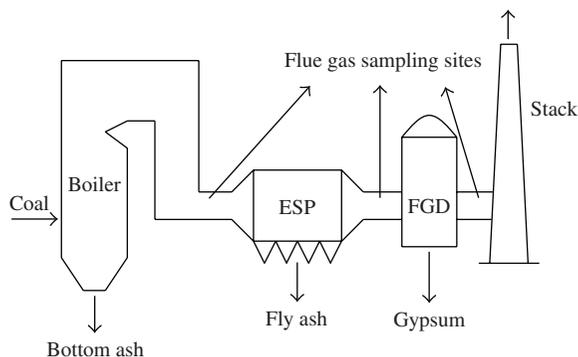
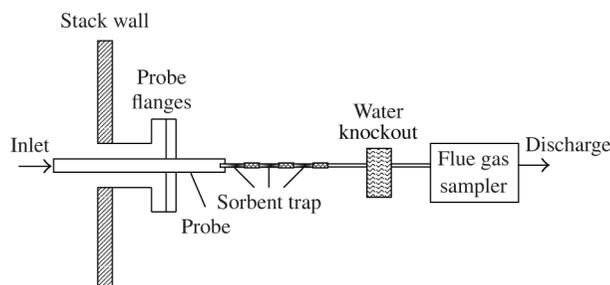
remained for 5 minutes. The CVAAS at the end of the equipment in Figure 1 was connected in order to monitor any mercury in gas path. After trapping, the mercury content in coal ash was tested with the same procedure of coal, and the average mercury concentration (1.50 ± 0.11 mg/kg, $n = 15$) was obtained.

Field mercury emission measurements based on this method were practiced in three coal-fired power plants in China. Table 2 and Figure 4 present the detailed configurations and sampling sites during the course of flue gas in the three power plants tested in this study.

As shown in Figure 5, the test gases were sampled from the flue gas stream at an appropriate flow rate (0.3–0.5 L/min) through a heated probe which prevented the condensation to occur before the gas entered the sorbent trap(s). A water knockout after the sorbent trap was placed to prevent the water in sample line from entering the flue gas sampler (TH-600, Tianhong, China). The samples were simultaneously taken from both the inlet and outlet of the pollutants control devices, for example, ESP and WFGD, as shown in Figure 2. According to the results of pretest, 2 hours were enough for the gas phase mercury detection. After the sampling, each tube of the sorbent trap was immediately placed into Hg-free containers for subsequent laboratory detection. The characteristics (such as gas temperature, static pressure, and gas velocity) and

TABLE 2: Power plant configurations.

Power plant no.	Boiler type	Coal type	Capacity (Mw)	Pollution control device
1	Pulverized coal	bituminous coal	98	ESP + WFGD
2	Supercritical	bituminous coal	758	ESP + WFGD
3	Pulverized coal	bituminous coal	100	ESP + WFGD

**FIGURE 4:** Simultaneous power plant sampling locations.**FIGURE 5:** Sampling train in field.

main components (such as SO_2 , NO_x , and O_2) of the flue gas were determined by the flue gas analyzer (350-XL, Testo, German) during the sampling period. Solid samples, including the pulverized coal, bottom ash, fly ash captured in ESP, and gypsum in the WFGD, were taken after half-hour Hg trapping. The sampling was conducted two or three times for each power plant to obtain the parallel results and reduce uncertainties. The data obtained from tests at the same conditions were averaged to get the final results.

2.3. Detection of Hg in Flue Gases

The individual tube of the the sorbent trap and their respective components were detected separately. Sorbent media, quartz cotton, and fly ash deposited on the quartz cotton in each tube were moved into a glass funnel, which was covered with a piece of filter paper. Then, they were eluted by 50 mL 4 mol/L HCl-saturated KCl solution and the captured mercury was transferred into liquid solution. The mercury concentrations were measured on the CVAAS (SG-921, Jiangfen, China). The amount of Hg contents in tube 1 and tube 2 reflected the total mercury concentration in the flue gas. From the results detected in tube 3 spiked with known mass of Hg^0 , the recovery and performance of the combined sampling and analytical approach could be verified. The solid samples taken from the power plants were measured by a Zeeman Mercury Spectrometer (RA-915⁺, Lumex, Russia). All the instruments were calibrated before the samples analyses.

