

Solid-phase preconcentration and determination of mercury(II) using activated carbon in drinking water by X-ray fluorescence spectrometry

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A novel and simple method was developed for the preconcentration and determination of mercury(II) from hydrochloric acid solutions through their adsorption on Aliquat 336 (tri-octylmethylammonium chloride)-activated carbon. The determinations were made directly on the solid by X-ray fluorescence spectrometry, which had the advantage of eliminating the step of elution of the mercury retained.

This preconcentration system enabled the determination of Hg(II) in drinking water samples at $\mu\text{g l}^{-1}$ levels. A 1700-fold enrichment factor was obtained. The described methodology showed excellent reproducibility, accuracy, and detection limits improvement by eliminating the step of elution of the analyte, replacing those methods based on cold vapor generation, reducing reagent consumption, and handling of samples. Copyright © 2013 John Wiley & Sons, Ltd.

Introduction

Mercury can be present as a trace contaminant in all environmental compartments as a result of both natural origin and anthropogenic activities, and the determination of this element is of considerable interest because of its toxicity and ability of bioaccumulation in many organisms.^[1] It is well known that organomercury compounds are much more toxic than mercury in its inorganic forms. It has been found that mercury may accumulate itself in vital organs and tissue such as the kidney and brain.^[2] Mercury may enter in the human body by inhalation of mercury vapor (mainly in the form of Hg^0), drinking water (mainly as inorganic mercury, Hg^{2+}), and/or by the consumption of fish and fish products (mainly as methyl mercury, CH_3Hg^+). The toxicological implications of the mercury contents in the environments have encouraged the development of very sensitive methods for its determination.^[3,4]

Because of the extremely low levels of mercury in various matrices, e.g. the upper limit for total mercury concentration in drinking water recommended by the European Community is $1 \mu\text{g l}^{-1}$,^[5] highly sensitive and sophisticated analytical procedures are required for its determination; the most commonly used ones are cold vapor generation–atomic absorption spectrometry,^[6,7] cold vapor generation–atomic fluorescence spectrometry,^[8,9] and inductively coupled plasma mass spectrometry.^[10] In addition, a preconcentration step has usually been needed for mercury determination in environmental samples.^[11]

X-ray fluorescence (XRF) spectrometry has also been used for analysis and speciation of Hg(II).^[12,13] The main advantage of this technique is its capability for direct analysis of solid and liquid samples, avoiding sample handling or at least reducing it to a minimum. However, XRF has a number of limitations when dealing with aqueous samples, such as short linear range, the requirement of closely matching standards to overcome matrix effects, and poor sensitivity. The use of preconcentration materials as retention media for the analytes of interest offers the possibility to overcome these problems, ensuring lower limits

of detection, extending the linear range, and unifying matrix effects by fixing the same conditions for both standards and samples. Moreover, it has to be taken into consideration that unlike other methods, in which an elution step is required to recover the metal from the solid sorbent used to preconcentrate, X-ray spectrometry offers the possibility of direct quantification of metal species held in the solid sorbents, reducing the number of chemicals required and minimizing sample handling.

Solid-phase extraction is the most common technique used for the preconcentration of analytes in environmental waters because of its advantages of high enrichment factor (EF), high recovery, rapid phase separation, low cost, low consumption of organic solvents, and the possibility of combination with different detection techniques in on-line or off-line mode.^[14]

Many sorbents have been used for the preconcentration of metal ions.^[15–22] Activated carbon (AC) is still by far the most important in current use in the environmental pollution control because of its large surface area, high adsorption capacity, porous structure, selective adsorption, and high purity.^[23] However, without any surface treatment, AC presents an adsorption capacity for metal ions from fair to as low as none, because metal ions often exist in solution either as ions or as hydrous ionic complex.^[24] For this reason, modification and impregnation techniques have been used to increase the surface adsorption and the removal capacity in order to add selectivity to AC.^[25–30]

The usefulness of amines as convenient extractants for removal ions from aqueous solutions is usually considered to depend essentially on the ability of metal ions to form anionic species in the aqueous phase, which are extracted by amines

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according to an anion-exchange process. In the case of mercury (II) in hydrochloric acid solutions, the following metal species may exist in the aqueous phase: Hg^{2+} , HgCl^+ , HgCl_2 , HgCl_3^- , and HgCl_4^{2-} .^[31,32] In this work, a novel method was developed for the preconcentration and determination of mercury(II) from hydrochloric acid solutions through their adsorption on Aliquat 336-AC. The determinations were made directly on the solid by XRF spectrometry, which had the advantage of eliminating the step of elution of the mercury retained. A high preconcentration factor was achieved for a very low sample volume.

