

Inorganic speciation of As(III, V), Se(IV, VI) and Sb(III, V) in natural water with GF-AAS using solid phase extraction technology

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Abstract

The paper presents a procedure for the multi-element inorganic speciation of As(III, V), Se(IV, VI) and Sb(III, V) in natural water with GF-AAS using solid phase extraction technology. Total As(III, V), Se(IV, VI) and Sb(III, V) were determined according to the following procedure: titanium dioxide (TiO₂) was used to adsorb inorganic species of As, Se and Sb in sample solution; after filtration, the solid phase was prepared to be slurry for determination. For As(III), Se(IV) and Sb(III), their inorganic species were coprecipitated with Pb-PDC, dissolved in dilute nitric acid, and then determined. The concentrations of As(V), Se(VI) and Sb(V) can be calculated by the difference of the concentrations obtained by the above determinations. For the determination of As(III), Se(IV) and Sb(III), palladium was chosen as a modifier and pyrolysis temperature was 800 °C. Optimum conditions for the coprecipitation were listed for 100 ml of sample solution: pH 3.0, 15 min of stirring time, 40.0 μg l⁻¹ Pb(NO₃)₂ and 150.0 μg l⁻¹ APDC. The proposed method was applied to the determination of trace amounts of As(III, V), Se(IV, VI) and Sb(III, V) in river water and seawater.

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1. Introduction

In recent years, speciation analysis of As, Se and Sb is very attractive due to the toxicity of their combinations. The toxicity of the different species for the same element is different, in which the inorganic compounds are more toxic than the organic compounds and the toxicity of As(III), Se(IV) and Sb(III) is stronger than that of As(V), Se(VI) and Sb(V) [1–3]. In addition, selenium has been also reported as an essential element for human body at low concentration [2]. Therefore, a high sensitive and simple method is necessary for determining the concentration of the different oxidation state of the inorganic compounds for these elements present in environment. With volatilization for their hydrides, hydride generation (HG) as a sampling tech-

nology, which is coupled with the detected systems, such as atomic absorption spectrometry (AAS) [4], inductively coupled plasma atomic emission spectrometry (ICP-AES) [5] and inductively coupled plasma mass spectrometry (ICP-MS) [6,7], has been applied in analytical field. Though requirement for the determination of trace level elements is met for HG, speciation analysis for these elements is difficult because the sampling technology has trouble separating the different oxidation state for a single element in complicated matrix. With introduction of separation technique, chromatography coupled with the above instruments, the hyphenated systems, has been utilized to perform speciation analysis. Because of high performance liquid chromatograph (HPLC) with excellent separation for species in aqueous samples, there are a great many reports [1,8–14] on these elements with HPLC-AAS, HPLC-ICP-AES and HPLC-ICP-MS. However, HPLC are suitable for separating species in aqueous sample, but it cannot perform enrichment for the detected element. Therefore, for ultratrace As, Se and Sb in natural waters, the coupled detectors except for ICP-MS, are poor in sensitivity; ICP-MS with high sensitivity is too expensive for the

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most researchers to be equipped. Furthermore, the combination of instruments makes the determined procedure more complex and the continuous determining mode is also not suitable for GF-AAS.

With development of filtration technology, some macromolecule compounds can be collected on filtration membrane with the precipitation of the similar structure compound [15–17]. Owing to the selectivity of macromolecule for the specific oxidation state of some elements, the species of the different oxidation state can be separated during the stage of pretreatment. APDC as a typical macromolecule has been utilized to selectively collect these inorganic ions of As(III), Se(IV) and Sb(III). In the method, As(III), Se(IV) and Sb(III) are determined by using APDC; total concentration for each element is determined according to the same method after As(V), Se(VI) and Sb(V) are reduced to As(III), Se(IV) and Sb(III) [15,16,18]. The speciation procedure for multi-element is mainly used for neutron activation analysis, but for GF-AAS, there are only a few available for single element [15]. Different from the detector and determination of total concentration in the above multi-element analysis, a method has been proposed on simultaneous determination of total As, Se and Sb with GF-AAS by TiO₂-slurry sampling in previous paper [19]. In the study, speciation of these elements in aqueous samples was performed by simultaneously determining total concentration for each element [19] and the concentration of As(III), Se(IV) and Sb(III) according to the subsequent analytical procedure.

The aim of the study is to perform the determination of As (III, V), Se(IV, VI) and Sb(III, V) in the environmental water samples. The optimal conditions for coprecipitation including furnace condition, effect of modifier, pH of preconcentration, and coexisting ions were investigated in detail. The proposed method was applied to river water and seawater.

