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Low temperature hydrogen plasma assisted chemical vapor generation for Atomic Fluorescence Spectrometry

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ABSTRACT

Chemical vapor generation techniques have long been considered as important ways of sample introduction for analytical atomic spectrometry. In this paper, a low temperature plasma assisted chemical vapor generation method which avoids the massive use of consumptive chemical agents was proposed by using atmospheric pressure dielectric barrier discharge. The plasma was generated by hydrogen doped argon gas flow through a quartz tube, serving as a dielectric barrier, which had a copper wire inner electrode and a copper foil outer electrode. An alternative high voltage was applied to electrodes to ignite and sustain the plasma. Sample solutions were converted to aerosol by a nebulizer and then mixed with the plasma to generate hydrides. To confirm the utility of this method, four hydride forming elements, As, Te, Sb and Se, were determined by coupling the low temperature plasma assisted chemical vapor generation system with an atomic fluorescence spectrometer. Responses of As, Te, Sb and Se were linear in the range of 0.5–20 μg mL^{-1}. The RSDs of As, Te, Sb and Se in the present method were less than 4.1% and the absolute detection limits for As, Te, Sb and Se were 0.6 ng, 1.0 ng, 1.4 ng and 1.2 ng, respectively. Furthermore, four arsenic species were determined after HPLC separation. The method is green and simple compared with hydride generation with tetrahydroborate and the most attractive characteristic is micro-sampling. In principle, the method offers potential advantages of miniaturization, less consumption and ease of automation.

1. Introduction

Chemical vapor generation coupling to atomic spectrometry has been widely applied to determine trace elements in environmental and biological samples [1,2]. This technique has gained popularity owing to its superiority in terms of sensitivity, selectivity and linear range for the determination of hydride-forming elements, such as As, Te, Sb and Se from trace to ultra-trace levels [3]. Hydride generation by sodium tetrahydroborate ($NaBH_4$) has been applied successfully in separating analytes from the matrix to reduce interference and in pre-concentrating the analytes [4,5]. However, tetrahydroborate is so unstable and difficult to store that it should be used immediately after preparation [5]. In addition, those chemical agents are quite consumptive during the analysis; therefore the method of hydride generation by tetrahydroborate does not conform to the principle of energy conservation and emission reduction.

To avoid shortcomings of the conventional hydride generation method, various reducing agents and sources of nascent hydrogen have been suggested in order to convert the elements into hydrides, although the mechanism for the formation of hydrides is still a matter of debate [6–9]. Sturgeon's group has reported an approach of vapor generation; in the presence of low molecular weight organic acid solutions, inorganic Se(IV) is converted by UV light to volatile Se species, which are rapidly transported to a heated quartz tube atomizer for detection by atomic absorption spectrometry [10]. Wang et al. have explored a method for online pre-reduction of Se(VI) with a newly designed UV/TiO_2 photocatalysis reduction device [11], which could be applied to selenium speciation by coupling HPLC to the atomic fluorescence spectrometer [12]. Zhang et al. have developed a method of electrochemical HG system which consists of a graphite tube cathode and a platinum anode for analyses of As, Sb [13] and Hg [14]. Techniques mentioned above have partly overcome the disadvantages of conventional hydride generation, but inorganic or organic reagents are still required, which possibly pollute the environment.

These problems can be solved by generating nascent hydrogen in hydrogen plasma by dielectric barrier discharge (DBD) without chemical agent, provided that the production of nascent hydrogen in DBD plasma has been previously demonstrated [15]. As a mature means to create low temperature plasma (LTP), DBD which has been successfully used in previous studies of atomic spectrometry, offers several advantages like forming at atmospheric pressure, small size and low power consumption [16,17]. A DBD source with argon or helium plasma has been reported as an atomization source, or to

