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Flow Injection On-Line Sorption Preconcentration Coupled with Hydride Generation Atomic Fluorescence Spectrometry for Determination of (Ultra)trace Amounts of Arsenic(III) and Arsenic(V) in Natural Water Samples

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A flow injection on-line sorption preconcentration and separation in a knotted reactor (KR) was coupled to hydride generation atomic fluorescence spectrometry (HG-AFS) for speciation of inorganic arsenic in natural water samples. The method involved on-line formation of the As(III)−pyrrolidinedithiocarbamate (PDC) complex over a sample acidity of 0.001−0.1 mol L^{-1} HCl, its adsorption onto the inner walls of the KR made from 150-cm long × 0.5-mm i.d. PTFE tubing, elution with 1 mol L^{-1} HCl, and detection by HG-AFS. Total inorganic arsenic was determined after prereduction of As(V) to As(III) with 1% m/v L-cysteine. The concentration of As(V) was calculated by the difference of the total inorganic arsenic and As(III). A 1 mol L^{-1} concentration of HCl was employed not only as the efficient eluent but also as the required medium for subsequent hydride generation. Potential factors that affect adsorption, rinsing, elution, and hydride generation were investigated in detail. The low cost, easy operation, and high sensitivity are the obvious advantages of the present system. With consumption of a 6 mL sample solution, an enhancement factor of 11 and a detection limit (3*s*) of 0.023 μg L^{-1} As(III) were obtained at a sample throughput of 32 h^{-1}. The precision for 14 replicate measurements of 1 μg L^{-1} As(III) was 1.3% (RSD). The recoveries from natural water samples varied from 96.7 to 105% for 2 μg L^{-1} of As(III) spike and from 97.1 to 107% for 2 μg L^{-1} of As(V) spike. The analytical results obtained by the present method for total arsenic in the certified reference materials, SLRS-4 (river water) and NASS-5 (seawater), agreed well with the certified values. The developed method was also successfully applied to the speciation of inorganic arsenic in local natural water samples.

The arsenic biogeochemical cycle occurs mostly in the aquatic environment.[1] Arsenic contamination in natural water has been related to different diseases in some countries.[2,3] The toxicity of arsenic depends strongly on its chemical forms. Inorganic compounds of arsenic are far more toxic than their organic counterparts.[4] The more important forms in natural waters are mainly the inorganic arsenic species, As(III) and As(V), due to the release of arsenic compounds from minerals. The ratio between these forms depends on the pH values and the local redox conditions. To understand the effect of arsenic on the aquatic environment and differences in toxicity of arsenic compounds, it is increasingly important to monitor the concentrations of arsenic species in different aquatic systems.

For the determination of arsenic species in natural water samples, where the concentrations of arsenic are usually below 1 μg L^{-1}, highly sensitive and selective techniques are required owing to the low concentrations of the species. When the analytical techniques employed do not offer sufficient detection capability for such determination, preconcentration and separation steps are always necessary. The preconcentration can be divided into two types by and large: off-line and on-line procedures. The off-line procedures, such as ion exchange, precipitation, coprecipitation, and liquid−liquid extraction, are often time-consuming and labor-intensive, require large sample volumes, and suffer great risks of contamination and analyte loss. With on-line preconcentration using flow injection (FI) techniques, the drawbacks of batchwise operation can be overcome to a great extent. To date, various batch-type procedures have been adapted to FI on-line preconcentration for atomic spectrometry.[5] However, the majority of on-line preconcentration systems are based on sorption principles. These systems include those using microcolumns packed with chelating ion-exchange resins, reversed phase silica gel sorbents with octadecyl functional groups (C_{18}), and those using open tube knotted reactors (KRs) made from poly(tetrafluoroethylene) (PTFE) tubing.[6] The microcolumn systems have been limited by not being able to use higher sample loading rates for achieving better enrichment factors owing to high hydrodynamic impedance in the microcolumns.[7] FI on-line preconcentration and separation

