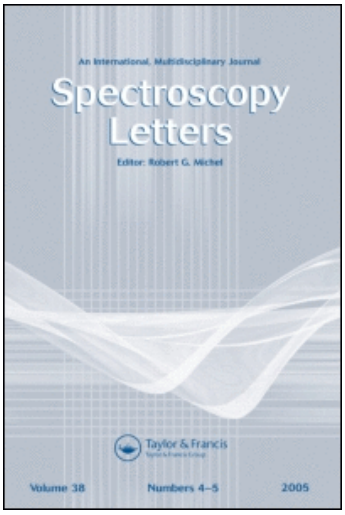

Spectroscopy Letters
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597299

Optimization of Pretreatment Method for Alkylmercuries Speciation in Coal by High-Performance Liquid Chromatography Coupled with UV-Digestion Cold Vapor Atomic Fluorescence Spectrometry

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To cite this Article He, Bin , Gao, Erle , Shi, Jianbo , Liang, Lina , Yin, Yongguang and Jiang, Guibin(2006) 'Optimization of Pretreatment Method for Alkylmercuries Speciation in Coal by High-Performance Liquid Chromatography Coupled with UV-Digestion Cold Vapor Atomic Fluorescence Spectrometry', Spectroscopy Letters, 39: 6, 785 — 796
To link to this Article: DOI: 10.1080/00387010600969945
URL: http://dx.doi.org/10.1080/00387010600969945

Spectroscopy Letters, 39: 785–796, 2006

ISSN 0038-7010 print/1532-2289 online
DOI: 10.1080/00387010600969945

Optimization of Pretreatment Method for Alkylmercuries Speciation in Coal by High-Performance Liquid Chromatography Coupled with UV-Digestion Cold Vapor Atomic Fluorescence Spectrometry

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Abstract: Exhaustive extraction of analytes in their original chemical forms from samples with complex matrices is a pivotal step for speciation analysis. Herein we propose a pretreatment method for extracting and preconcentrating methylmercury and ethylmercury from coal samples by using $KBr–H_2SO_4/CuSO_4–C_6H_5CH_3–Na_2S_2O_3$ system. The extraction conditions, including the volume of the organic phase and the extraction time, were optimized in detail. Speciation analysis of alkylmercuries was carried out by high-performance liquid chromatography online coupled with UV-digestion and cold vapor atomic fluorescence spectrometry. The detection limits were 0.6 ng mL^{-1} for methylmercury and 1 ng mL^{-1} for ethylmercury, respectively. The recoveries of methylmercury and ethylmercury spiked in a sample were 84% and 82%, respectively. The method was applied successfully to analysis of alkylmercuries in four coal samples collected from northeast China.

Keywords: Alkylmercuries, coal samples, pretreatment method, speciation analysis

Received 5 April 2006, Accepted 22 August 2006

The authors were invited to contribute this paper to a special issue of the journal entitled "Spectroscopy and Automation". This special issue was organized by Miguel de la Guardia, Professor of Analytical Chemistry at Valencia University, Spain.

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INTRODUCTION

Mercury (Hg) is a highly toxic element and present in all kinds of environments around the world. Exposure to mercury will cause various adverse effects.[1] The toxicity, mobility, and bioavailability of the mercury compound depend more on its chemical form than its total concentration. As the most toxic and the most common organic mercury in the environment, methylmercury (MeHg) draws the most attention of humans. Ethylmercury (EtHg) is less toxic than MeHg. Besides anthropogenic EtHg discovered in an industrial polluted site,[2] heterogenic EtHg was also detected in samples from an everglade[3] and a national park,[4] respectively. Therefore, both MeHg and EtHg should be a concern for determination in order to evaluate the toxicology and contamination of organomercuries.

Coal is mainly used as a kind of energy for electricity and industrial use. The content of mercury is relatively high in coal, and mercury will release to the environment during the process of combustion, cleaning, or storage of coal. Because of the huge amounts of coal consumed, mercury from coal has accounted for the biggest portion of mercury pollution all over the world. Although most of the mercury compounds are transformed to inorganic mercury by combustion released into air, ash, or residue, speciation of mercury compounds in coal may predict the biogeochemistry of the original coal.