Experimental

Reagents and apparatus

The extractant tricaprylmethylammonium chloride ($\text{C}_7\text{H}_{15}\text{N}[(\text{C}_7\text{H}_{15})_3\text{N}^+\text{CH}_2\text{CH}_2]_3\text{Cl}^-$; Aliquat 336) was purchased from Fluka, Switzerland. A stock mercury standard ($1000 \mu\text{g ml}^{-1}$) was prepared from mercury(II) chloride (Merck, Darmstadt, Germany) in nitric acid (Merck, Darmstadt, Germany) and made to 1000 ml with ultrapure water. Ultrapure water ($18.1 \text{ M}\Omega \text{ cm}^{-1}$) was obtained from Barnstead (Iowa, USA) EASYpure Reservoir Field water system.

All reagents were of analytical reagent grade, and the presence of mercury was not detected within the working range.

Measurements were performed with a Philips PW1400 XRF spectrometer. The Hg L_{α} line was used for measurements. The conditions were appropriately optimized and are shown in Table 1. The pH of the solutions was measured using an Orion 701-A pH meter with an Ag/AgCl electrode. An M-23 digital orbital (Buenos Aires, Argentina) was employed for sample agitation.

Preparation of the activated carbon

The AC (Merck; 200 meshes) was used after pretreatment with acid.^[33–35] AC was heated at a temperature of 60°C with 10% (v/v) hydrochloric acid (Merck) for 30 min, then with 10% (v/v) nitric acid (Merck) for 20 min, and finally washed with deionized water until neutral pH.^[36]

Preparation of the loaded activated carbon

Under the optimized adsorption conditions, the anion-exchanger extractant was loaded on the ground AC. Thus, an AC portion (1 g) was shaken for 12 h with 40 ml of a 5% Aliquat solution in methanol at room temperature. The AC was then filtered, washed with distilled water, and dried in an oven at 110°C .

Table 1. Main instrument parameters for Hg determination

Parameters	
Line	Hg L_{α} 35,61 2θ
Tube	Rh
Crystal	LiF 200 (2d = 4,028 Å)
Collimator	F (fine)
Operating conditions of the tube	75 kV, 40 mA
Mask	2 (14 mm)
Upper level of the windows	70%
Lower level of the windows	15%
Detector	F (flow) or S (scintillation)
Counting time	100 s

Batch adsorption experiment

A series of standards or sample solutions containing Hg(II) were transferred into 25-ml beakers, and the pH was adjusted to the desired value with either 0.1 mol l^{-1} HCl or 0.1 mol l^{-1} NaOH. After evaluation, the best adsorption conditions were achieved at a pH value of 1.5, which was selected for further experiments. After pH adjustment, the volume was made to 50 ml with ultrapure water and 100 mg of Aliquat 336-AC was added. This mixture was then placed in a shaker for 10 min to facilitate adsorption of the metal ions onto the sorbent.

Preparation of the pellet

Tablets were made from boric acid, which was deposited as a powder on a portion of 100-mg Aliquat 336-AC-Hg(II) and then compacted with a hydraulic press, always working under infinite thickness conditions.^[37] Thus, speed was achieved by avoiding the traditional step of elution of the retained mercury.

A series of solutions and appropriately diluted standards of Hg in water and synthetic water samples for human consumption were preconcentrated on the Aliquat 336-AC by the method described earlier.

Results and discussion

Effect of shaking time on the sorption

The shaking time is an important factor in determining the possibility of application of Aliquat 336-AC to the extraction of metal ions. In this work, considering the percentage of extraction of Hg(II) on Aliquat 336-AC, different shaking times ranging from 1 to 30 min were studied. As previously reported by Catrall and Daud,^[38] the reaction equilibrium was rapidly reached.