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based on sorption of organometallic complexes in a KR have been successfully coupled with atomic (mass) spectrometry for trace element analysis.[8] The KR produces significantly lower back pressures than packed columns at similar flow rates, so that higher sample loading rates are readily applicable. Also, the KR is easily laboratory-constructed with no need for packing materials and offers almost unlimited lifetime as well as stability in sorption properties, allowing the analysis to be conducted at low cost.[7−9]

In recent years, the development of simple hydride generation atomic fluorescence spectrometers (HG-AFS) has provided a suitable tool for As determination.[10] Gómez-Ariza et al.[11] compared ICPMS and AFS for arsenic speciation analysis in environmental samples and demonstrated that AFS and ICPMS gave similar performance results (detection limits, reproducibility, repeatability, and sensitivity). However, AFS also presents the benefits of much lower running costs, shorter warm times prior to analysis, and easy handling in comparison with ICPMS.[11]

Coupling of FI on-line KR sorption preconcentration and separation to HG-AFS is expected to be an attractive technique for routine (speciation) analysis of hydride-forming elements owing to the low cost, easy operation, high selectivity, and sensitivity. However, to our knowledge, no such work, to date, has been reported.

In the current work, an attempt was made to couple an FI on-line KR sorption preconcentration and separation system with HG-AFS for determination of As(III) and As(V) in natural water samples. Selective determination of As(III) in the presence of As(V) was achieved by selective formation of the As(III)−pyrrolidinedithiocarbamate (PDC) complex, its simultaneous adsorption onto the inner walls of the KR made from PTFE tubing, elution with diluted hydrochloric acid, arsine generation with KBH_4, and detection by AFS. Total inorganic arsenic was determined after prereduction of As(V) to As(III) by 1% m/v L-cysteine. The concentration of As(V) was calculated by the difference of the total inorganic arsenic and As(III). Potential factors that affect adsorption, rinsing, elution, and hydride generation were investigated in detail.

EXPERIMENTAL SECTION

Apparatus. A model XGY-1011A nondispersive atomic fluorescence spectrometer (Institute of Geophysical and Geochemical Exploration (IGGE), Langfang, China) was employed throughout. A high-intensity arsenic hollow cathode lamp (Ningqiang Light Sources Co. Ltd., Hengshui, China) was used as the radiation source. A quartz tube (7-mm i.d. × 14-cm length) was used as the atomizer, into which the volatile species and the hydrogen evolved from the reactor were swept by an argon flow. The gas mixture is self-ignited at the outlet of the furnace, and a hydrogen−argon-air entrained flame is maintained without the addition of any auxiliary hydrogen. A W-type gas−liquid separator (GLS) (Part No. B0193722, Perkin-Elmer) was used. The argon flow was controlled by a rotameter. The operating parameters of the AFS instrument are given in Table 1.

Table 1. AFS Parameters

parameter	setting
arsenic hollow cathode lamp	
primary current	60 mA
boost current	50 mA
quartz furnace temp	200 °C
quartz furnace height	6 mm
negative high voltage of photomultiplier	260 V

A model FIA-3100 flow injection analyzer (Vital Instrumental Co. Ltd., Beijing, China) was used throughout this work. The FIA-3100 consists of two peristaltic pumps and a standard rotary injection valve (8-channel 16-port multifunctional injector). The KRs were laboratory-made from PTFE tubing of 0.5-mm i.d. by tying interlaced knots.[7,12] Tygon peristaltic pump tubings were employed to propel the sample and reagent. PTFE tubing with 0.5 mm i.d.was used for all connections. These connections were kept as short as possible to minimize the dead volumes.

Reagents. All the reagents were at least of analytical grade. Doubly deionized water (DDW) was used throughout. Arsenite stock solutions of 100 mg L^{-1} were bought from Yingtianyi Standards Co., Beijing, China. The arsenate stock solution of 1000 mg L^{-1} was prepared by dissolving $Na_2HAsO_4{\cdot}7H_2O$ (Beijing Chemicals Co. Ltd., Beijing, China) in DDW directly. Working standards solutions were prepared daily by stepwise diluting the stock solutions. A 2.0% m/v KBH_4 solution was prepared by dissolving KBH_4 (IGGE) in 0.2% m/v KOH solution (IGGE). The chelating agent solution was prepared by dissolving ammonium pyrrolidinedithiocarbamate (APDC) (Sigma-Aldrich) in DDW. A 1% m/v L-cysteine solution was prepared as the prereductant by dissolving L-cysteine (Sigma-Aldrich) in DDW.