TABLE 3: Recovery of spiked Hg⁰ in tube 3 in lab test ($n = 3$).

Coal consumption (g)	Hg emission from coal (μg)	Hg ⁰ spiked in tube 3 (μg)	Hg ⁰ recovered from tube 3 (μg)	Average recovery (%)
0	0	0.25	0.243 \pm 0.002	97.3
1.0	3.11	0.25	0.247 \pm 0.012	98.7
2.0	6.22	0.25	0.241 \pm 0.020	96.2
3.0	9.33	0.25	0.224 \pm 0.003	89.7
4.0	12.44	0.25	0.263 \pm 0.041	105
5.0	15.55	0.25	0.238 \pm 0.036	95.4

Hg emission from coal (μg) = (4.61 – 1.50) ($\mu\text{g/g}$) \times coal consumption (g). Average recovery (%) = [Hg⁰ recovered from tube 3 (μg) \div 0.25 (μg)] \times 100%.

3. RESULTS AND DISCUSSION

3.1. Recovery of Hg Analysis in Lab

3.1.1. Recovery of Spiked Hg⁰ in Tube 3 of the Sorbent Traps

The ability to recover and accurately quantify Hg⁰ on the sorbent media was evaluated by performing the analytical bias test on the tube 3 spiked with Hg⁰. Mercury concentrations in the tube 3 were analyzed using the same procedure above. In Table 3, the average Hg⁰ recovery of 97.3% in tube 3 was got without coal combustion. The specific verification of the performance of the combined sampling and detection approach was also provided by performing the spiked Hg⁰ recovery test. As shown in Table 3, the average Hg⁰ recovery of tube 3 varied from 89.7% to 105% (averaged 97.0%) with increasing of coal consumption. It is demonstrated from the aforementioned results that the sorbent media in tube 1 and tube 2 possessed efficient capacity of mercury adsorption and there was no vapor phase Hg breakthrough. Also spiked Hg⁰ in tube 3 was not affected by the flue gas passed by. Therefore the approach of sampling and detection in this study was validated in summary.

3.1.2. Recovery of Hg in Flue Gas

Hg recovery in tube 1 and tube 2 of the traps after sampling was calculated to evaluate the feasibility and accuracy of this method. In Table 4, the Hg content in original sorbent media before sampling was under the lowest test limitation, which indicated that Hg was thoroughly removed from the sorbent during the baking procedure. And as the increasing of coal consumption, Hg content in tube 1 increased simultaneously with a good value of the square of the linear correlation coefficient (i.e., $R^2 = 0.998$) as shown in Figure 6. However, Hg content in tube 2 presented irregular and negligible compared to that of tube 1. The average recovery of tube 1 and tube 2 varied from 83.0% to 94.3% (averaged 87.7%). Due to the complex composition of flue gas, the adsorption of Hg by the sorbent was affected by many uncertain factors, such as fluctuations in coal, ash, flue gas flow rates, sampling uncertainty, and measurement uncertainty. Yokoyama et al. [17] considered that the results between 70 and 130% are acceptable due to the difficulties in estimating each uncertainty.

3.1.3. Reliability Verification of Flue Gas Measurements with OH Method in Lab

In order to verify the reliability of flue gas measurements performed by the new sorbent, the approved method, OH method has been used simultaneously in lab. As shown in Table 5, the Hg measurement results of this method were closed to those of OH method. The relative errors of the results between two methods

TABLE 4: Recovery between Hg trapped in flue gas and Hg emission from coal ($n = 3$).

Coal consumption (g)	Hg emission from coal (μg)	Hg trapped in tube 1 (μg)	Hg trapped in tube 2 (μg)	Average recovery (%)
0	0	Not detected	Not detected	Not detected
1.0	3.11	2.92 ± 0.03	0.017 ± 0.003	94.3
2.0	6.22	5.56 ± 0.10	0.052 ± 0.000	90.3
3.0	9.33	7.72 ± 0.16	0.023 ± 0.002	83.0
4.0	12.44	10.8 ± 0.2	0.026 ± 0.003	86.9
5.0	15.55	13.0 ± 0.6	0.036 ± 0.004	84.0

Average recovery (%) = [Hg recovered from tube 1 and tube 2 (μg) \div Hg emission from coal (μg)] \times 100%.