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provide energy for atomic emission processes. For instance, a wall-stabilized argon plasma for the determination of some volatile hydride-forming elements has been reported [18]. Gras et al. and Hou et al. have explored the DBD as a gas chromatographic (GC) detector [19,20]. A DBD atomizer for AAS or AFS has been proposed by our group for the determination of hydride-forming elements [21,22,24,26,27] and further extended to serve as the light source of atomic emission spectrometry by Wang's group [23,25]. Also the combination of LTP with ICP-MS proved that LTP probe is easily coupled to various elemental analysis tools for thin layer or direct solid sample analysis in microarea [28]. Recently, Zhu et al. have reported a plasma-chemical vapor generation of cadmium [29]. However, analyses of common hydride generated elements, such as As, Te, Sb and Se, have not been successful. Since a large number of active hydrogen species, such as H, H^+, H_2^+, and H_3^+ exist in H_2 plasma [30], and these species possess much higher reduction abilities than molecular H_2, we proposed to use a hydrogen plasma to replace the hydrides generation system with $NaBH_4$ for the elemental analysis.

In the present work, we used hydrogen plasma to generate chemical vapors without the use of $NaBH_4$, organic acid and other chemicals such as TiO_2. Four elements have been examined including As, Te, Sb and Se by coupling the low temperature plasma assisted hydrides generation method with the atomic fluorescence spectrometer. The proposed method was validated by analyses of certified reference materials (kelp, laver and underwater sediments). Also, a mixture of four arsenic species has been analyzed after HPLC separation. The results met with our expectation that the chemical vapor generation can be generated in a DBD-hydrogen plasma.

2. Experimental section

2.1. Reagents

All chemicals were at least of analytical grade and all solutions were prepared using ultra-pure water with a resistivity of 18.2 MΩ cm, obtained from a water purification system (Thermo Fisher, USA). The stock standard solutions of Se, Te, As and Sb (1000 mg L^{-1}) were supplied by National Analysis Center for Iron and Steel (Beijing, China). Working standard solutions were prepared daily by stepwise dilution of stock solutions with 5.0% (v/v) hydrochloric acid. Argon (99.999%) and hydrogen (99.999%) from Huayuan Co. (Beijing, China) were used and the flow rates were controlled and measured by two mass and volumetric flow controllers (Cole-Parmer Co. Ltd., USA).

For conventional HG-AFS, 2.0% (m/v) $NaBH_4$ solution was prepared by dissolving the appropriate amount of $NaBH_4$ into 0.5% (m/v) NaOH. For arsenic speciations, arsenite (As(III)), arsenate (As(V)), monomethylarsonic acid (MMA) and dimethylarsinic acid (DMA) standard solutions were provided by National Institute of Metrology (NIM). The HPLC eluent was prepared with $(NH_4)_2HPO_4$ (Merck), and the pH was adjusted to 6.0 by addition of an aqueous NH_3 solution (Merck).

2.2. Instrumentation

A commercial AFS was employed in the present study (AFS 9800, Hai Guang Co., China). High-intensity hollow cathode lamps (General Research Institute for Non-ferrous Metals, Beijing, China) were used as light sources. The optimized experimental conditions of AFS used for this study are summarized in Table 1. High voltages from a DBD pulse generator (Coronalab Co. Ltd., Nanjing, China) were applied to the central copper wire and the outer electrode. The HPLC system consisted of a six-port valve with a 100 μL injection loop (Rheodyne), a Hamilton PRP X-100 anion-exchange column (250 mm × 4.1 mm i.d., 10 μm) and a guard column packed with the same material. The outlet of the column was connected to on-line continuous DBD-hydrogen plasma – Atomic Fluorescence Spectrometry system.

Table 1
The operation conditions of AFS.

Parameters	As	Te	Sb	Se
Lamp primary current (mA)	60	60	70	80
Lamp boost current (mA)	30	30	35	40
Negative high voltage of PMT (V)	300	300	290	300
Shield gas flow rate (mL min^{-1})	900	900	900	900
Atomizer observation height (mm)	8	8	8	8

2.3. Samples

To validate the accuracy of the developed method, the certified reference materials, underwater sediments (GBW07311), kelp (GBW08517) and laver (GSB-14) were obtained from the National Research Center for Certified Reference Material of China. Samples were decomposed by a microwave-assisted digestion system. Briefly, 0.5 g of the samples was added to Teflon vessels containing 6.0 mL HNO_3 and 2.0 mL HCl for GBW07311, 4.0 mL HNO_3 and 2.0 mL H_2O_2 for GBW08517 and GSB-14, respectively. Teflon vessels were closed tightly and put into the microwave digestion system (MARS5, CEM, USA) for digestion. The heating program was 120 °C for 5 min, 140 °C for 5 min, 160 °C for 15 min and 185 °C for 5 min. After cooled to ambient temperature, digests were quantitatively transferred into a Teflon volumetric flask. The residues were flushed with ultra-pure water into the volumetric flask and the solutions were adjusted to the finally contained 5% (v/v) HCl.