Results shown in Fig. 1 demonstrate the rapid extraction reaction of Hg(II) with Aliquat 336; the maximum signal was obtained after 5 min of stirring; thus, a 10-min stirring period was selected.

Effect of pH on the retention

As previously mentioned, the extraction of metals by amines as well as by cationic extractants is affected by the pH. The effect of pH on the extraction of Hg(II) with Aliquat 336 is shown in

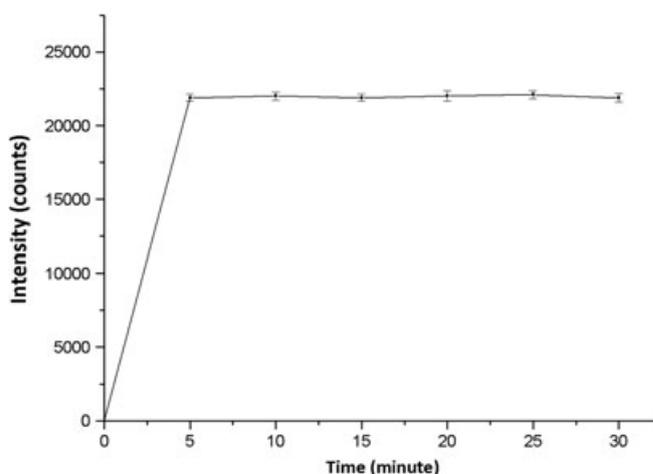


Figure 1. Effect of shaking time on the adsorption of 0.5 mg l^{-1} Hg(II) on Aliquat 336-AC.

Fig. 2. To evaluate the effect of pH, 50-ml solutions containing 0.5 mg l^{-1} of Hg(II) were adjusted to different pH values. They were mechanically shaken with 100-mg portions of Aliquat 336-AC for 10 min. The Aliquat 336-AC was then isolated by filtration and washed with distilled water, and later, the L_x lines of the analyte were measured by the XRF spectrometer. As shown in Fig. 2, mercury extraction improved as pH increased and became practically constant at pH higher than 1 (Hg extraction percentage more than 95%).

On the other hand, satisfactory mercury extraction efficiency at a low pH value is an interesting property to consider and achieve in order to obtain a selective enough separation method for mercury from other heavy metals, which are preferentially extracted at higher pH values. A similar behavior on selective pH mercury extraction has been found elsewhere using diverse extractants.^[38–40] On the basis of these results, pH 1 was selected for further experiments.

Sample volume and preconcentration factor

Because of the importance of obtaining high preconcentration factors, the effect of sample volume on recovery of Hg on the solid phase was also examined. The results are depicted in Fig. 3, Hg(II) ions were quantitatively (>95%) recovered within a volume range from 50 to 250 ml. Therefore, this range was adopted for the separation/preconcentration of Hg.

Determination of the maximum retention capacity of the Aliquat 336-AC

Solutions containing different concentrations of Hg(II) were adjusted to pH 1, shaken, and filtered to determine the quantity of analyte that saturated the Aliquat 336-AC material. The metal L_x line was measured by XRF. XRF intensity of Hg L_x as a function of the Hg(II) concentration is shown in Fig. 4. For very low amounts of Hg(II), the concentration was proportional to the Hg L_x intensity. Thus, the curve was linear up to 20 mg l^{-1} of Hg(II) and became constant at higher Hg concentrations. The total retention capacity was determined to be 200 mg of Hg(II) per gram of sorbent material. The amount retained was always greater than 95%.