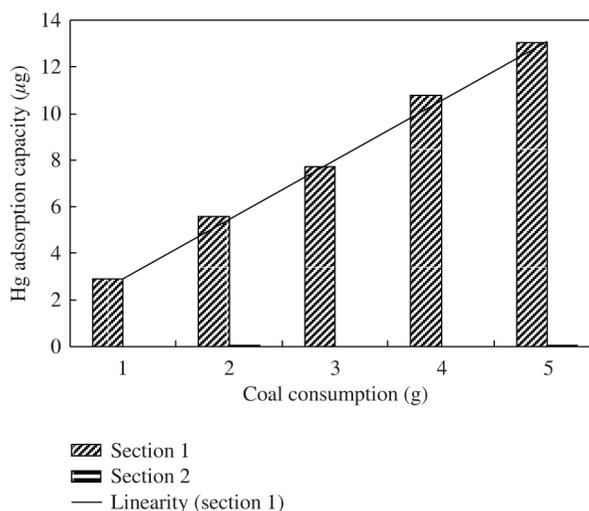


FIGURE 6: Distribution of Hg in tube 1 and tube 2.

were less than 5% in different Hg concentrations in flue gas, which verified that this measurement method of Hg in flue gas was reliable.

3.2. The Practice of the Analysis Method in Power Plants

In order to evaluate the efficiency of this method, the processes of field mercury emission measurements have been practiced in three coal-fired power plants in China. In plant-3, the OH method has been used simultaneously to get the reference data.

3.2.1. Coal Analyses

The coals consumed in three power plants were from different areas and their major components of these coals are listed in Table 6. The mercury contents in three coals are 0.161, 0.064, and 0.225 mg/kg respectively, which agree with mercury contents of 0.01–1.0 mg/kg in Chinese coals [18].

3.2.2. Distribution of Mercury

During the process of coal stream in power plant, the mercury in coal is distributed into bottom ash, ESP ash (fly ash), FGD reactant (gypsum), and flue gas. The relative distribution of Hg and the Hg concentrations

TABLE 5: Comparison of Hg measurements in flue gas between the new sorbent method and OH method in lab ($n = 3$).

Coal consumption (g)	Hg emission from coal (μg)	Recovered Hg (μg)		Average recovery (%)	
		This method	OH method	This method	OH method
1.0	3.11	2.88 ± 0.07	2.81 ± 0.11	92.6	90.4
3.0	9.33	8.01 ± 0.12	8.21 ± 0.23	85.9	88.0
5.0	15.55	13.2 ± 0.5	13.8 ± 0.6	84.8	88.7

TABLE 6: Major components of coals in three power plants (dry air basis).

Power plant no.	Volatile (%)	Ash (%)	Moisture (%)	S (%)	N (%)	Cl (mg/kg)	Hg (mg/kg)
1	37.3	31.6	8.50	0.57	0.79	628	0.161
2	34.9	11.6	3.43	0.54	0.86	472	0.064
3	33.0	23.4	4.40	0.92	0.99	663	0.225

in solid/gas samples in three power plants are summarized in Table 7. These values were the average of the data which varied depending on factors such as the coal type and the operation conditions of plants.

3.2.3. Overall Mass Balance of Mercury

Mercury mass balances were estimated and the results are shown in Table 8. The operation parameters such as coal/lime feeding rate, bottom ash production rate, fly ash, and gypsum generation rate were provided from operational records by power plants.

The mercury mass balances were 91.6%, 77.1%, and 118% for the three power plants, respectively. Some uncertainties, which mainly come from the inhomogeneity of the components in the coal/ash/gypsum, slight variations of boiler operating conditions, and the errors of sampling and chemical analysis procedures, will affect the accuracy of the mass balance estimation. If the mass balance closures are between 70% and 130%, the results are acceptable [17]. All of the results of the three power plants were within this acceptable range, which evidence that the measurement process of Hg in flue gas was practicable. Some other previous investigations are in relatively similar ranges of recovery. Zhang et al. [19] calculated the Hg mass balances of six power plants, which varied from 100.3% to 139.5%. Yokoyama et al. [17] estimated that the mass balances of Hg for combustion of three type coals were between 89% and 138%. The recovery rates of 82% and 99% were referred by Lee et al. [20].