For arsenic speciation analysis, 0.2 g of the pulverized sample was added into the extraction cell at room temperature and 10.3 MPa pressure with 50% (v/v) methanol solution. Then it was extracted for 5 min and purged by nitrogen for 60 s. This procedure was repeated three times; the combined extracts were extracted with rapid swirling flow concentrator and concentrated at 65 °C. After the concentrate was diluted with ultrapure water and fixed weighed to 25.0 g, the sample solution was obtained.

2.4. Safety consideration

In case of the high voltage shock, contacting the electrode should be avoided during generating the plasma. Direct glaring at the plasma should also be avoided to prevent harm from ultraviolet radiation.

3. Results and discussion

3.1. Design of the LTP assisted chemical vapor generation system

The schematic diagram and pictures of experimental setup used in the present study are shown in Fig. 1. The copper wire and copper foil served as electrodes, while the quartz served as the dielectric barrier. The copper wire was sealed inside a quartz tube to avoid the contamination of samples and was placed at the center of a quartz cell. When a high voltage was applied to electrodes, the plasma was ignited and sustained. The plasma could easily be generated in Ar or a mixture of Ar with He, N_2 or H_2 at atmospheric pressure.

The experiments were carried out by coupling DBD-hydrogen plasma with AFS for the determination of As, Sb, Te, and Se samples at the concentration of 10 μg mL^{-1}. The standard solutions were introduced into the system by a syringe at a flow rate of 10 μL min^{-1}, respectively. After nebulization with argon gas,

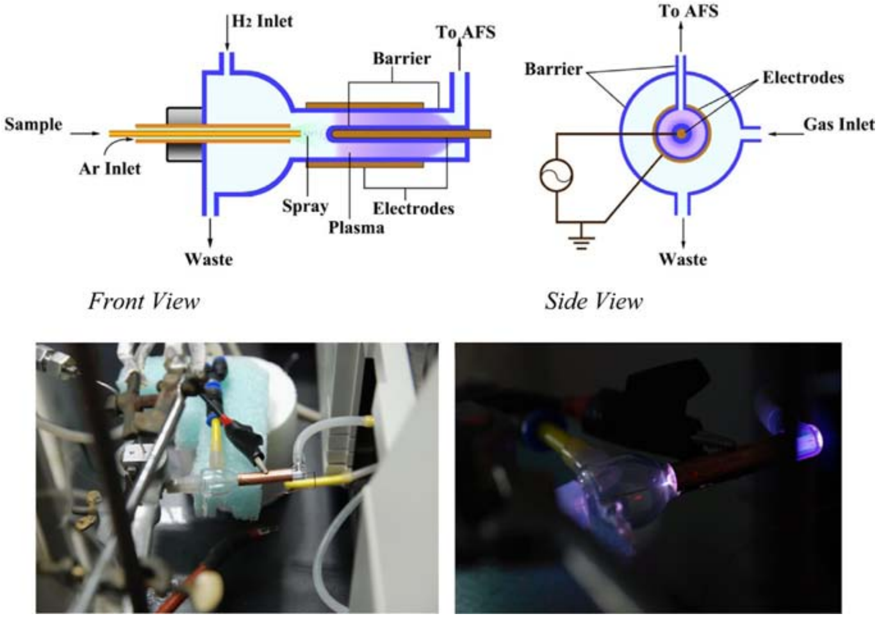

Fig. 1. The schematic diagram of the LTP hydride generator (upper) and pictures of experimental setup.

aerosols carried by Ar gas were mixed with H_2 and passed through the plasma to generate chemical vapors. The formed chemical vapors were transported to the atomizer for AFS detection.

