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Evaluation of different extraction procedures for determination of organic Mercury species in petroleum by high performance liquid chromatography coupled with cold vapor atomic fluorescence spectrometry

Zhaojun Yun, Bin He*, Zhenhua Wang, Thanh Wang, Guibin Jiang

State Key Laboratory of Environmental Chemistry and Ecotoxicology, Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, PO Box 2871, Beijing 100085, PR China

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ABSTRACT

An extraction procedure for extracting organic mercury species including methylmercury (MeHg) and ethylmercury (EtHg) from petroleum samples was developed. Three extraction methods (shaking, ultrasonic and microwave assisted extraction) using different extraction solvents (TMAH, KOH/CH_3OH, HCl and acidic $CuSO_4$/KBr) were investigated by comparing the extraction efficiency of the organic mercury species. Microwave assisted extraction at 60 W for 5 min using TMAH (tetramethylammonium hydroxide, 25%, m/v) provided the most satisfactory extraction efficiency for MeHg and EtHg in petroleum at 86.7% ± 3.4% and 70.6% ± 5.9%, respectively. Speciation analysis of mercury was done by on-line coupling of high performance liquid chromatography with cold vapor generation atomic fluorescence spectrometry (HPLC-CV-AFS). The proposed method was successfully applied to analyze several crude oil and light oil samples. The concentrations of MeHg ranged from under detection limit to 0.515 ng g^{-1}, whereas EtHg was not detected in the samples. This method can be a very useful tool in evaluating the risk of mercury emissions from petroleum.

1. Introduction

Mercury (Hg) is considered as a global pollutant due to its toxicity, persistence, long range transport potential and bioaccumulation [1]. Emission and distribution of Hg to the environment can occur from both natural process and human activities [2]. It has been estimated that global mercury emissions caused by human activities range from 6.6 to 9.4×10^6 kg yr^{-1}, which is considered to be much higher than those originating from natural sources [3]. The toxicity of mercury strongly depends on its chemical species, and increasing studies show that the organic mercury species, especially methylmercury (MeHg), are more toxic than inorganic mercury [4,5].

Petroleum is one of the most important energy sources for the modern society, and also the raw material for many industrial products such as chemical fertilizer, plastics and more. It is reported that the concentration of Hg in crude oil and natural gas varies between 0.01 ng g^{-1} to 10 μg g^{-1} (wt.), which is highly dependent on its geological location [6]. In the USA, about 6000 kg Hg is emitted to the atmosphere from combustion of liquid hydrocarbon fuels every year [7]. Oil spill is another source of Hg pollution. Some studies demonstrated that the concentration of Hg increased significantly in the air [8], seabird [9,10] and other animals [11] at oil spill areas, which indicates increased environmental health risk from Hg released from oil spill. It is therefore meaningful to monitor not only the total Hg concentration in petroleum, but also the various organic mercury species.

The methodologies currently used for mercury are based on a chromatographic technique, either gas chromatography (GC) or high performance liquid chromatography (HPLC), hyphenated with a sensitive element detector. The HPLC method is widely used because of its ease of use and requires no derivatization. A variety of element specific detectors could be coupled with HPLC for speciation of mercury, such as atomic absorption spectrometry (AAS) and inductively coupled plasma mass spectrometry (ICP-MS). However, each of these detectors has drawbacks such as poor sensitivity of AAS and high operating cost for ICP-MS. Atomic fluorescence spectrometry (AFS) could compensate for these shortcomings for its similar detection limits with ICP-MS but lower instrumental and running cost.

Accurate determinations of total mercury and speciation of mercury compounds depend on not only rigorous analytical procedures but also appropriate sample preparation. Several sample preparation methods for the determination of total mercury have been published: acid digestion [12], microwave-assisted sample decompositions [13], improved combustion/trap

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* Corresponding author. Tel./Fax: +86 10 62849179.
E-mail address: bhe@rcees.ac.cn (B. He).

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technique [14], closed-system combustion [15], in-situ thermal decomposition [16] and electro-thermal vaporization [17]. However, studies about speciation of Hg in petroleum are still very scarce. To date, instrumental methods developed for the mercury species in petroleum or gas condensate include GC-ICP-MS [18], GC-AFS [19] and HPLC-ID-ICP-MS [20]. Either of these methods require derivatization or cost too much for routine analysis.