3.2.4. Reliability Verification of Flue Gas Measurements with OH Method in Plant-3

In plant-3, the Hg measurements in flue gas were performed by the new sorbent method and OH method simultaneously. As shown in Table 9, the Hg concentrations in flue gas measured through two methods were closed at each sampling site. Although the relative errors between two methods in field were a little higher compared to those in lab, the results got through the new sorbent method were considered acceptable and reliable due to the complex ingredients in real flue gas.

TABLE 7: Relative distribution of Hg and the mean concentrations of Hg in solid/gas samples in three power plants ($n = 3$).

Plant no.		1	2	3
Distribution	Bottom ash (%)	0.110	1.00×10^{-2}	4.00×10^{-2}
	ESP (%)	4.30	2.17	8.75
	WFGD (%)	58.0	2.21	87.1
	Stack (%)	29.2	72.7	21.8
Concentration	Bottom ash ($\mu\text{g}/\text{kg}$)	6.96 ± 1.16	0.420 ± 0.996	3.25 ± 3.86
	Fly ash ($\mu\text{g}/\text{kg}$)	30.6 ± 5.50	8.50 ± 1.31	92.2 ± 28.6
	Gypsum ($\mu\text{g}/\text{kg}$)	156 ± 6.46	6.22 ± 1.86	1560 ± 74.3
	Stack flue gas ($\mu\text{g}/\text{m}^3$)	2.27 ± 0.58	4.29 ± 0.45	1.56 ± 0.68

TABLE 8: Mass balances of Hg between input and output streams in three power plants ($n = 3$).

	Stream		Plant-1	Plant-2	Plant-3
Input	Coal	g/hr	4.48	16.9	8.24
	Bottom ash	g/hr	5.00×10^{-3}	2.00×10^{-3}	3.00×10^{-3}
Output	Fly ash	g/hr	0.193	0.366	0.721
	Gypsum	g/hr	2.60	0.373	7.18
	Stack flue gas	g/hr	1.31	12.3	1.79
Mass balance	(Input/output) \times 100	%	91.6	77.1	118

TABLE 9: Comparison of Hg measurements in flue gas between the new sorbent method and OH method in plant-3 ($n = 3$).

Hg Concentration in flue gas ($\mu\text{g}/\text{m}^3$)	This method	OH method	Relative error
Before ESP	9.14 ± 3.54	10.3 ± 1.9	11.3%
After ESP (before WFGD)	7.01 ± 2.83	7.95 ± 2.32	11.8%
After WFGD	1.56 ± 0.68	1.38 ± 0.84	14.3%

4. CONCLUSIONS

This study developed the measurement of mercury in flue gas based on aluminum matrix sorbent. A sorbent trap was used to collect vapor phase Hg from flue gas and the Hg on trap was measured using CVAAS followed by acid leaching.

Hg adsorbed by sorbent was recovered completely by leaching process. The average bias of the analytical technique was 2.68%, and the average recovery of spiked Hg^0 in tube 3 varied from 89.7% to 105% (averaged 97%). Hg breakthrough in tube 2 was negligible compared to Hg content in tube 1. The average recovery of tube 1 and tube 2 was considered acceptable with the variation of 83.0% to 94.3% (averaged 87.7%).

The mercury in coal is distributed into bottom ash, ESP ash (fly ash), WFGD reactant (gypsum), and flue gas, and the relative distribution varied depending on factors such as the coal type and the operation conditions of plants.

The Hg mass balances of three power plants were 91.6%, 77.1%, and 118% for mercury in coal (0.161 mg/kg, 0.064 mg/kg, and 0.225 mg/kg), respectively, and the relative errors of the results between this method and OH method were less than 5% and 15% for measurements practiced in lab and field, respectively, which indicated that this method can be applied to measure Hg emissions from coal-fired combustion sources.

Further study is needed for Hg speciation in flue gas and its model prediction.

ACKNOWLEDGMENT

This study was supported by the High-Tech R&D Program of China (863) under Grant no. 2007AA06Z340.

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This article should be cited as follows:

Juan Wang, Wei Xu, Xiaohao Wang, and Wenhua Wang, “Measurement of Mercury in Flue Gas Based on an Aluminum Matrix Sorbent,” *TheScientificWorldJOURNAL*, vol. 11, pp. 2469–2479, 2011.



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