The key step of speciation is to extract the mercury compounds from coal. Both high recovery and the original form of organomercuries are required. Two methods for the extraction of mercury compounds from solid samples are usually used (i.e., alkaline digestion and acidic leaching). The alkaline digestion extraction system is mainly used for biological samples.[5] The $KBr-H_2SO_4/CuSO_4-$organic solvent system is mainly used for the extraction of organomercuries from soil and sediment. Organomercuries were released from sample matrices by ion exchanging effected by acid and copper ions.[6,7] After releasing from solid matrix, mercury compounds were extracted into organic phase and back-extracted in chelating agent (i.e., $Na_2S_2O_3$), for cleaning and preconcentration.

The most widely used techniques for the speciation of organomercuries are gas chromatography (GC) and high-performance liquid chromatography (HPLC) coupled with element-specific detector for mercury, such as atomic absorption spectrometry (AAS),[8,9] atomic emission spectrometry (AES),[10] inductively coupled plasma mass spectrometry (ICP-MS),[11,12] and AFS.[13,14]. However the AAS and AES have lower sensitivity, and ICP-MS is too expensive despite its multielement capability and extreme sensitivity. Although GC is the widely used separation technique for mercury speciation, the derivatization is too laborious. HPLC has the advantages of facilitated sample pretreatment, ambient separation temperature, large volumes of sample injection, and easy automatization.[15,16] Most of the HPLC methods for speciation of organomercuries are based on the reversed-phase separation technique, which contains a chelating or ion-pair reagent, then coupling a ultraviolet lamp

(UV) to digest organomercuries to inorganic mercury, followed by forming the volatile mercury vapor to be detected. Regarding the advantages of atomic fluorescence spectrometry (AFS) (i.e., excellent sensitivity, wide linear range, little spectral interference, and lower cost), the HPLC-CV-AFS coupled technique has become one of the most useful coupled systems for the determination of organomercuries in various environmental and biological samples.[17–19]

Our work is directed to the optimization of extraction method and the speciation of alkylmercuries (MeHg and EtHg) from coal samples.

MATERIALS AND METHODS

Instrumentation

A HPLC system consisted of a quaternary pump (P680 HPLC Pump, Dionex, Sunnyvale, CA, USA) and a reversed-phase Agilent Zorbax ODS column (4.6 × 150 mm, 5 μm) was used to separate alkylmercuries. Sample injection was performed on a Rheodyne model 7725i injection valve (Rheodyne, Cotati, Rohnert Park, CA, USA) with a 20-μL sample loop. The mercury species were separated by a 10% (v/v) acetonitrile solution containing 60 mmol L^{-1} sodium acetate and 0.01% (v/v) 2-mercaptoethanol as mobile phase.

The effluent of HPLC was delivered to an 8-m PTFE digestion coil (i.d. 0.8 mm) wrapped around an 8-W UV lamp where the decomposition of alkylmercuries to inorganic mercury took place with $K_2S_2O_8$ in HCl converged by a peristaltic pump as oxidant. After the KBH_4 solution was introduced by a peristaltic pump, the produced mercury cold vapor was separated in the gas–liquid separator and carried to the detector by an argon stream.

Determinations were carried out using a model AF-610A nondispersive atomic fluorescence spectrometer (Beijing Raileigh Analytical Instrument Co., Beijing, China) with a high-intensity hollow cathode mercury lamp at 253.7-nm line source (Beijing Tiangong Analytical Instrument Factory, Beijing, China) running at 280 V of PMT voltage and 40 mA of lamp current. A personal computer fitted with an AF-610A software was applied for the control of the AFS and the integration of the peak areas.

A general view of the HPLC-UV-CV-AFS system applied in this experiment is shown in Fig. 1. The geometry of the commercial gas–liquid separator is shown in Fig. 2,[20] and the experimental conditions are shown in Table 1.

Reagent and Standards

Stock solutions of 1000 mg L^{-1} alkylmercuries (as Hg) were prepared by dissolving appropriate amounts of methylmercury chloride and ethylmercury

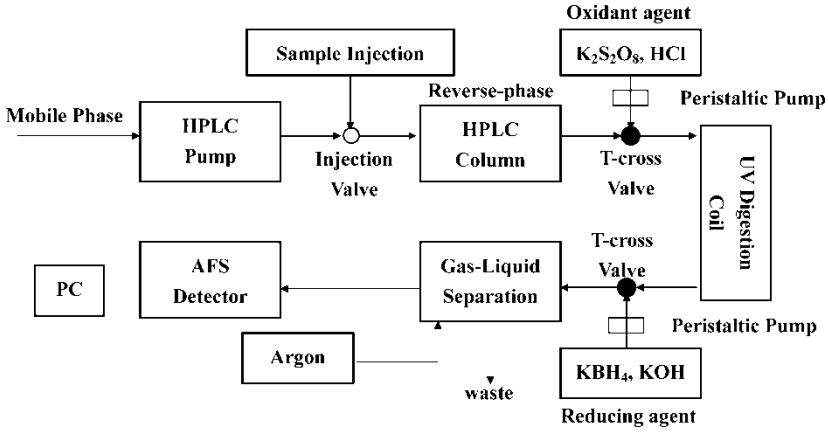

Figure 1. Schematic diagram of HPLC-UV-CV-AFS system.