3.2. *The effect of H_2 in the system*

To explore the role of H_2 in the designed system, we had conducted some experiments. There were only negligible fluorescence signals for the four samples when we used N_2 or He as carrier gas without H_2, as shown in Fig. 2a. However, when we introduced H_2 into the plasma, the fluorescent signals increased significantly (Fig. 2a). Obviously, H_2 contributed greatly to the fluorescent signals.

We were confused that how hydrogen influenced the fluorescent signals. There were two parts in the system, where H_2 might have reacted with the sample aerosols. One was the plasma; another was the quartz furnace atomizer of AFS. To find which one was the real reaction place, we tried to change the position of H_2 introduction to the position between the plasma and the atomizer (the position down to the plasma). We found that there were only weak signals for the four elements as shown in Fig. 2b. When we introduced H_2 to the original position and to the position down to the plasma, the signals increased significantly. On comparing Fig. 2a to b, we can conclude that reactions of H_2 and aerosols take place in the plasma. We further compared the signal intensities with the on and off status of plasma. As shown in Fig. 2c, enhanced fluorescent signals occurred when the plasma was turned on while only background signals occurred when the plasma was turned off.

Therefore, we can confirm that the enhancement of fluorescent intensities is caused by the introduction of hydrogen to the plasma and the hydrogen reacts with sample aerosols in the plasma. For the mechanism of the reaction process, we can make an assumption. When hydrogen is introduced to Ar plasma, H_2 is excited by Ar plasma to generate active hydrogen species, such as H, H^+, H_2^+, and H_3^+ further to form the hydrogen plasma. Then the sample aerosols generate the chemical vapors (hydrides) by the reduction abilities of those active hydrogen species.

3.3. *Optimizing the hydrogen plasma assisted chemical vapors generation system*

Operation parameters of the chemical vapors generation system were optimized with respect to hydrogen gas flow rate, argon gas flow rate and DBD discharge power. Various experimental parameters were optimized individually while others were kept at fixed values.

The hydrogen gas flow rate is a major factor that has a crucial effect on the performance of the designed system. Influences of H_2 gas flow rate on the fluorescence signals of As, Te, Se and Sb were studied over the range of 50 to 250 mL min^{-1}. As shown in Fig. 3a, the fluorescence signals increased with increasing H_2 flow rate and reached a maximum at 150 mL min^{-1}. The flow rates above this value caused the fluorescence signal to decrease.

Argon gas plays a dual role in the system. First, after the sample solutions are nebulized with Ar, the argon gas carries the aerosols into the quartz cell. Second it helps the formation of hydrogen plasma. Therefore the fluorescence signal is affected by changes in transport rate. The flow rate of argon gas was examined to be in the range of 200–600 mL min^{-1} (Fig. 3b). The maximum fluorescence signal was achieved at Ar flow rate of 400 mL min^{-1}, which was used for subsequent studies.

The plasma is promptly generated as the discharge power is loaded on the electrodes. Effects of discharge power on the signal intensities were investigated. Maximum fluorescence signals were observed at 16 W when the power changed from 7.8 W to 18.2 W, as shown in Fig. 3c. Signals decreased dramatically when the power was lower than 14 W, while the discharge transformed to arc discharge, which caused the plasma to be unstable, when the power was higher than 16 W.

3.4. *Analytical performance*

The standard solutions of As, Te, Sb with concentrations ranging from 0.5 to 20 μg mL^{-1} respectively and the standard solutions of Se with concentrations ranging from 0.5 to 50 μg mL^{-1} were injected into the DBD-hydrogen plasma and detected by AFS. Fig. 4 shows typical fluorescence signals at different concentrations. Using the peak height as a quantitative parameter, the calibration curve of