The most used sample preparation step for speciation of mercury species is wet extraction. Solvents such as TMAH [21], KOH/CH_3OH [22] or HCl [23] or Cu^{2+} in acid circumstance [24] have often been used as the extractants. However, there is still no comprehensive evaluation of preparation for mercury speciation in petroleum.

In this work, three extraction methods (shaking, ultrasonic and microwave assisted extraction) using different extraction solvents (TMAH, KOH/CH_3OH, HCl and acidic $CuSO_4$/KBr) were compared by the extraction efficiency of organic mercury species, which was characterized by the recoveries. A simple and effective method for the speciation of mercury was proposed for routine analysis, and this method was used to evaluate the organic Hg species in several petroleum samples.

2. Experimental

2.1. Instrumentation

The separation of mercury species was achieved on a Shim-pack CLC-ODS C18 column (150 mm × 6 mm I.D. × 5 μm, Shimadzu, Japan). The HPLC system consisted of a P680 pump (P680 HPLC Pump, Dionex, USA) and a Rheodyne model 7725i injector (Rheodyne, Cotati, CA, USA) equipped with a 100 μL sample loop. Mercury species were determined on an AF-610D2 atomic fluorescence spectrophotometer (Beijing Rayleigh Analytical Instrument Corporation, Beijing, China) with a mercury hollow cathode lamp (HCL) (General Research Institute of Non-Ferrous Metals, Beijing, China), operated at the wavelength of 253.7 nm as the radiation source.

Microwave assisted extractions and the digestion of the petroleum samples in the experiment were conducted by a microwave accelerated reaction system (Mars5 HP500, CEM Corporation, USA).

Shaking extraction and ultrasonic extraction was achieved on the DDHZ-300 oscillator (Taicang City Experimental Equipment Factory, Jiangsu, China) and KQ 300DE ultrasonic system (Shumei Instrument Corporation, Jiangsu, China), respectively.

2.2. Reagents and standards

The divalent mercury stock solution (1000 mg L^{-1} as Hg) was prepared from an appropriate amount of mercury chloride (≥99.5%, Beijing Chemical Reagents Company, Beijing, China), which was dissolved in 5% HNO_3 (65%, Merck, Darmstadt, Germany). The stock solutions of MeHg and EtHg (1000 mg L^{-1} as Hg) were prepared by dissolving an appropriate amount of methylmercury chloride and ethylmercury chloride (≥98%, Merck, Darmstadt, Germany) in HPLC-grade methanol. All stock solutions were stored in the dark at 4 °C. 2-mercaptoethanol (≥98%) was obtained from Alfa Aesar (Ward hill, Ma, USA). Tetramethylammonium hydroxide (TMAH, 25%, v/v) was purchased from National Medicines Corporation (Beijing, China), HPLC-grade methanol and dichloromethane were purchased from J. T. Baker (Phillipsburg, NJ, USA). All other chemicals were analytical grade or better. De-ionized water (18.2 MΩ cm) used throughout the experiment was from a Millipore ultrapure water system (Millipore, Bedford, USA).

Table 1
Procedures of microwave digestion for total mercury.

Stage	Power W	Ramp Min	Control °C	Hold Min
1	1200	6	120	2
2	1200	6	160	8
3	1200	5	190	30

Acidic KBr solution was prepared by dissolving 18 g KBr and 5 mL concentrated H_2SO_4 in 100 mL deionized water.

$CuSO_4$ solution was prepared by dissolving 16 g $CuSO_4$ in 100 mL de-ionized water.

KOH/CH_3OH solution (25%, m/v) was prepared by dissolving 25 g KOH in 100 mL CH_3OH.

The mobile phase for the HPLC-CV-AFS system consisted of 1 g L^{-1} L-cysteine, and 0.06 mol L^{-1} ammonium acetate.