chloride (both from Merck-Schuchardt, Whitehouse Station, NJ, USA) in methanol and stored at 4°C in darkness. Working solutions diluted with methanol for analysis were prepared daily prior to use.

A 3 mol L^{-1} ammonium acetate (CH_3COONH_4) solution was prepared and stored at 4°C in darkness. The HPLC mobile phase was prepared by mixing 20 mL of 3 mol L^{-1} CH_3COONH_4, 0.1 mL of 2-mercaptoethanol, and 100 mL of acetonitrile (CH_3CN) first, and then diluted to 1000 mL with deionized water. The mobile phase was prepared daily and filtered through a 0.45-μm membrane filter and degassed by ultrasonic prior to use.

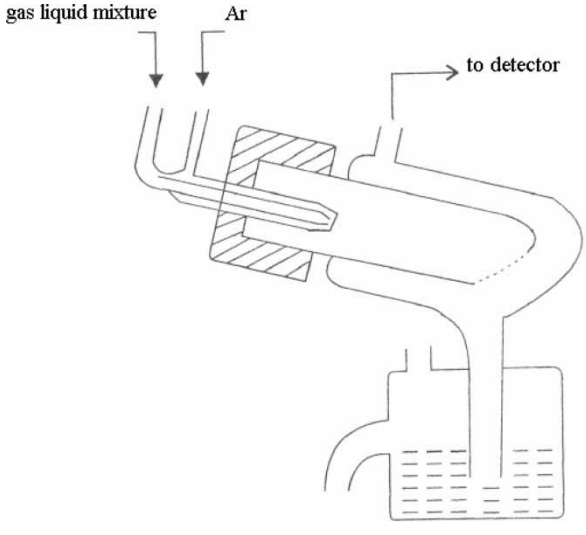

Figure 2. The geometry of the commercial gas–liquid separator.[18]

Table 1. Experimental conditions of HPLC-UV-CV-AFS

HPLC	
Column	Agilent Zorbax ODS column, 4.6 × 150 mm, 5 μm
Mobile phase	10% (v/v) CH_3CN; 60 mmol L^{-1} CH_3COONH_4; 0.01% (v/v) 2-mercaptoethanol
Flow rate of mobile phase	1.0 mL min^{-1}
Sample injection volume	20 μL
Hydride generation	
Oxidant solution	0.5% (m/v) $K_2S_2O_8$ in 10% (v/v) HCl, 1.8 mL min^{-1}
Reducing solution	0.5% (m/v) KBH_4, 3.6 mL min^{-1}
AFS	
Lamp	Hollow cathode mercury lamp, 253.7 nm
PMT voltage	280 V
Primary current	40 mA
Carrier gas	Argon, 500 mL min^{-1}

KBr 90 g was dissolved in 100 mL water. Concentrated H_2SO_4 25 mL was added in 50 mL water. After cooling to room temperature, the two solutions were mixed and diluted to 500 mL with deionized water to prepare acidic potassium bromide solution.

$CuSO_4$ (1 mol L^{-1}) solution was prepared by dissolving 25g $CuSO_4 \cdot 5H_2O$ in 100 mL water. $Na_2S_2O_3$ solution (0.01 mol L^{-1}) was prepared by dissolving 0.2482 g $Na_2S_2O_3 \cdot 5H_2O$ in 100 mL water. The two solution were stored at 4°C in darkness.

KBH_4 0.5% (m/v) solution was prepared daily by dissolving 5 g KBH_4 in 1000 mL 0.2% (m/v) KOH solution; 0.5% $K_2S_2O_8$ (m/v) solution was prepared daily by dissolving 5 g $K_2S_2O_8$ in 1000 mL 10% (v/v) HCl solution.

All reagents were of analytical grade except where stated, and deionized water from EASY pure LF (Barnstead Co., Center Barnstead, NJ, USA) was used throughout.

Sample Preparation and Extraction

Coal samples were crushed with a grinder, passed through a 40-mesh sieve, dried at −45°C, then homogenized completely and kept at −18°C in darkness.