Sample Pretreatment. Five lake and two river water samples were collected. Immediately after sampling, the samples were filtered and stored at 4 °C in low-density polyethylene (LDPE) bottles. Suitable amounts of 1 mol L^{-1} HCl were added to adjust the acidity of samples. Prereduction of As(V) to As(III) for the determination of total arsenic was carried out with 1% m/v L-cysteine. These samples were analyzed immediately after acidity adjustment so that the disturbance of the original distribution of As(III)/As(V) was kept as minimum as possible.

The following certified reference materials (CRMs) (NRCC, Ottawa, Canada) were used to check the accuracy of the developed method: SLRS-4 (river water); NASS-5 (open ocean seawater). These water samples were analyzed directly because their acidity was in the optimal range of sample acidity for determination of As(III).

Procedures. The FI manifold for the present on-line KR sorption preconcentration coupled with HG-AFS is illustrated in Figure 1. Details of FIA-3100 program and sequence of operation are described as follows. In the prefill stage, pump 2 was activated and the injector valve was in the injection position, so that the tubing was filled with sample and APDC solution. This prefill stage was used only when a new sample was introduced but omitted for replicate preconcentration. In step 1, pump 2 was active while the injector valve turned to the fill position. In this step, the As(III)−PDC complex was formed on-line and sorbed onto the

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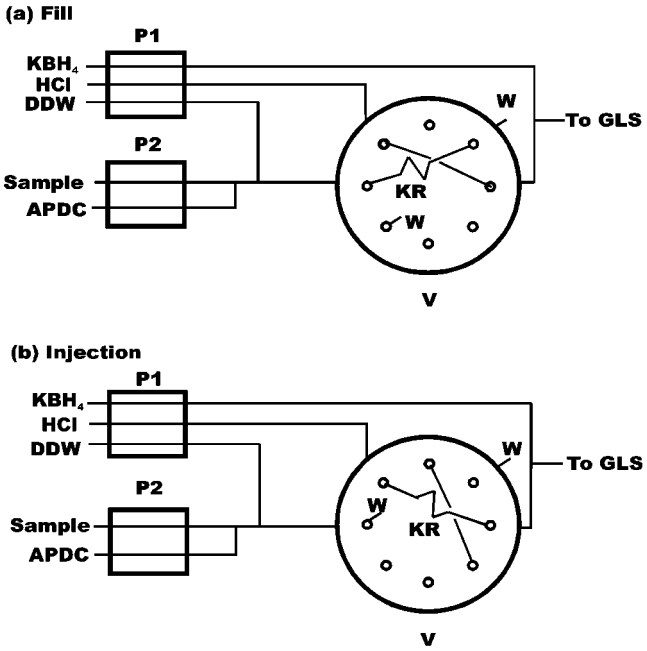

Figure 1. FI manifold and operational sequence for the on-line KR sorption preconcentration coupled with HG-AFS: P1, P2, peristaltic pump; W, waste; KR, knotted reactor (150-cm long × 0.5-mm i.d. PTEF tubing); V, injector valve (the inner is the rotor); GLS, gas−liquid separator; injector valve position (a) fill, (b) injection.