2.3. Sample preparation

For total mercury: The oil samples were digested with the microwave accelerated reaction system. The digestion procedures are shown in Table 1. Briefly, (1) 0.1–0.2 g samples were weighed exactly into PTFE digestion tubes, then 5 mL HNO_3 and 3 mL H_2O_2 were added into each tube. (2) The microwave tubes were irradiated for 2 min after temperature was raised to 120 °C in 6 min, then held 8 min after rising to 160 °C in 6 min, and then held 30 min after rising to 190 °C in 5 min. Finally, the microwave tubes were cooled down to room temperature. (3) The samples were diluted with de-ionized water to 100 mL, and the solutions were then passed through 0.22 μm filter prior to analysis. The total mercury concentrations were determined with AFS.

For organic mercury (classified by different extraction methods):

A. Shaking extraction. (1) 0.10–0.50 g samples were weighed exactly into 40 mL glass vials, and then 2 mL TMAH (25%, m/v), or 2 mL KOH/CH_3OH (25%, m/v), or 2 mL HCl (6M) or 4 mL acidic KBr/$CuSO_4$ solution (3:1, v/v) was added into each vial as extractant. Then the glass vials were mechanically shaken at 250 rpm for 3 h. (2) 6 mL dichloromethane was added (for the two alkaline extraction procedures, 1.5 mL concentrated HCl was added into the vial drop by drop before adding dichloromethane) and the mixture was shaken for 45 min to extract organic mercury species into the dichloromethane phase. (3) After centrifugation at 3000 rpm for 15 min, 4–5 mL dichloromethane phase was transferred into a 10 mL glass vial and the volume of removed portion was recorded accurately. 1 mL 10 mM $Na_2S_2O_3$ for re-extraction was added and then shaken for 15 min. (4) After centrifugation for another 15 min at 3000 rpm, 0.6–0.8 mL aqueous phase was drawn out and to pass through 0.22 μm fiber filter. The filtrate was stored in a refrigerator before injecting directly into the HPLC.

B. Ultrasonic extraction. This method was almost similar to the shaking extraction except that the vial with the mixture (samples and each extractant) was sonicated at 300 W for 3 h instead of shaking in the first step. The subsequent procedures were then the same.

C. Microwave assisted extraction. (1) 0.10–0.50 g petroleum samples were weighed into 50 mL PTFE tubes, and then the tested extraction solvent was added. The tubes were vertically mixed, screwed and placed in the microwave accelerated reaction system. (2) The microwave tubes were irradiated for 5 min at the power of 30, 60 or 90 W, and then allowed to be cooled down to room temperature. (2) 1.5 mL concentrated

HCl was added drop by drop into the tube which was prior added with TMAH and KOH/CH_3OH as extractant. The mixture was transferred into a 40 mL glass centrifuge vial and 0.5 mL 6 M HCl was added into the PTFE tube for cleaning. The clean fluid was thereafter added to the glass vial, too. Then 6 mL dichloromethane was added into the glass vial. The subsequent procedures were the same as for the shaking and ultrasonic extraction.

2.4. Total Mercury determination and speciation of Mercury

Total mercury was determined by AFS after microwave digestion. Speciation of mercury species was performed by HPLC-CV-AFS, which has previously been successfully applied to analyzing Hg in samples such as certified reference material (DORM-2, dogfish muscle) and biological samples [25].

3. Result and discussion

3.1. Optimization of HPLC-CV-AFS parameters

A simple on-line high performance liquid chromatography cold vapor generation atomic fluorescence spectrometry (HPLC-CV-AFS) method by adding L-cysteine into the mobile phase was used and the parameters which could influence the chromatographic separation and the fluorescence signals have been optimized in our previous work [25]. In brief, the mobile phase of HPLC was 1 g L^{-1} L-cysteine and 0.06 mol L^{-1} ammonium acetate in water at a flow rate of 1 mL min^{-1}. Mercury in the sample was transferred by 10% HCl (v/v), reduced to Hg^0 vapor by KBH_4 (0.5%, in 0.2% KOH solution) at room temperature and then could be detected by AFS directly without further atomization. Argon was used as carrier gas and auxiliary gas and both at a flow rate of 300 mL min^{-1}. The separation and determination of MeHg and EtHg could be achieved by this system without any organic solvent and strong oxidants.