2. Experimental

2.1. Instrumentation

A simultaneous multi-element graphite furnace atomic absorption spectrometer, SIMAA 6000, from Perkin-Elmer was used with a transversely heated graphite atomizer (THGA), longitudinal Zeeman-effect background correction and an AS-72 autosampler. Standard THGA graphite tubes (Part no. B0504033) and Perkin-Elmer 'EDL system 2' electrodeless discharge lamps (As: 193.7 nm, 380 mA; Se: 196.0 nm, 290 mA; Sb: 223.1 nm, 380 mA) were applied. The measurements were in peak area mode (integrated absorbance). A pH meter (HM-26S) manufactured by DKK-TOA Corporation and an analytical balance (Sartorius model BP301S-EA BR-1010, 300 g × 0.0001 g) were employed. Ultrasonic (Branson 12, branson) was used to disperse the slurry to improve adsorption and homogeneity of slurry. A magnetic stirrer (ADVANTEC SR 506) was used to stir. A Milli-Q Water System, Millipore, was employed to produce ultrapure water. In addition, before being used, all glassware, pipette tips and storage bottles were soaked in dilute HNO₃ for 24 h and rinsed at least three times according to the order of water, purity water and ultrapure water.

2.2. Reagents and materials

All the reagents were of analytical grade. The standard solutions of As(III), Sb(III) and Sb(V) (100 μg l⁻¹) were prepared on a daily basis by diluting appropriate aliquots of a 1000 mg l⁻¹ stock metal solution, respectively. The stock solutions (1000 mg l⁻¹) of As(V) and Se(VI) were separately prepared by dissolving the corresponding salts (Na₂HAsO₄·7H₂O and Na₂SeO₄) and adding 0.75 ml of 60% nitric acid and 1.0 ml of 30% hydrogen peroxide. The stock solution (1000 mg l⁻¹) of Se(IV) was obtained by dissolving Na₂SeO₃. These solutions were properly diluted to use. The two stock solutions of Sb(V) and Sb(III) with 20% HCl was from SPEX CERTIPREP LTD. Ammonium pyrrolidinedithiocarbamate (APDC, 10,000 mg l⁻¹) solution was prepared by weighing 1.0 g of APDC powder and dissolving in 100 ml of ultrapure water. Pb(NO₃)₂ (4000 mg l⁻¹) solution was obtained by dissolving 0.4 g of Pb(NO₃)₂ in 100 ml of ultrapure water. Hydrochloric acid (35%), nitric acid (60%) for poisonous metal analysis and sulfuric acid were from KANTO CHEMICAL CO. INC. Titanium(IV) dioxide (anatase form, 99%, -0.5 μm) was utilized as a sorbent. ZrO(NO₃)₂·2H₂O was employed to coat the graphite tube. A river water sample was collected in the Edokawa river in Tokyo and a seawater sample was obtained from Inage coast in Chiba in Japan. The samples were filtrated with a 0.45 μm pore size cellulose acetate membrane filter (Advantec, Tokyo) and treated for determination in 24 h. The above reagents without the source and the other reagents used in this work were from Wako Co. in Japan.

2.3. Analytical procedure

2.3.1. Determination of total As, Se and Sb [19]

The enrichment ratio of 20 was used in the study. Hundred-milliliter of sample solution with less than 5.0 μg l⁻¹ of As, Se and Sb was placed in a beaker; pH was adjusted to 2.0 with 2.0 M HNO₃. The sample solution added 20.0 mg of TiO₂ was stirred by a magnetic stirrer about 10 min, and immersed in a sonication bath about 10 min. After the solution was filtrated with a 0.45 μm membrane filter, the remained solid phase was transferred to a 15.0 ml centrifugal tube, and then 5.0 ml ultrapure water was added to form 5.0 ml of the slurry sample. Then, 20 μl of the slurry was injected into a graphite tube by an autosampler for each cycle.

2.3.2. Determination of As(III), Se(IV) and Sb(III)

The enrichment ratio of 20 was used for investigating the influential factors for coprecipitation. Hundred-milliliter of sample solution with less than 5.0 μg l⁻¹ of As(III), Se(IV) and Sb(III) was placed in a beaker; pH was adjusted to 3.0 with 1.0 M HCl. 1.5 ml of 10,000 mg l⁻¹ APDC and 1.0 ml of 4000 mg l⁻¹ Pb(NO₃)₂ solution was added and stirred for 15 min. After filtration with a 0.45 μm membrane filter, the remained solid phase was transferred to a 15.0 ml centrifugal tube and dissolved in 1.0 ml HNO₃ (1:1) solution; the solution was diluted to 5.0 ml. Then 20 μl of sample solution was injected into a graphite tube,

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