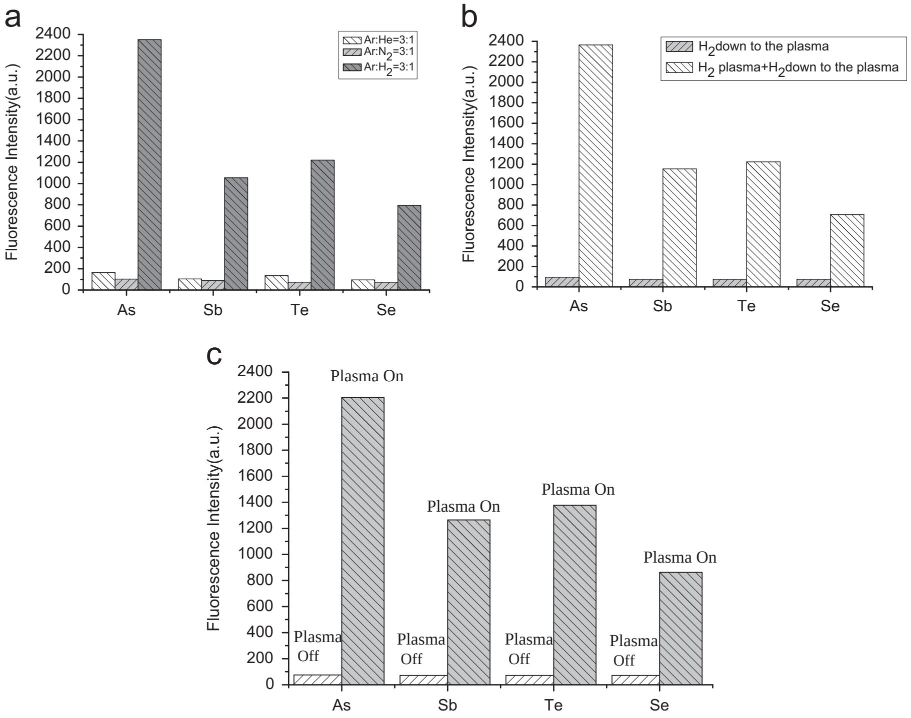

Fig. 2. Fluorescence signals of As, Sb, Te and Se (10 μg mL^{-1}) recorded under different conditions: (a) using He, N_2 or H_2; (b) the position of H_2 down to the plasma; (c) plasma was set on and off when the sample injected through the experiment.

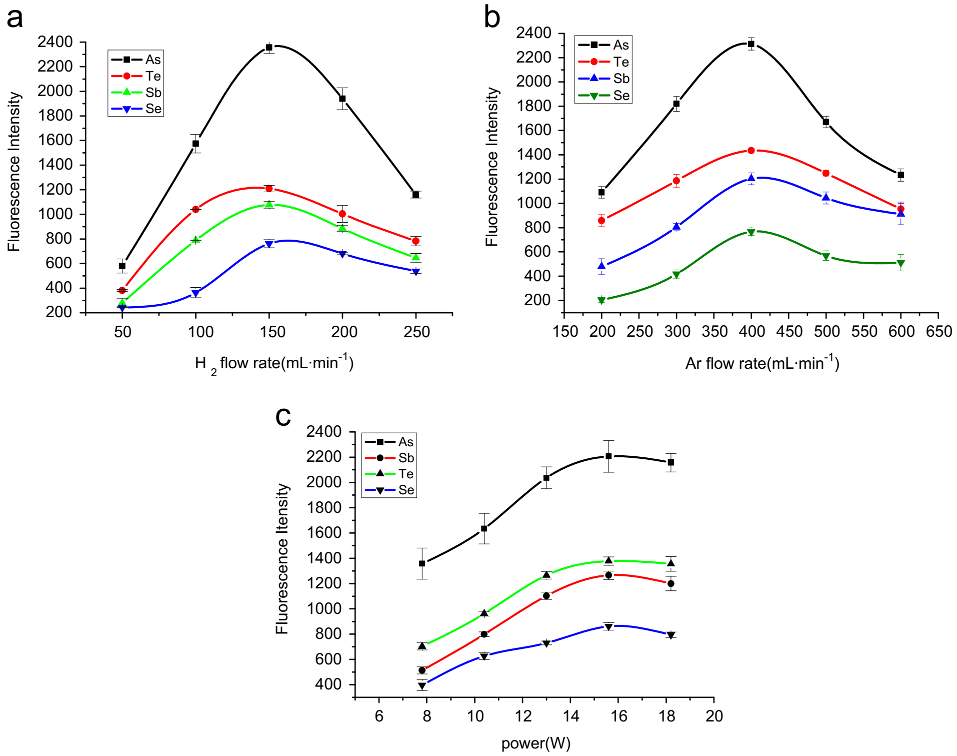

Fig. 3. Optimization of different parameters.