The analytical features of the HPLC-CV-AFS were listed in Table 2. For the speciation of organic mercury species under the optimized conditions, the detection limits based on three levels of the background were 0.05 and 0.07 μg L^{-1} (as Hg) for MeHg and EtHg by using a 100 μL sample loop. The RSD ($n=5$) was less than 6% for both MeHg and EtHg species. Fig. 1 shows the chromatogram of the standard solution (1 ng mL^{-1}) and a petroleum sample.

3.2. Optimization of different extraction methods and extractants

A. Shaking extraction. The extraction efficiency of this method using four extraction solvents are shown in Fig. 2. None of the extractants could provide satisfactory extraction efficiency for both MeHg and EtHg after shaking for 3 h. TMAH provided more efficient extraction of MeHg, while HCl was the most efficient extractant for EtHg.

Table 2
Analytical performance of the HPLC-CV-AFS system.

Compound	Linearity equation	Correlation coefficient	Detection limit/ ng mL^{-1}	RSD (%)[a]
Total mercury	$y=10354x+2440$	0.9994	0.019	1.7
MeHg	$y=6339x+201.2$	0.9995	0.05	1.8
EtHg	$y=6695x+39.53$	0.9999	0.07	2.1

[a] Standard concentration, 5 ng mL^{-1}, $n=5$.

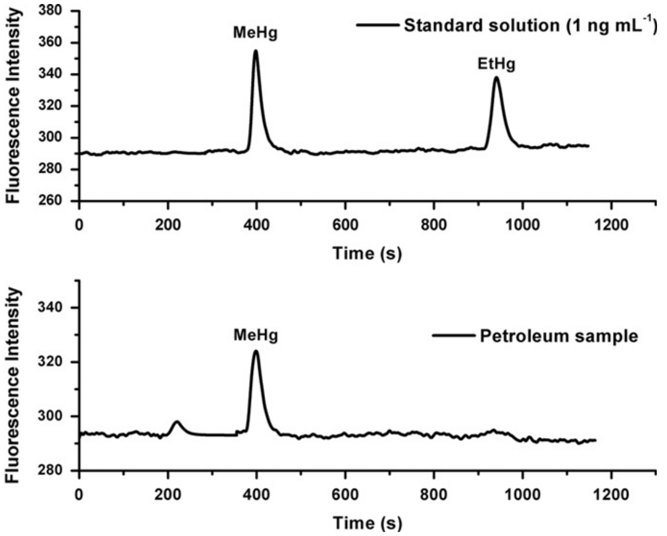

Fig. 1. Chromatogram of a standard solution (1 ng mL^{-1}) and petroleum sample.

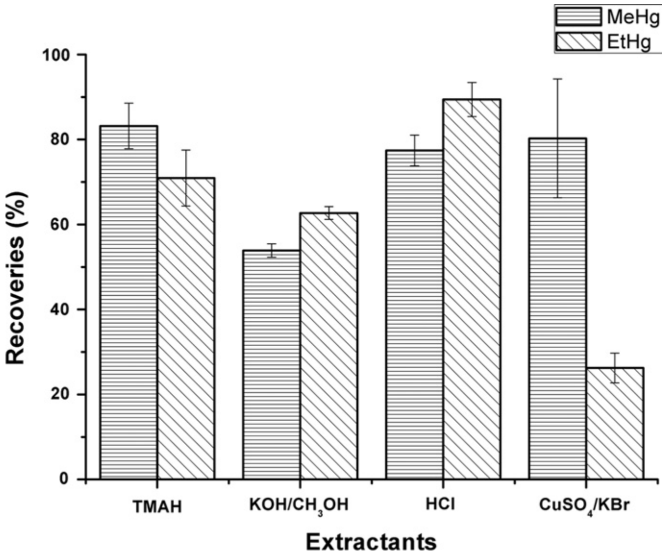

Fig. 2. Extraction efficiency of the shaking extraction for MeHg and EtHg using different extraction solvents.