For extraction, 0.5 g of coal sample was weighed. For spiked recovery studies, appropriate aliquots of standard solution of alkylmercuries were added into the coal samples. Then samples were wetted by 5 mL deionized water in a 40-mL glass centrifuge tube and then 4 mL acidic KBr/$CuSO_4$ solution (3:1, v/v) was added. The tube was mechanically shaken for 12hr for digestion. Then 6 mL toluene was added into the tube and shaken for 60 min to extract organomercuries into toluene. After centrifugation for 20 min at 3500 rpm, 3.5 mL

toluene layer was transferred into a 10-mL glass centrifuge tube. The alkylmercuries were back-extracted into 1 mL $Na_2S_2O_3$ solution after shaking for 60 min and then centrifuging for another 20 min at 3500 rpm. The water phase was pipetted into a 2-mL PET microcentrifuge tube. After centrifugation for 30 min at 12,000 rpm, the solution was injected directly without further filtration and determined by the HPLC-CV-AFS system.

RESULTS AND DISCUSSION

Choice of Extraction Solvent

CH_2Cl_2, benzene, and toluene are commonly used organic solvents for extracting organomercury from various samples. In our experiments, we found that when CH_2Cl_2 was used as extractant, part of fine coal powder was suspended in the lower CH_2Cl_2 layer and difficult to be separated, only by centrifugation at high speed. Extra filtration with membrane or SPE cartridge is needed, which may result in contamination or loss of analytes. This problem could be avoided when benzene or toluene was applied as extractant because they are on the top of the extraction system. Although benzene and toluene have the similar extraction efficiency for alkylmercuries, the latter was chosen as extractant because of less toxicity.

The Volume of the Organic Phase and the Extraction Time

As the volume of toluene and the extraction time would influence the efficiency of extraction, we optimized these two conditions with standard solution containing MeHg and EtHg. The results shown in Fig. 3 indicate that the extraction efficiency increased with the increase of extractant volume when shorter extraction time was applied. When extraction time of 60 min was used, the volume of organic solution between 6 and 8 mL has no significant effect on extraction efficiency. Considering the complexities of the real coal samples and the enrichment factor, 6 mL toluene and 60-min extraction time showed the best recovery and working efficiency, so 6 mL toluene and 60-min extraction time was adopted as optimum.

Effect of $K_2S_2O_8$ and KBH_4 Concentration on Cold Vapor Generation

$K_2S_2O_8$ (in HCl) was used as an oxidant and KBH_4 (in KOH) was used as reducing agent. The concentration of $K_2S_2O_8$ would affect the sensitivity significantly. Low concentration of $K_2S_2O_8$ could lead to poor decomposition efficiency of organic mercury, whereas excess $K_2S_2O_8$ would react with KBH_4,

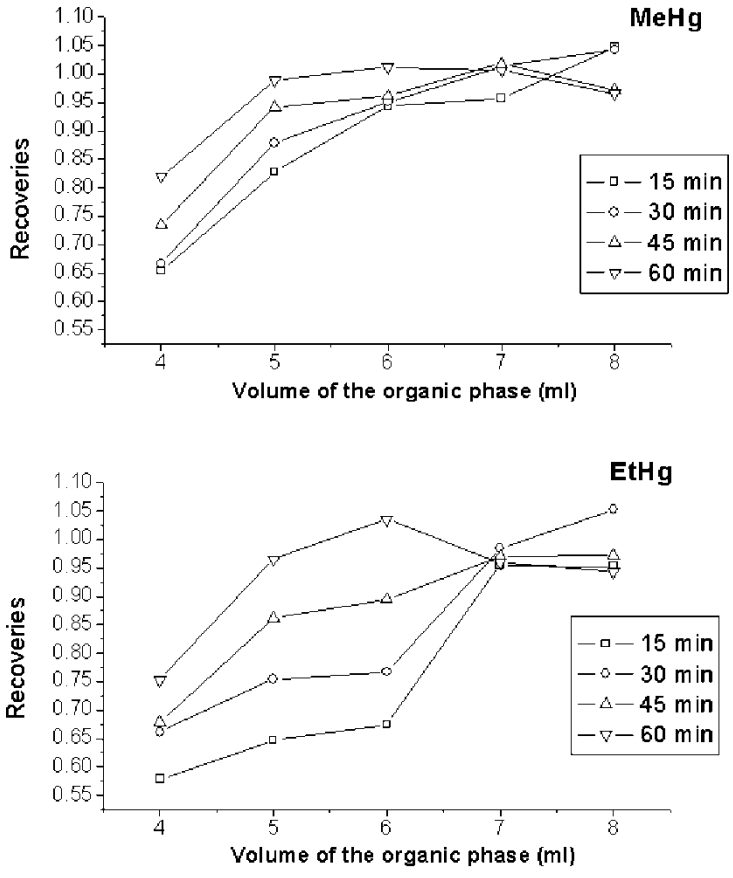

Figure 3. Effect of the volume of the organic phase and the extraction time.