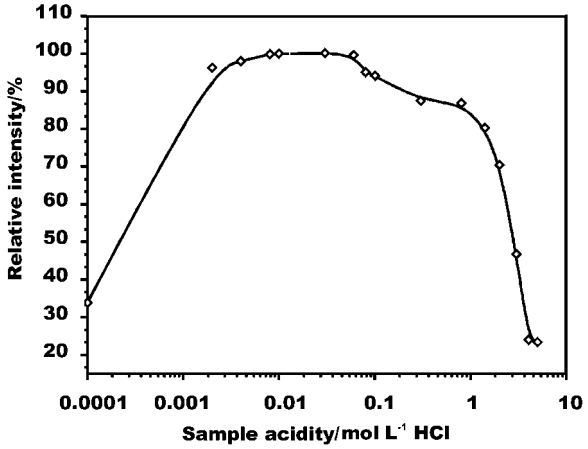

Figure 2. Influence of sample acidity on the preconcentration of 1 μg L^{-1} As(III) with a preconcentration time of 60 s. All other conditions are as in Figure 1 and Table 1.

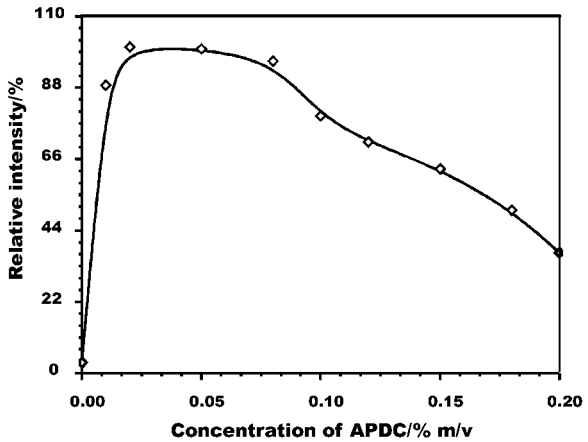

Figure 3. Effect of APDC concentration on the preconcentration of 1 μg L^{-1} As(III) with a preconcentration time of 60 s. All other conditions are as in Figure 1 and Table 1.

inner walls of the KR, the effluent from the KR flowing to waste. In step 2, the valve was still in the fill position, but pump 2 was stopped and pump 1 was actuated, so that DDW was sucked through the KR to remove the residual matrix. In step 3, the valve was turned to the injection position while pump 1 was still running so that 1 mol L^{-1} HCl solution was pumped to elute the sorbed analyte and also to provide the required acidic medium for subsequent hydride generation. Meanwhile, a flow of KBH_4 solution was introduced to merge with the HCl solution containing the eluted analyte just before entering GLS to generate gaseous arsine and hydrogen. The generated gaseous mixture was transported to the atomizer with an argon flow at 320 mL min^{-1}. At same time, the atomic fluorescence signal was detected by AFS. The total time required for a single determination lasted 110 s.

RESULTS AND DISCUSSION

Sample Acidity. The effect of sample acidity on the preconcentration of As(III) was tested at a fixed APDC concentration of 0.05% m/v. As shown in Figure 2, the optimum sample acidity ranged from 0.001 to 0.1 mol L^{-1} HCl. The lower sensitivity at lower acidity (<0.001 mol L^{-1} HCl) probably resulted from unfavorable complex formation between As(III) and APDC. Higher acidity (>0.1 mol L^{-1} HCl) may be not favorable for adsorption of the complex on the inner walls of the KR, leading to decrease in sensitivity. For further experiments, an acidity of 0.05 mol L^{-1} HCl was used. Because As(V) could not be complexed with APDC, and no As(V) could be preconcentrated, it is possible to selectively preconcentrate As(III) in the presence of As(V).

APDC Concentration. The influence of APDC concentration on the preconcentration of As(III) was tested when sample and reagent flow rates were kept at 6.0 and 3.0 mL min^{-1}, respectively. The result is shown in Figure 3. In the absence of APDC, no As(III) was sorbed on the walls of the KR. The signal intensity significantly increased with increase in concentration of APDC up to 0.02% m/v. These results indicate that it was the As(III)−PDC rather than As(III) that was sorbed on the inner walls of the KR. However, the absorbance decreased slightly in the range of 0.02−0.08% m/v APDC and reduced remarkably with further increase in APDC concentration, probably due to the competition of excessive complexing reagent for the active sites on the KR walls.[9] Accordingly, an APDC concentration of 0.05% m/v was employed for further work.