B. Ultrasonic extraction. Fig. 3 shows the extraction efficiency of this method using four different extractants. All extractants used in the ultrasonic method for the extraction of MeHg from the petroleum matrix were more effective than that by shaking extraction except acidic KBr/$CuSO_4$ solution (3:1, v/v). On the contrary, the extraction efficiencies for EtHg were generally poor. Moreover, EtHg could not be detected at all by using the acidic KBr/$CuSO_4$ solution as extractant. This result suggested that the organic mercury might be decomposed by the application of ultrasound, and this phenomenon has been reported in some other literature [26].

C. Microwave assisted extraction. The extraction efficiency by using microwave system was investigated in regards to the power of the microwave and extraction time. Due to the complex matrix of the petroleum, the extraction time was fixed at 5 min. In this experiment, extraction efficiencies of different power (30, 60 and 90 W) by using four extractants were investigated (shown in Fig. 4). For all of the four extractants, the extraction efficiency by microwave assisted extraction at 30 W were similar to those at 90 W, while the efficiency at 60 W were higher than those at the other two sets of extraction power. It is therefore perceived that the extraction efficiencies increased at first and then decreased

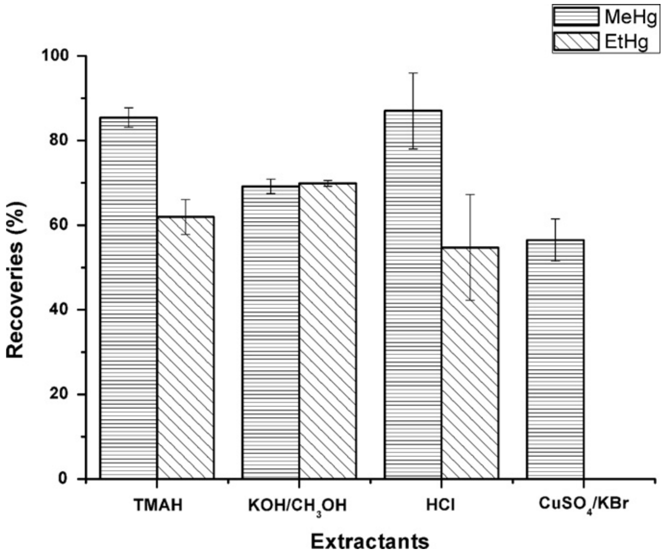

Fig. 3. Extraction efficiency of the ultrasonic extraction for MeHg and EtHg using different extraction solvents.

with the increase of extraction power. It could be concluded that 60 W is more suitable for the extraction of both MeHg and EtHg. 30 W might be unsufficiently powerful to extract both MeHg and EtHg from petroleum, and it seems that the organic mercury could be decomposed when the extraction power was increased to 90 W.

At the microwave power at 60 W, KBr/$CuSO_4$ solution (3:1, *v*/*v*) was the most efficient extractant for MeHg among the four extraction solvents, but the most inefficient one for EtHg. Moreover, the KOH/CH_3OH solution provided moderate extraction efficiency for both MeHg and EtHg. The efficiency of both TMAH and HCl extraction were satisfactory, but TMAH was the more appropriate extractant because the extraction efficiency of MeHg using TMAH was higher than that using HCl. Moreover, it is usually considered that MeHg is more toxic than EtHg and should thus be given higher priority.

It could be concluded that the extraction efficiency of the microwave extraction (at 60 W) was more effective than those of shaking or ultrasonic extraction by comparing the recoveries of different extraction methods. These results indicated that the organic mercury could be released more completely from petroleum with the assistance of microwave irradiation. It is however difficult to determine whether alkaline or acid digestion is more appropriate for the extraction of MeHg and EtHg from petroleum because no clear pattern was found by the different extractants.

As a result, microwave assisted extraction at the power of 60 W for 5 min using TMAH as the extractant was considered the optimized condition to extract both MeHg and EtHg from petroleum.

