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Detection tube method for trace level arsenic

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ABSTRACT

Arsenic pollution of surface and ground waters has been reported in many developing countries, and it is therefore an important task to detect arsenic rapidly using a simple and inexpensive tool. This work focused on the detection of arsenic at 0.01 mg As L^{-1} by visual determination. A small column packed with the poly(vinylchloride) particles coated with a quaternary ammonium salt was used as a detection tube. Molybdoarsenic heteropoly acid (molybdenum blue) was derived from arsenate under modified reaction conditions. The molybdenum blue solution (20 mL) was introduced into the detection tube by suction with a syringe to form color band. As(III) was measured after oxidation with sodium dichloroisocyanuric acid. The color band length in the detection tube was correlated linearly with the arsenic (As(III) + As(V)) concentration in the range of 0.01–0.1 mg As L^{-1}, and the relative standard deviations in the concentration range were around 1%. Arsenic was successfully detected at 0.01 mg As L^{-1} using this detection tube.

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Introduction

Arsenic pollution in potable drinking water has been observed around the world, and many people have been afflicted with serious arseniasis [1–6]. It is therefore an urgent subject for the people in these regions to employ arsenic removal technologies to decontaminate drinking water supplies and to monitor arsenic pollution levels of the surface and groundwater. Common arsenic removal technologies include precipitation and adsorption with a metal oxide such as iron oxide [7–11]. Hydrotalcite compounds are also useful adsorbents for As(V) [12,13]. In these methods, As(III) is removed after oxidation to As(V), and hence, it is an important consideration in the monitoring of the As(V) concentration in treated water.

The World Health Organization (WHO) has recommended an acceptable arsenic level for drinking water at 0.01 mg As L^{-1}. As(V) concentration can be detected by the molybdenum blue (MB) method, which is similar to the phosphate detection method. However, the MB method cannot directly detect As(V) at 0.01 mg As L^{-1}, and preconcentration of arsenic is necessary because of extremely low absorbance of MB at this concentration level [14–17]. Therefore, atomic absorption spectrometry (AA) or an inductively coupled plasma atomic emission spectrometry (ICP-AES) is commonly used for detecting low level arsenic. These analytical methods are not convenient for rapid monitoring. A rapid and simple detection method is required for the following cases: on-site checking of risk level of source water for drinking use and of effluents from small-scale arsenic removal plants. Even for field research, a rapid monitoring tool may be useful.

Some kinds of spot test kits for detecting arsenic are available including a test strip type and a gas-detection tube type [18,19] based on the generation of arsine. These spot tests enable visual determination, but the accuracy is usually low because the detected concentration increases exponentially with the increase of color intensity or color band length.

In our previous works [20–24], detection tube methods were developed for the monitoring of phosphate, nitrite/nitrate or ammonium. The color band was formed by ion-pair formation between anionic colored compound developed with analyte and quaternary ammonium salt coated on polyvinyl chloride (PVC) particles. The methods needed two steps, i.e., the color development and the color band formation, but higher accuracy was obtained because of the linear relationship between the analyte concentration and the color band length in the detection tube. In addition, it was indicated that the detection range can be easily controlled by modification of the preparation condition of the adsorbent and/or the measurement conditions [24].

In this study, we focused on the visual detection of As(V) at 0.01 mg As L^{-1} using a detection tube method in order to enable visual determination with an inexpensive analytical kit. The color

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development was caused by molybdenum blue (MB) method because of the anionic character of molybdoarsenic heteropoly acid. However, the reaction rate of molybdoarsenic heteropoly acid formation is slower that that of phosphate, and therefore the conditions of the color development reaction were modified. The reaction conditions controlling the disturbance with co-existing components and the procedure of As(III) detection were also examined.

Experimental

Reagents and solutions

Standard solutions: arsenate and arsenite standard solutions were prepared with $Na_2HAsO_4 \cdot 7H_2O$ and with As_2O_3, respectively, and the concentration range was 0.002–1.0 mg As L^{-1}.

Reagent A: 3.6 g of ammoniummolybdeate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$), 0.0822 g of potassium antimonyl tartrate ($K(SbO)C_4H_4O_6 \cdot 1/2H_2O$), and 22 mL of H_2SO_4 were dissolved in 100 mL of distilled-deionized water, where the purchased reagents (reagent grade) were used without further purification.

Reagent B: the ascorbic reagent was prepared with the following method: ascorbic acid (2 g) and NaCl (8 g) were mixed and ground.

Reagent C: the ascorbic solution of 0.7832 g L^{-1} was also prepared.

Reagent D: the dichloroisocyanuric acid (DCI) solution of 0.75 g L^{-1} was prepared with $C_3Cl_2N_3NaO_3$.

All reagents except DCI were obtained from NACALAI TESQUE (Kyoto, Japan), while DCI was from Tokyo Chemical Industry (Tokyo, Japan).