Sample Flow Rate and Preconcentration Time. The influence of sample flow rate on the preconcentration of As(III) was evaluated at a fixed APDC flow rate of 3.0 mL min^{-1}. It was found that the signal intensity increased linearly up to a sample flow rate of 8.0 mL min^{-1}. Studies on the effect of sample preconcentration time shows that analyte signal intensity increased almost linearly up to 150 s. This trait gives the feasibility of determination of the samples with different analyte concentration levels. Short preconcentration time could be used for higher analyte concentrations, whereas long preconcentration time would be beneficial to the analysis of the samples with lower analyte concentrations. In this work, a preconcentration time of 60 s and a sample flow rate of 6 mL min^{-1} were used.

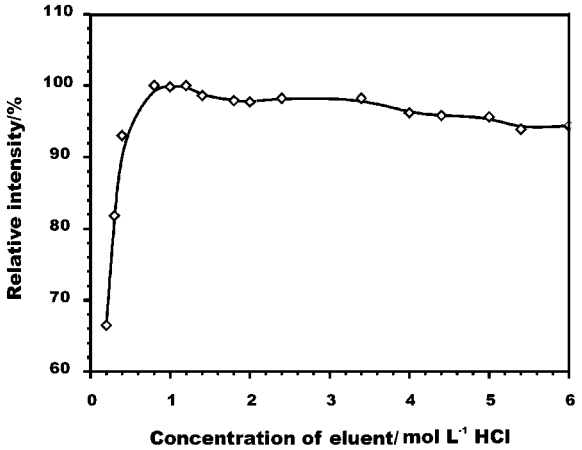

Figure 4. Effect of HCl concentration in eluent on the preconcentration of 1 μg L^{-1} As(III) with a preconcentration time of 60 s. All other conditions are as in Figure 1 and Table 1.

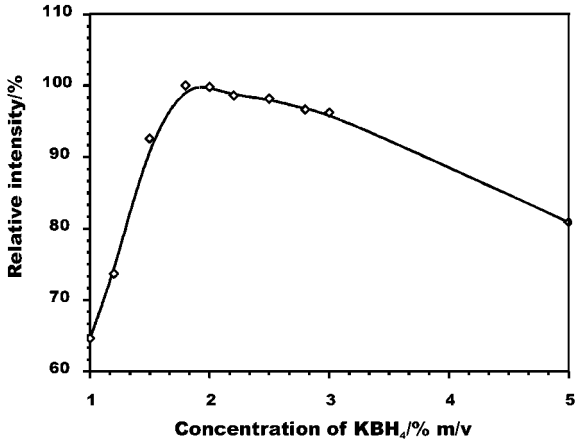

Figure 5. Influence of KBH_4 concentration on the preconcentration of 1 μg L^{-1} As(III) with a preconcentration time of 60 s. All other conditions are as in Figure 1 and Table 1.

KR Rinsing. After sample loading into the KR, the residual solution in the KR may contain dissolved salts, which could interfere the determination of As(III). Thus, a rinsing step was used to remove the residual matrix in the KR before elution. In this work, air, DDW, and diluted HCl solution were tested as the rinsing solution. Air was found not able to remove the residual matrix completely. When diluted HCl solution was used as the rinsing solution, the signal decreased with increase in the concentration of HCl. However, DDW was found to remove the residual matrix effectively while not stripping off the sorbed analyte. Accordingly, DDW was chosen as the rinsing solution.

Choice of Eluent. Choice of a proper eluent is one of the keys to the successful coupling of FI on-line KR sorption preconcentration to HG-AFS. In addition to the sufficiently strong elution capability required, the eluent employed in FI on-line KR sorption preconcentration and separation for HG-AFS should facilitate the ensuing hydride generation reaction. For these reasons, diluted HCl solution was employed as the eluent. The effect of HCl concentration was investigated to determine the optimal concentration for efficient elution and hydride generation. As shown in Figure 4, the signal intensity increased as the concentration of HCl increased up to 0.8 mol L^{-1} and then leveled off until 2.6 mol L^{-1} HCl. Higher concentrations of HCl would cause serious effervescence, and splashing of solution droplets on the GLS walls due to the fast reaction, leading to a decrease in the signal and poor precision. Therefore, a solution of 1 mol L^{-1} HCl was employed throughout this work.