fluorescence signal intensities versus sample concentrations was linear over the range of 0.5–20 μg mL^{-1} with a correlation coefficient of 0.9998 for both As and Sb. The linearity of Te was ranged from 0.5 to 20 μg mL^{-1} with a correlation coefficient of 0.9999. Se concentration was linear over the range of 0.5–50 μg mL^{-1} with a correlation coefficient of 0.9999. These results indicate that the present method shows good precision performance. The relative standard deviations (RSDs) of As, Te, Sb and Se in this method, shown in Table 2 are 4.1%, 1.4%,

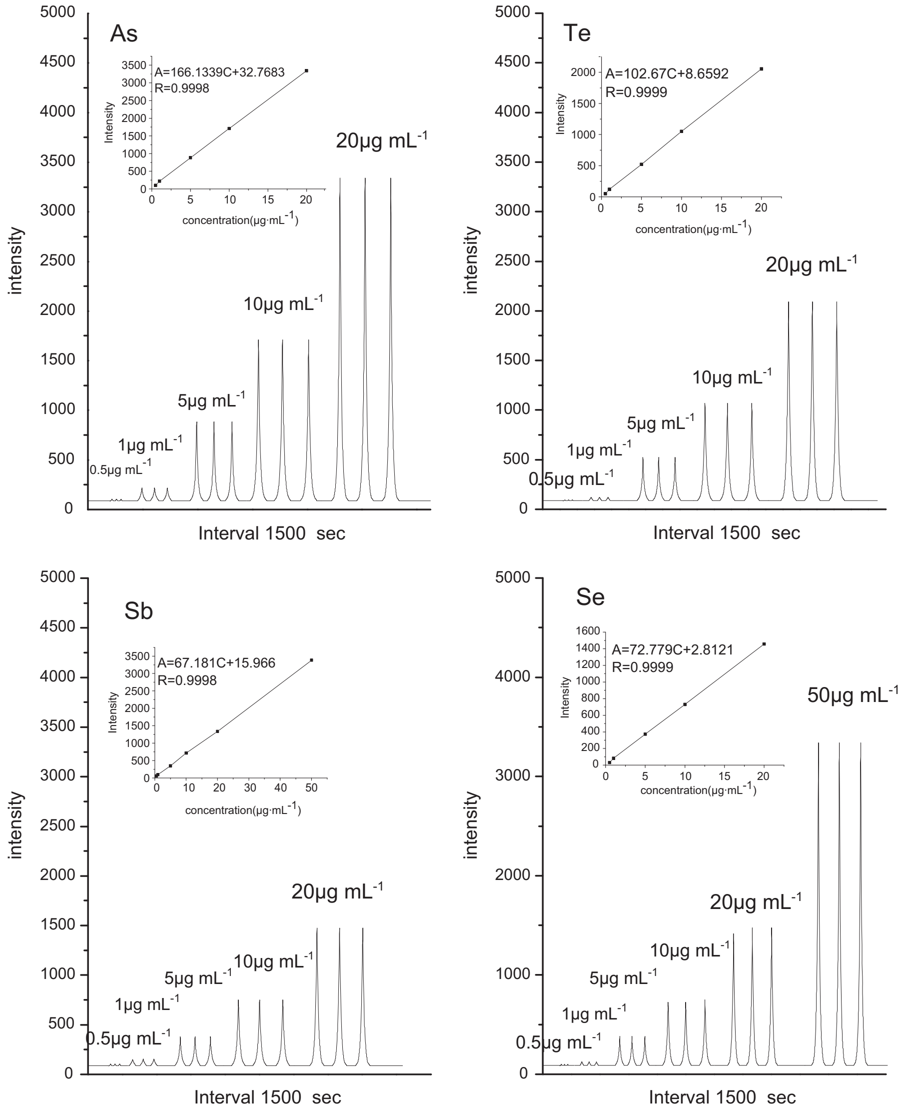

Fig. 4. Typical signal calibration curve and fluorescence signals of As, Te, Sb and Se under different concentrations.