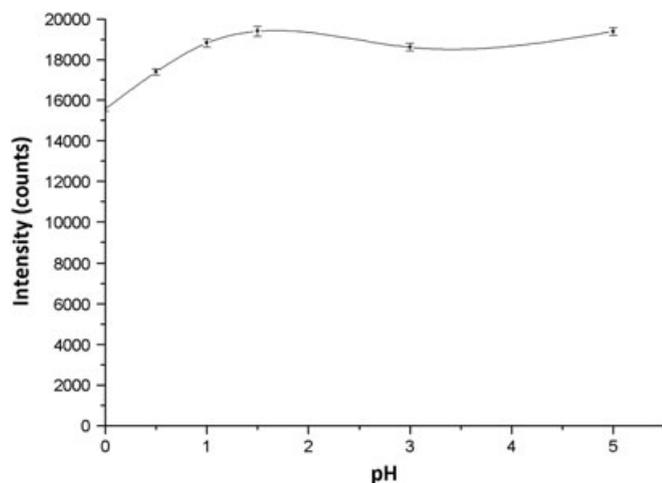


Figure 2. Effect of pH on the adsorption of 0.5 mg l^{-1} Hg(II) on Aliquat 336-AC.

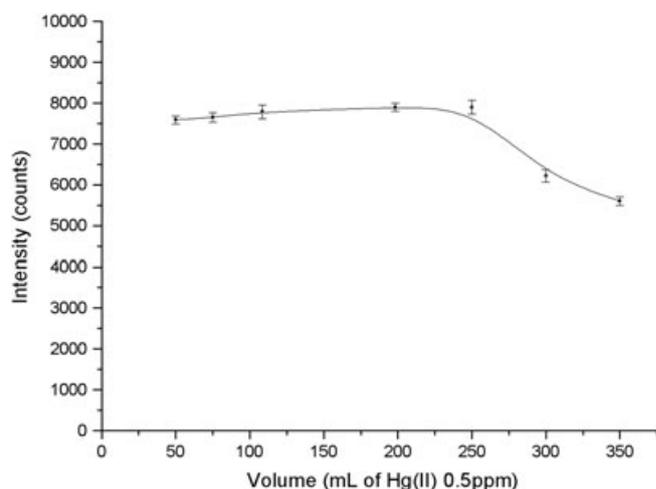


Figure 3. Effect of the sample volume on the solid-phase extraction recovery of Hg(II).

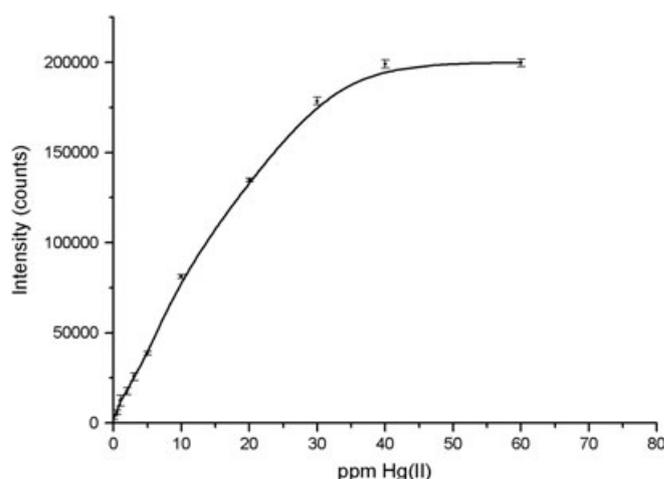


Figure 4. Retention capacity of Aliquat 336-AC for Hg(II).

Analytical performance

The EF is defined as $EF = [QT/QM]/[QT_0/QM_0]$,^[41] where QT_0 and QT are the analyte quantities before and after the preconcentration, respectively, and QM_0 and QM are the quantities of the matrix before and after the enrichment, respectively. The EF was calculated considering that the tablets obtained from the 50-ml solution of $500 \mu\text{g l}^{-1}$ Hg(II) had a final thickness of 0.64 mm and a final diameter of 16.7 mm; the volume deposited on the pellet was 0.14 cm^3 . The preconcentration factor obtained was 350-fold, which represented a highly satisfactory value for trace analysis by XRF. When working with the initial 100-ml and 250-ml sample volumes, preconcentration factors of 700-fold and 1700-fold, respectively, were obtained.

One of the most important features of the use of the proposed preconcentration method was the improvement of the detection limits. Various Hg preconcentration procedures have been reported in the scientific literature. Table 2 shows a comparison among the most influential ones.