Color development conditions

The concentrations of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$, $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$ and H_2SO_4 contained in the Reagent A varied in the range of 1.2–4.8 g/100 mL, 0.0274–0.1096 g/100 mL and 22–40 mL/100 mL, respectively. One milliliter of the above reagent solution and 0.1 g of the Reagent B were added into the As(V) standard solutions (1.0 and 0.1 mg As L^{-1}), and absorbance at 840 nm was monitored for 60 min with a UV–vis spectrophotometer (V-530, Jasco Co., Tokyo, Japan) equipped with a 5 cm cell, where the temperature of the optical chamber was controlled by a water circulation unit. The reaction temperature was controlled at 50, 60, 70, and 80 °C by circulation of water from a water bath.

Reaction conditions for arsenite oxidations

The Reagent D of 0.4 mL was added into 20 mL of the arsenite standard solution (1 mg As L^{-1}) and oxidation of arsenite was conducted for 10 min after shaking. The reaction temperature was controlled in the range from room temperature to 80 °C. Then, 0.4 mL of Reagent C was added into the reaction mixture to degrade the residual oxidant. One milliliter of the Reagent A and 0.1 g of the Reagent B were added into the reaction mixture and heated at 60 °C for 30 min to develop molybdenum blue. The absorbance at 880 nm of the final solution was measured by a UV–vis spectrophotometer (V530, JASCO, Tokyo, Japan). This procedure was also applied to the arsenate standard solution (1 mg As L^{-1}).

Preparation of packing materials and detection tubes

The adsorbent packed in the detection tube was prepared following the method described in our previous works [21,24]. PVC (particle size: 0.1 mm) was used as a support material and was coated with a mixture of benzylcetyldimethylammonium chloride (BCDMA) and biphenyl by the following procedure: (1) same weight of BCDMA and biphenyl were dissolved in methanol, (2) PVC was added to the solution, and (3) the methanol was evaporated with a rotary evaporator and dried in an oven at 60 °C for 5 h. The contents of BCDMA and biphenyl were 3.0% each. All chemicals were obtained from NACALAI TESQUE (Kyoto, Japan).

The detection tube was made by packing the adsorbent (0.130 g) into poly(propylene) columns (i.d.: 3 mm; length: 60 mm; bed height: 54 mm; a plastic drinking straw), and two pieces of melamine foam were used as stopper of the column.

Procedure of detection tube method

As(V) solution: both the Reagent A (1 mL) and the Reagent B (0.1 g) were added into 20 mL of the As(V) standard solution (0.005–0.1 mg As L^{-1}). The mixture was allowed to react at 60 °C for 30 min followed by cooling in the water bath (10 min). Two milliliters of the colored solution were introduced into the detection tube by suction, where a disposable syringe was connected to the detection tube with a silicon tube. A syringe stopper made of stainless steel was used to stabilize the solution volume introduced into the column. The length of the color band formed in the column was measured with a ruler. Because the front of the color band fluctuated a little, maximum and minimum lengths of the color band were measured, and the average value of both lengths was used as the color band length (CBL).

As(III) solution: the standard solutions (0.005–0.1 mg As L^{-1}) containing both As(III) and As(V) (50/50) were prepared. The 20 mL solution was oxidized by adding 0.4 mL of the Reagent D for 10 min at room temperature. The residual DCI was decomposed by adding 0.4 mL of the Reagent C. The Reagent A (1 mL) and the Reagent B (0.1 g) were added into the mixed solution, and the color development and color band formation were documented using the same procedure as described above.

Results and discussion

Modification of the color development reagent

When molybdenum blue (MB) method was employed for the detection of arsenate, the following two concerns were pointed out: the color development reaction was very slow especially in the case of low arsenate concentration. When the reagent solution mentioned in the US Standard method [14] was employed for 0.1 mg As L^{-1} arsenate solution, stable absorbance of molybdenum blue was obtained after 120 min. Another concern was the instability of the ascorbic acid solution used commonly in the MB method. The solution cannot be stored beyond 1 week.

In order to address the latter, Reagent B was used in this work because solid ascorbic acid is stable and NaCl used as a builder did not influence color intensity of molybdenum blue. Acceleration of the color development was examined by increasing the reagent concentration and raising the reaction temperature.

The basic reagent solution (Reagent X1) was prepared by dissolving 1.2 g of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$, 0.0274 g of $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$, and 22 mL of H_2SO_4 in 100 mL of distilled-deionized water. When this reagent was used, the final concentration of each chemical was equal to that described in US Standard method [14]. The concentrations of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ and $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$ increased as shown in Table 1.

The absorbance of the reaction mixture at 840 nm was monitored for 60 min and the results are shown in Fig. 1. In the case of 1.0 mg As L^{-1} solution, the absorbance reached stable value rapidly by using the Reagents X2 and X3. In the case of 0.1 mg As L^{-1} solution, the reaction rate decreased obviously.

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