Table 2
Figures of Merit.

DBD-hydrogen plasma coupled with AFS detection	Arsenic	Tellurium	Antimony	Selenium
Precision (RSD[a] %)	4.1	1.4	2.9	1.1
Relative DL (μg mL^{-1})	0.03	0.05	0.07	0.06
Absolute DL (ng)[b]	0.6	1.0	1.4	1.2

[a] 10 μg mL^{-1}, $n=11$.

[b] Absolute DL=relative detection limit × minimum injection volume. The minimum injection volume was 20 μL in this test.

2.9% and 1.1%, respectively. The absolute detection limits for As, Te, Sb and Se are 0.6 ng, 1.0 ng, 1.4 ng and 1.2 ng, respectively.

The present method was validated by analyzing certified reference materials, GBW07311, GBW08517 and GSB-14, which also demonstrated the accuracy of the established method. A comparison of the results obtained by ICP-MS, conventional HG-AFS and the present method is shown in Table 3. Most of results are in good agreement with the certified values, and indicate that the four elements can be detected at the same time by the proposed method. Moreover, the present method shows good analytical performance in the detection of As, Te and Sb.

3.5. Arsenic speciation by HPLC–DBD hydrogen plasma – Atomic Fluorescence Spectrometry

To evaluate the usefulness of the developed method for the speciation analysis, an attempt was made to couple HPLC with DBD-hydrogen plasma–AFS for the analysis of four arsenic species. The chromatogram of arsenic species standard solutions is shown in Fig. 5. The analytical characteristic data of the present HPLC with DBD-hydrogen plasma–AFS for speciation of the four arsenic species are summarized in Table 4. The precisions (RSD) of the migration time and peak areas for seven replicate injections of a mixture of 0.6 μg mL^{-1} of four individual species were 4.8%, 3.8%, 6.5% and 3.8% for arsenite (As(III), arsenate (As(V), monomethylarsenic acid (MMA), and dimethylarsenic acid (DMA), respectively. Limits of detection for the four species based on peak area measurement ranged from 0.02 to 0.08 μg mL^{-1}. The results in Table 4 show that the proposed method is suitable for analyses of the four arsenic species.

Table 3
Results for As, Te, Sb and Se determination in samples.[a,b]

Samples	Certified value (μg g^{-1})	ICP-MS (μg g^{-1})	Conventional HG-AFS (μg g^{-1})	This work (μg g^{-1})
GBW07311				
Arsenic	188 ± 20	178.4 ± 3.8	177.5 ± 2.2	179.7 ± 2.7
Tellurium	(0.36)	0.300 ± 0.005	0.290 ± 0.002	nd
Antimony	14.9 ± 1.8	12.2 ± 0.2	13.2 ± 0.1	14.1 ± 0.1
Selenium	0.20 ± 0.06	0.200 ± 0.001	0.280 ± 0.005	nd
GBW08517				
Arsenic	13.9 ± 2.4	13.8 ± 0.5	14.6 ± 0.4	13.6 ± 0.3
Tellurium	nd	nd	nd	nd
Antimony	(0.066)	0.078 ± 0.009	0.071 ± 0.005	nd
Selenium	0.062 ± 0.009	0.060 ± 0.001	0.059 ± 0.002	nd
GSB-14				
Arsenic	27 ± 6	23.8 ± 1.7	25.3 ± 1.1	25.8 ± 1.8
Tellurium	nd	nd	nd	nd
Antimony	0.026 ± 0.006	0.023 ± 0.002	0.027 ± 0.001	nd
Selenium	0.124 ± 0.014	0.119 ± 0.006	0.121 ± 0.002	nd

[a] Results are given as average ± standard deviation ($n=3$).
[b] nd = not detectable, less than detection limit.