KBH_4 Concentration. The effect of KBH_4 concentration on the signal intensity is shown in Figure 5. The optimal concentration of KBH_4 ranged from 1.8 to 2.0% m/v. Higher concentrations of KBH_4 (>2.5% m/v) would also cause serious effervescence and splashing of solution droplets on the GLS walls due to the fast reaction. As a result, water vapor and/or mist of reagents might condense on the transfer line and consequently trap the volatile arsine, resulting in a significant decrease of the signal intensity. Low KBH_4 concentrations (<1.8% m/v) probably gave incomplete reduction of the analyte and/or provided insufficient hydrogen to maintain the argon−hydrogen flame, leading to low signal intensity. So, a KBH_4 concentration of 2.0% m/v was selected for the determination of arsenic.

Table 2. Effect of Interfering Ions on the Determination of 1 μg L^{-1}As(III)

interfering ion	concn/μg L^{-1}	recovery (%)
Cu(II)	10	96.4
	100	76.8
Fe(III)	100	99.1
	1000	36.5
Ni(II)	100	95.2
	1000	49.2
Hg(II)	100	94.1
	1000	45.5
Se(IV)	100	101
	1000	97.3

Table 3. Characteristic Performance Data for the FI On-Line Preconcentration for HG-AFS Determination of As(III)

calibration range/μg L^{-1}	0.1−10
sampling freq/h^{-1}	32
enhancement factor	11
sample consumption/mL	6
reagent consumption/mL	
0.05% m/v APDC	3
2.0% m/v KBH_4	4
1 mol L^{-1} HCl	4
precision (RSD, $n = 14$)/%	1.3 (1 μg L^{-1})
detection limit ($3s$)/μg L^{-1}	0.023
calibration function (I, signal intensity; C, μg L^{-1}; 14 standards)	$I = 123.84C - 1.65$
corr coeff	0.9995

KR Tubing Length. The influence of KR tubing length on the preconcentration of As(III) was investigated at a sample flow rate of 6.0 mL min^{-1} and APDC flow rate of 3.0 mL min^{-1}. It was observed that the signal intensity increasd with increase in KR tubing length from 50 to 150 cm and then decreased from 150 to 250 cm. This result could be explained in terms of adsorption efficiency and dispersion.[9] Below a KR tubing length of 150 cm, low adsorption efficiency might dominate the signal intensity, while, over the length of 200 cm, high analyte dispersion probably controlled the signal. So, a KR of length of 150 cm was selected for the further work.

Interferences. Interfering ions may compete with the analyte for complexing agent and/or subsequently for reductant. In this

Table 4. Analytical Results (Means ± *s*, *n* = 5) of As(III), As(V), and Total Inorganic Arsenic in Water Samples

sample	tot. As certified/μg L^{-1}	detnd/μg L^{-1}		calcd/μg L^{-1}	recovery of As(III)[a]/%	recovery of As(V)[b]/%
		As(III)	tot. As	As(V)		
SLRS-4 (river water)	0.68 ± 0.06	nd[c]	0.67 ± 0.01	0.67 ± 0.01		
NASS-4 (seawater)	1.27 ± 0.12	nd	1.24 ± 0.02	1.24 ± 0.02		
tap water		nd	nd	nd	98.0	97.8
lake water 1		0.72 ± 0.01	2.20 ± 0.02	1.48 ± 0.08	105	97.1
lake water 2		0.28 ± 0.09	1.85 ± 0.06	1.57 ± 0.09	96.7	100
lake water 3		0.34 ± 0.03	1.02 ± 0.09	0.68 ± 0.09	97.4	98.5
lake water 4		0.48 ± 0.02	1.28 ± 0.02	0.80 ± 0.02	101	106
lake water 5		0.68 ± 0.05	1.11 ± 0.07	0.43 ± 0.07	99.6	107
river water 1		0.18 ± 0.06	2.08 ± 0.11	1.90 ± 0.11	99.2	100
river water 2		0.50 ± 0.09	1.31 ± 0.06	0.81 ± 0.09	99.6	106