resulting in lower efficiency of cold vapor generation. The concentration of $K_2S_2O_8$ was optimized at different concentration of KBH_4. Results in Fig. 4 indicate that 0.5% $K_2S_2O_8$ (m/v) in 10% HCl (v/v) and 0.5% KBH_4 (m/v) in 0.2% KOH (m/v) were optimal and have been used throughout the experiment.

Figures of Merit of the Speciation

Typical HPLC-UV-CV-AFS chromatograms of mixed alkylmercury standard at 20 ng mL^{-1} level, sample S1, sample S2, and spiked sample S1 are shown in Fig. 5. The two species of alkylmercuries were separated absolutely in 25 min. The retention times for MeHg and EtHg were 5.7 min and 19.1 min, respectively. Some analytical properties of this method are shown

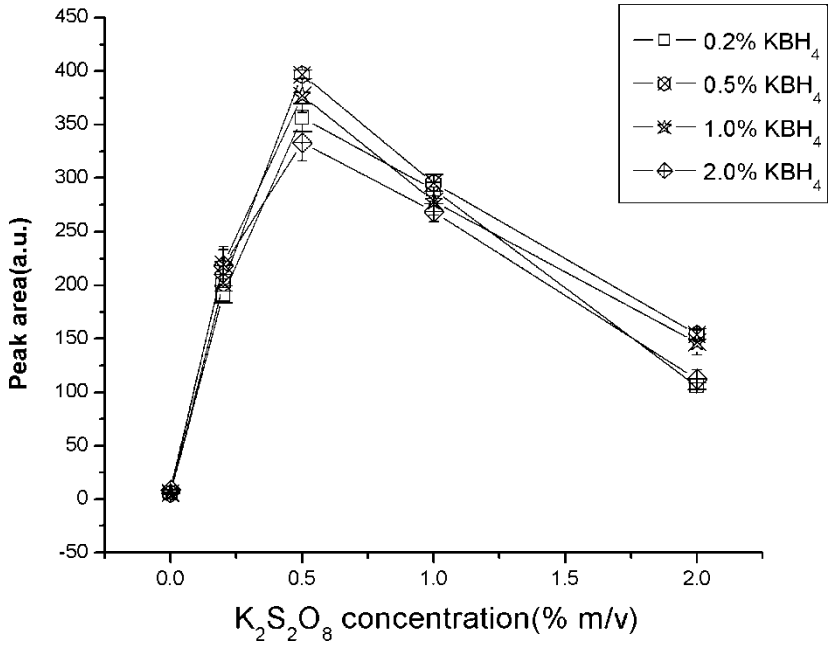

Figure 4. Effect of the $K_2S_2O_8$ and KBH_4 concentration on the sensitivity of the mercury compounds. The injected MeHg was 20 ng mL^{-1}. Other conditions are shown in Table 1.

in Table 2. The linearity of MeHg and EtHg was from 0 to 100 ng g^{-1}. The detection limits (DLs), calculated as three times the standard deviation of the baseline noise, were 0.6 ng mL^{-1} for MeHg and 1 ng mL^{-1} for EtHg, respectively. These DLs are lower than most of the results presented in previous literature,[21,22] about half of the DLs obtained by Hintelmann and Wilken[23] and similar to the system used by E. Ramalhosa.[19] The relative standard deviations (RSD, n = 5) for MeHg and EtHg at 10 ng mL^{-1} were 3.87% and 3.55%, respectively.

Table 2. Some characteristics of the HPLC-UV-CV-AFS

Mercury species	Calibration curve	Correlation coefficient	Linear range (ng mL^{-1})	Detection limit (ng mL^{-1})	RSD (%)[a]
MeHg	y = 20.585x − 4.4841	0.9998	0–100	0.6	3.87
EtHg	y = 12.719x − 5.3836	0.9987	0–100	1.0	3.55

[a]Concentration = 10 ng Hg mL^{-1}, n = 5.