Calibration was performed against aqueous standards prepared under the same preconcentration conditions described earlier.

Table 2. Comparison of several procedures for preconcentration and determination of Hg(II)

Type of samples	Detection limit($\mu\text{g l}^{-1}$)	Enrichment factor	Sample amount(ml)	Technique	References
Drinking water	0.001	700	100	XRF	This work
Biological samples	0.01	22	0.2	ETAAS	[2]
Water samples	0.004	50	50	FI-ICP-OES	[5]
Biological and environmental samples	0.01	13	25	CV-AAS	[11]

CV-AAS, cold vapor generation-atomic absorption spectrometry; ETAAS, electrothermal atomic absorption method; FI-ICP-OES, flow injection-inductively coupled plasma optical emission spectrometry; XRF, X-ray fluorescence.

A detection limit, calculated as $(3/m)(I_b/t)^{1/2}$, where m is the slope of the calibration curve, I_b is the background intensity (counts s^{-1}), and t is the counting time (s), of 1 ng l^{-1} was obtained. The precision, expressed as relative standard deviation, for ten replicate determinations at 0.5 mg l^{-1} Hg(II) level was 4.0%.

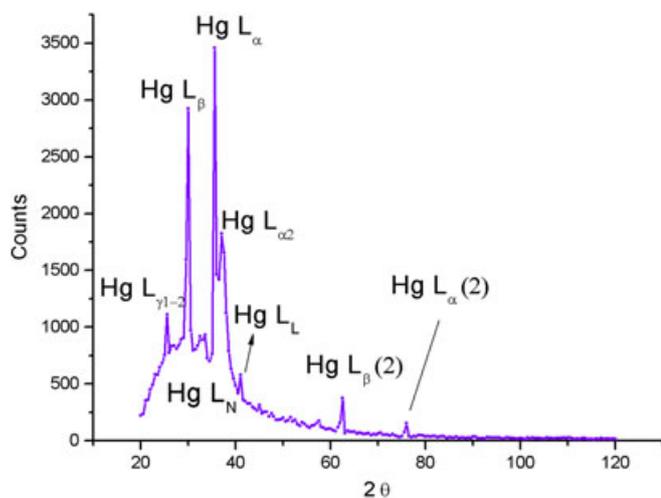
Recuperation and validation studies

To evaluate and demonstrate the validity of this method, 500 ml of drinking water sample was collected in our laboratory and divided in ten aliquots of 50 ml each. The proposed methodology was applied to six portions, and the average quantity of mercury obtained was taken as a base value. Then, increasing quantities of mercury were added to the other aliquots, and after that, mercury was determined by the same method. The results are shown in Table 3. Other elements present in the sample did not

Table 3. Concentrations of Hg(II) in water for human consumption (95% confidence level; $n=6$)

Base ($\mu\text{g l}^{-1}$)	Hg(II) added ($\mu\text{g l}^{-1}$)	Hg(II) found ($\mu\text{g l}^{-1}$)	Recovery (%) ^a
0.0	0.0	0.0	—
0.0	0.5	0.497 ± 0.04	99.4
0.0	1.0	0.985 ± 0.03	98.5
0.0	1.5	1.51 ± 0.05	100.6

^a $[(\text{Found} - \text{base})/\text{added}] \times 100$.

**Figure 5.** Scanning X-ray fluorescence spectrum of a sample of water of 0.5 mg l^{-1} Hg(II) on Aliquot 336-AC under the proposed preconcentration methodology.

show spectral interferences in the Hg determination. The study of possible interferences was performed by scanning an XRF spectrum of a sample of water under the proposed preconcentration methodology (Fig. 5).

Conclusions

The preconcentration methodology here presented enabled the determination of Hg(II) at ng l^{-1} levels in drinking water samples. In addition to the 1700-fold EF achieved, the analyte elution step was not required; these aspects allowed a substantial improvement of Hg detection limit by XRF. The methodology showed excellent reproducibility and accuracy and constituted an interesting and valid alternative of replacement of those methods based on Hg determination by cold vapor generation approaches. In addition, reagent consumption reduction and sample handling minimization are important advantages to be highlighted from the proposed methodology.

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