[a] Recovery for spiking with 2 μg L^{-1} As(III). [b] Recovery for spiking with 2 μg L^{-1} As(V). [c] nd, not detectable.

work, three typical transition metal ions, Cu(II), Fe(III), and Ni(II), and two vapor-forming elements, Hg(II) and Se(IV), were chosen to study the interferences. The results are shown in Table 2. The effect is expressed as the recovery in the presence of interfering ions relative to the interference-free response. Differences less than 5% are probably insignificant, but those greater than 10% are considered to be a result of interference effects. As can seen from Table 2, a 10-fold excess of Cu(II), 100-fold of Fe(III), Ni(II), and Hg(II), and 1000-fold of Se(IV) had no significant influence on the signal intensity of As(III), whereas a 100-fold excess of Cu(II) and 1000-fold of Fe(III), Ni(II), and Hg(II) interfered, to a certain extent, with determination of arsenic.

Analytical Preformance of the FI On-Line Preconcentration and Separation System for HG-AFS. The analytical characteristic data of FI on-line KR sorption preconcentration and separation system coupled with HG-AFS are summarized in Table 3. The detection limit (3*s*) and the sampling frequency were determined to be 0.023 μg L^{-1} and 32 h^{-1}, respectively. The precision for 14 replicate determinations at the 1 μg L^{-1} level was 1.3% (RSD). The recoveries were tested for spike of 2 μg L^{-1} As(III) and As(V) in the studied samples and ranged from 96.7 to 105% for As(III) and from 97.1 to 107% for As(V), respectively, indicating no interference encountered from these sample matrices.

The analytical characteristic data of the present FI KR sorption preconcentration HG-AFS (detection limits, reproducibility, repeatability, and sensitivity) are comparable to those obtained by a previous FI KR sorption preconcentration ICPMS system for determination of As(III) and As(V).[9] The obvious advantages of the present system are its simplicity and low instrument and running costs.

Compared with conventional HPLC with which high dilution factors are always associated, a distinct advantage of the present separation by sorption on the KR is that the analyte species is at the same time preconcentrated. Because the FI preconcentration and separation were completed in fractions of a second, shifts of the As(III)/As(V) equilibrium after one part had been removed were almost impossible within the time frame of the experiment. However, with the coupling of HPLC to atomic spectrometry the detection of organic As species is additionally possible.

The accuracy of the present method was further demonstrated by determining the total arsenic in two water CRMs, SLRS-4 (river water) and NASS-5 (seawater). The results are shown in Table 4. The total arsenic concentrations determined by the present method using a simple aqueous standard calibration technique were in good agreement with the certified values. No As(III) was detected in the CRMs probably because the material had been stabilized with nitric acid for a long time; thus, As(III) had been oxidized to As(V).

The developed method was also applied to determination of As(III), total inorganic arsenic, and As(V) in seven local natural water samples including tap water, river water, and lake water samples to evaluate the arsenic pollution. The analytical results obtained by the present method using a simple aqueous standard calibration technique are given in Table 4. As can be seen, there was no arsenic detected in the tap water. However, the concentrations of As(III) and total inorganic As in the lake and river water samples ranged from 0.18 to 0.72 and 1.02 to 2.20 μg L^{-1}, respectively. The concentration ratios of As(V) to As(III) in these lake and river water samples varied from 0.63 to 10.6, indicating different redox conditions existing in these aquatic environments.

CONCLUSIONS

The results of this work demonstrated the feasibility of the developed FI on-line KR sorption preconcentraion and separation coupled with HG-AFS for determination of ultratrace As(III), As(V), and total inorganic arsenic in water samples. To our knowledge, this is the first work dealing with the on-line coupling of FI KR sorption preconcentration and separation technique to HG-AFS for trace element analysis. The low cost, easy operation, and high sensitivity of the present system make it very attractive for routine speciation of important species of hydride-forming elements in natural waters.

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