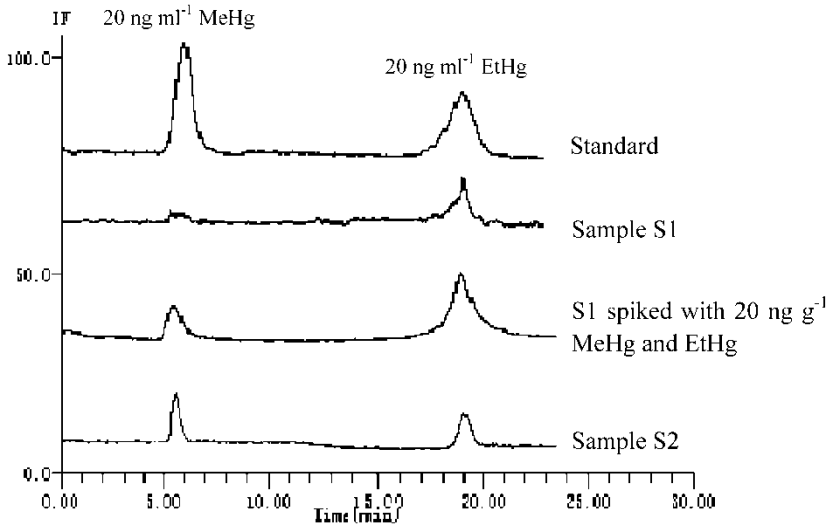

Figure 5. Chromatogram of MeHg and EtHg in standard solution, samples S1 and S2, and spiked S1.

Table 3. Analytical result of MeHg and EtHg in CRM IAEA-405 (ng g^{-1}, mean $\pm$ SD, n = 4)

	MeHg		EtHg	
CRM	Certified value	Determined	Certified value	Determined
IAEA-405 (Estuarine sediment)	5.49 $\pm$ 0.549	5.77 $\pm$ 0.471	NA	ND

NA, not available; ND, not detectable.

Table 4. Recoveries of MeHg and EtHg spiked into a coal sample

Mercury species	Contents in unspiked, (ng g^{-1})	Spiked (ng g^{-1})	Contents in spiked, (ng g^{-1})	Recovery (%)
MeHg	3.68 $\pm$ 0.06	20	19.98 $\pm$ 2.07	84 $\pm$ 9
EtHg	19.50 $\pm$ 0.77	20	32.37 $\pm$ 3.27	82 $\pm$ 9

Table 5. MeHg and EtHg in five coal samples collected from China

Sample	MeHg concentration[a] (ng g^{-1})	EtHg concentration[a] (ng g^{-1})
S1	4.52 ± 0.28	ND
S2	ND	2.16 ± 0.05
S3	1.85 ± 0.24	ND
S4	4.50 ± 0.34	6.75 ± 0.23

ND, not detectable.
[a]Mean value ± SD, n = 3.

Application of the Method

As there is no CRM of coal available for the analysis of organomercuries, a CRM IAEA-405 (estuarine sediment) was analyzed to validate the current optimal extraction and speciation method. The results are listed in Table 3. No EtHg was detected in the IAEA-405. The determined MeHg content (5.77 ± 0.471) was in good agreement with the certificated value (5.49 ± 0.549). Besides, the spiked experiments for MeHg and EtHg were carried out. The recoveries were 84% for MeHg and 82% for EtHg, respectively (Table 4), which demonstrated the feasibility of the proposed method. The proposed method was applied to the speciation analysis of MeHg and EtHg in four coal samples collected from northeast China. The MeHg and EtHg contents are shown in Table 5, which indicates that there is no correlation between MeHg and EtHg for individual samples.

CONCLUSIONS

A method based on $KBr/CuSO_4–C_6H_5CH_3–Na_2S_2O_3$ extraction and HPLC-UV-CV-AFS detection for the speciation analysis of alkylmercuries in coal samples was developed and validated by analyzing CRM estuarine sediment sample (IAEA-405). The analytical results were in good agreement with the certified values of the CRM. In addition, satisfactory recoveries were also obtained. Speciation analysis of MeHg and EtHg in four coal samples was successfully carried out, and both MeHg and EtHg were found in some samples without correlation. As the AFS instrument is much cheaper, HPLC-AFS coupling system can be applied in most laboratories. Besides, there is a large amount of coal for all kinds of use all over the world, and this proposed method may be useful to monitor the mercury pollution from coals.

ACKNOWLEDGMENTS

This work was jointly supported by the National Natural Science Foundation of China (20577057, 20477053) and the Chinese Academy of Science (KZCX3-SW-431)

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