3.3. Determination of total Mercury and speciation of organic Mercury in petroleum

Four crude oil samples, three gasoline samples and one diesel sample were analyzed by using the proposed method, and the results are shown in Table 3. The concentrations of total mercury in four crude oil ranged from 13.4 to 1252.8 ng g^{-1} while the concentrations of MeHg were all at the sub-ppb level, and EtHg was not detected at all in the four samples. The four petroleum samples were collected from different locations of China, and thus the concentration and pattern of mercury were quite different. This indicated that the distribution of mercury in petroleum can be affected by the natural geological conditions [27] and it has been found that the dominant dissolved species were mainly elemental mercury and ionic halides [6]. Besides the four crude oil samples, four light oils were analyzed also, and organic mercury

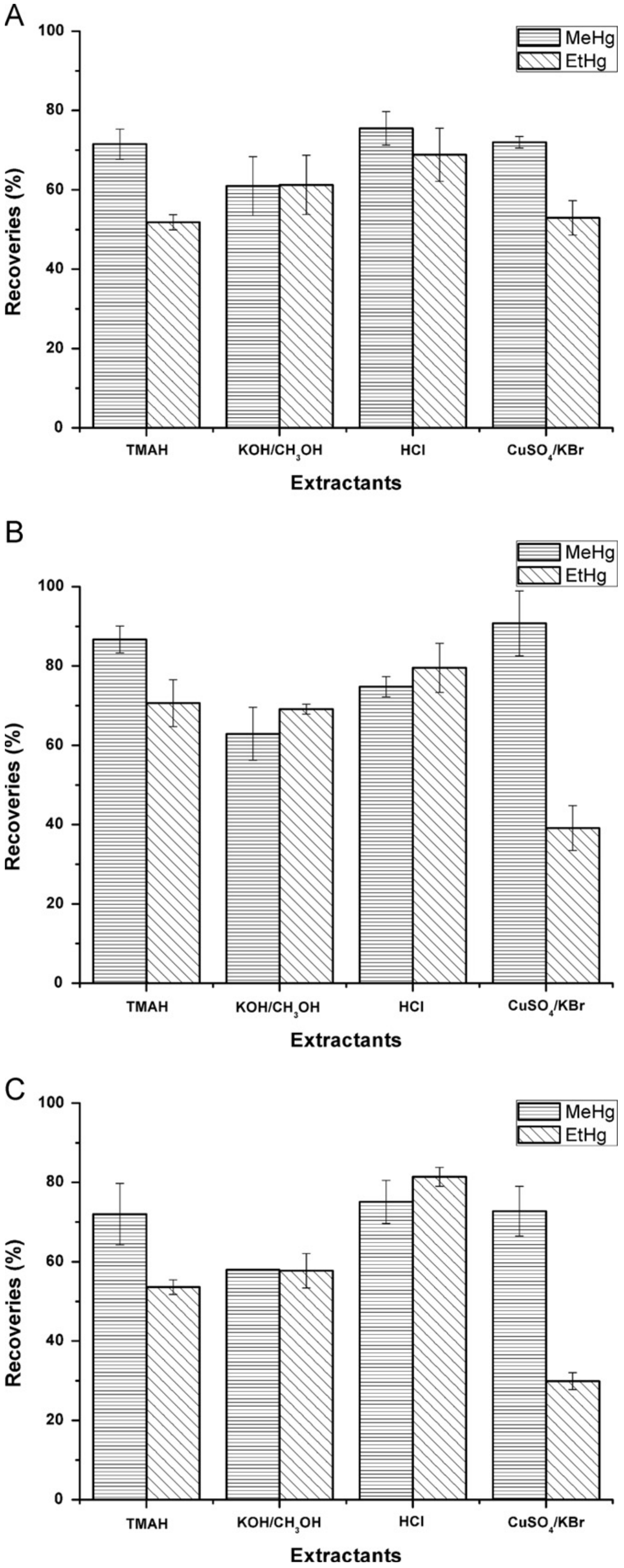

Fig. 4. Extraction efficiency of the microwave assisted extraction at different powers for MeHg and EtHg using different extraction solvents. (A). 30 W; (B). 60 W; (C). 90 W.

Table 3
Hg speciation in several petroleum samples.

Sample ID	Total Hg(ng g^{-1})	Methylmercury (ng g^{-1})	Ethylmercury (ng g^{-1})
Crude oil no 1	1258.8	0.515	ND[b]
Crude oil no 2	118.8	0.119	ND
Crude oil no 3	13.4	0.0276	ND
Crude oil no 4	36.2	0.184	ND
Gasoline no 5	0.26	N.D.	ND
Gasoline no 6	N.D.	N.D.	ND
Gasoline no 7	N.D.	N.D.	ND
Diesel no 8	N.D.	N.D.	ND

[b] ND: no detection.