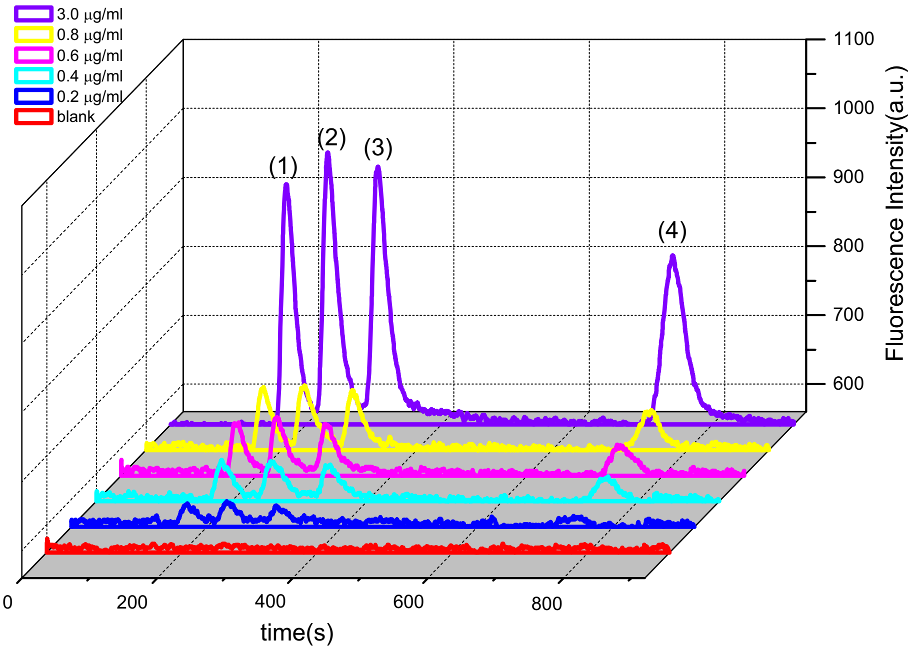

Fig. 5. Chromatogram of arsenic species standard solutions obtained by the HPLC–DBD-hydrogen plasma–AFS system: (1) As(III), (2) DMA, (3) MMA, (4) As(V).

Table 4
Optimal operational parameters and analytical performance of the HPLC–DBD-hydrogen plasma–AFS system for the arsenic speciation analysis.

Analyte	As(III)	DMA	MMA	As(V)
Parameters value				
Eluent concentration	a buffer solution of 15 mmol L^{-1} p-Hydroxybenzoic acid			
Elution flow rate (mL min^{-1})	1.0	1.0	1.0	1.0
The pH of elution	6.0	6.0	6.0	6.0
Analytical performance				
Calibration function (A, signal intensity; C, μg mL^{-1})	$A=3163.56C+30.09$	$A=3996.84C+31.52$	$A=4018.47C+61.41$	$A=3775.26C$
Linear range (μg mL^{-1})	0.2–3	0.2–3	0.2–3	0.2–3
Correlation coefficient	0.9977	0.9989	0.9971	0.9993
Detection limit (μg mL^{-1}, $n=11$)	0.03	0.02	0.08	0.05
RSD (%, 0.6 μg mL^{-1}, $n=7$)	4.8	3.8	6.5	3.8

4. Conclusions

We have demonstrated the feasibility of the proposed hydrides generation system for the formation of chemical vapors by a low temperature hydrogen plasma technique. Instead of using those consumptive chemical agents to produce the hydrides, the DBD method avoids the massive use of chemical reagents, thus providing low cost and easy to operate analysis tools for the elemental analysis. Some attractive perspectives of the present DBD-hydrogen plasma are friendly to environment, fast to analyze and easy to implement. The method was validated by analyzing certified reference materials, and showed good performance in the determination of As, Te and Sb. The most attractive characteristic of the proposed method is the micro-sampling, which is especially suitable for small quantities of sample solutions. Moreover, the speciation analysis of As(III), As(V), MMA and DMA by HPLC–DBD-hydrogen plasma–AFS can be easily carried out. In principle, the method offers potential advantages of miniaturization, less contamination and consumption, and ease of automation.

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