Table 4
The concentrations of Hg in different oil samples.

Number of samples	Concentration range (ng g^{-1})	Concentration of THg (ng g^{-1})		Analysis method	Ref.
		Mean (ng g^{-1})	SD		
76	NR[c]	1505	3278	GC-AFS	[5]
11	1.60–6.60	4.45	1.72	TD-AFS	[14]
2	28.8, 31.8	30.3	2.12	ID-ICP-MS	[18]
7	0.10–12.2	3.09	4.24	INAA	[28]
10	22.54–29688.0	3236.5	9315.4	INAA	[29]
4[d]	13.4–1258.8	356.8	603	CV-AFS	This work

[c] NR: not reported in the paper.
[d] 4: only four crude oil samples were counted.

species were not detected while the total mercury could be detected only in one sample (0.26 ng g^{-1}) and MeHg and EtHg were not detected. This result shows that most of the mercury could be removed in the refining process for light oil.

Since there is no certified reference material of petroleum or similar matrix for mercury quantification, it is only possible to evaluate the analytical performance of the proposed method by the recoveries of digestion and extraction. For total mercury detection, a few microliters Hg standard was added to the sample to make the concentration of total mercury increased 500 ng mL^{-1}. For the speciation of mercury, concentrations of both MeHg and EtHg were increased 0.5 ng mL^{-1} by adding MeHg and EtHg standard. The samples with standard were incubated for 24 h and then digestion and extraction experiments were repeated at least three times. The recoveries for total mercury ranged from 85% to 91% and the recoveries for extraction experiments were 86.7% $\pm$ 3.38% and 70.6% $\pm$ 5.92%, for MeHg and EtHg, respectively. These results showed that the proposed method could be both accurate and reliable.

3.4. Estimation the Mercury emissions from petroleum consumption

The concentrations of total mercury in crude oil ranged from 13.4 to 1252.8 ng g^{-1} in this work, and the average concentration was 356.8 ng g^{-1}. Table 4 shows the content of mercury in different literatures. These results imply that the mercury concentrations in crude oil can occur in a wide range, and the average concentration of all the crude oil was 1348 ng g^{-1}, which is quite close to the result by Wilhelm et al. (1500 ng g^{-1}) [6].

Both petroleum and coal are the most important sources of energy, and the mercury in the atmosphere is mainly from the consumption of oil and coal. People have been more concerned about the mercury emissions of coal in the past mainly due to the lack of effective methods for detecting mercury in oil. Due to the huge and growing consumption of petroleum, the risk of mercury emissions from petroleum consumption should be considered. The mean concentration of mercury in coal is about 200 ng g^{-1} [30], while the mean concentration is 1500 ng g^{-1} for crude oil [6]. It indicates that the petroleum contains a higher level of mercury and might cause higher mercury emissions. It has been shown that the atmospheric mercury emission from the combustion of liquid hydrocarbon fuels is approximately 10% of the annual emission rate from coal combustion in America [7]. However, because of the lack of sufficient data, the global mercury emission caused by petroleum consumption could not be accurately calculated. However, the mercury emission caused by petroleum would increase with the growth of oil consumption and this emission source should not be overlooked. Therefore appropriate mercury-removal protocol should be established before the crude oil is used.

4. Conclusions

In this work, different extraction methods for extracting organic mercury species including methylmercury and ethylmercury from petroleum by using different extraction solvents were developed and compared. Microwave assisted extraction for 5 min at the power of 60 W with TMAH as the extractant was the proposed condition to extract both MeHg and EtHg from petroleum. The extraction efficiency for both MeHg and EtHg were satisfactory by this method. The proposed process could be used as the protocol for routing analysis of organic mercury in petroleum and light oils.

Several petroleum and light oil samples were analyzed, and the results indicated that the mercury concentrations in oils can be affected by its type and location. More oil samples will be analyzed in the future works and the risk of mercury in petroleum should be evaluated more extensively.

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