Talanta 81 (2010) 516–520

Contents lists available at ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Determination of tin by in situ trapping of stannane on a resistively heated iridium treated tungsten coil surface and interference studies

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ARTICLE INFO

Article history:
Received 6 August 2009
Received in revised form
10 December 2009
Accepted 21 December 2009
Available online 4 January 2010

Keywords:
Hydride generation
Stannane
Iridium treated tungsten coil
In situ trapping
Preconcentration
Interference

ABSTRACT

A novel method was developed for the in situ trapping of stannane on an iridium-coated tungsten coil. Coating the tungsten coil with iridium has significantly improved the sensitivity. The tungsten coil can either be used as an on-line atomizer or as a trapping surface. The interference effect of some hydride-forming elements such as, As(III), Se(IV), Te(IV), Sb(III) was investigated. The interference effect of Sb(III) and Se(IV) could not be completely eliminated using in situ trapping mode but the magnitude of their interferences was reduced significantly when compared to quartz T-tube atomizer. The limit of detection with iridium-coated tungsten coil for a 60 s trapping period (sample volume 6 ml) was found to be 0.065 ng ml^{-1} and the calibration was linear over the range of 0.5–4.0 ng ml^{-1}. The precision of the analytical method was determined to be 2.2% RSD ($n = 11$) for 1.0 ng ml^{-1} Sn concentration. Analytical performance of the proposed method was checked by analyzing tap water, spring water and mineral water samples for Sn. The accuracy of the method was tested with two different certified reference materials; fortified water TMDA 61 (NWRI) and Dogfish Liver DOLT-3 (NRC). The results were in a good agreement with the certified values at 95% confidence level.

1. Introduction

The determination of tin at trace levels is of interest due to its toxicity for living organisms. Hydride generation atomic absorption spectrometry (HGAAS) is widely used for trace element determination because it is simple and relatively free from interferences when compared to electrothermal atomic absorption spectrometry (ETAAS).

In the last decade, various metal atomizers such as platinum, molybdenum, tungsten and tantalum were used as alternative to graphite tubes. Among these metal atomizers, the use of tungsten coil (W-coil) has been reported most frequently [1,2]. The main advantage of W-coil is that it requires a simple power supply when compared to graphite furnace. Moreover, the heating rate of W-coil is approximately 10 times faster than that of the graphite tube. In addition, it can be easily adapted to portable instrumentation [3]. W-coils can be used either as an on-line atomizer [4,5] or as a trapping surface in HGAAS.

In situ trapping is used for preconcentrating the hydride-forming elements on the atomizer surface prior to the atomization step which is the main reason behind using graphite tubes [6]. Coating the graphite tube surface with platinum group metals (PGMs) [7,8] or carbide forming elements [9,10] improves the trapping efficiency for the determination of stannane. In a recent publication, in situ trapping techniques other than graphite tubes were reviewed by Ataman [11].

Despite the fact that there are many studies using graphite tubes for in situ trapping of the generated hydrides, studies involving a W-coil as a trapping surface are rather limited. Docekal and Marek [12] determined Se and As hydrides by in situ trapping in a W-tube atomizer and found that trapping efficiency was improved by coating the trapping surface with Pt, Ir and Re. Also, Barbosa et al. [13] used Rh-coated W-coil for in situ trapping of selenium hydride and reported that the coating was stable for at least 300 measurements. Cankur et al. [14] trapped bismuth hydride on a resistively heated W-coil which was placed in the inlet arm of a quartz tube; then the trapped species were revolatilized and transported to flame heated quartz T-tube atomizer. The same device was used for trapping Se, Sb hydrides and Cd species. In the determination of Cd, the quartz T-tube atomizer was not heated [15–17]. Souza et al. [18] determined Se and As in biological and water samples by in situ hydride trapping on a W-coil. There are several studies of in situ trapping of stannane in graphite tubes [8,9,19–21] but, only limited studies have been performed to determine tin using hydride generation with W-coil without trapping [22,23].

The novelty of this study is to demonstrate that W-coil can be used as in situ trapping surface for the determination of tin. Also, the interference effects of some hydride-forming elements were compared to the flame heated quartz T-tube atomizer. To the best

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doi:10.1016/j.talanta.2009.12.035

of our knowledge, this is the first study reported for in situ trapping of stannane on a W-coil.

2. Experimental

2.1. Instrumentation

All measurements were performed using a Unicam 939 atomic absorption spectrometer equipped with a deuterium background corrector. A Sn hollow cathode lamp (SCP Science) was operated at 12 mA. The wavelength was set to 224.6 nm and the spectral band pass was 0.5 nm. For conventional hydride generation atomic absorption spectrometry (HGAAS), a flame heated quartz T-tube atomizer was used which had an optical path length of 120 mm and an i.d. of 8.0 mm; the inlet arm was 80 mm in length and 3.0 mm in i.d.

The W-coil obtained from 150 W, 15 V projector bulb (Osram), was placed in the middle of the glass cell from an open hole, sealed and fixed by play dough. The glass cell has an optical path length of 120 mm and an i.d. of 13 mm; the inlet arm was 40 mm in length and 4.0 mm in i.d.

The coil temperature was manually controlled by a variable potential power supply and a transformer. The transformer was used to obtain a 24 V output from the main electricity (220 V ac). The temperature of the W-coil during the trapping period was measured using a Pt/Rh thermocouple (TETCIS, Ankara) which was capable of measuring temperatures up to 1800 °C. During the measurements the tip of the thermocouple was kept at 1 mm in front of the central part of the W-coil, while a gas mixture of Ar and H_2 was flowing with an optimized composition and flow rate. Therefore, the measured trapping temperatures were those of the gas phase. The atomization temperatures were measured using a TI315 series portable infrared thermometer (TIME, Beijing, China) which was capable of measuring temperatures between 500 and 3000 °C.

2.2. Hydride generation manifold

Continuous flow hydride generation system was constructed using 1.85 mm i.d. Tygon peristaltic pump tubings (Ismatec). The connecting tubings, reaction and stripping coils (PTFE with 0.8 mm i.d.) were supplied from Cole Parmer (USA). Two peristaltic pumps were used. A three channel ALITEA VS 3 (Sweden) was used to pump the acidified sample and the reducing agent at a flow rate of 6 ml min^{-1} and a Gilson Minipuls 3 (France) was used for draining gas–liquid separator (GLS). A home-made cylindrical GLS was employed. A three-way connector was placed between GLS and the atomization cell to introduce hydrogen gas to the atomizer to prevent oxidation of the W-coil. Polypropylene tubings (4 mm i.d.) with 40 and 60 mm in length were used for the connections between GLS, the three-way connector and the atomization cell. The schematic diagram of the continuous flow hydride generation manifold is shown in Fig. 1.

2.3. Reagents

All working solutions were prepared in deionized water (18.2 MΩ cm) which was obtained from Millipore (Simplicity, 185 water purification system). Carrier gases of Ar and H_2 were obtained from Habaş, Turkey. Sample and standard solutions were prepared in HCl (Riedel de Haen) by appropriate dilutions. $NaBH_4$ (min. 96% purity, Aldrich), dissolved in 0.02% (w v^{-1}) NaOH (Merck), was used as a reducing reagent. Sn(II) standards were prepared by appropriate dilutions of a 1000 mg l^{-1} Sn(II) (High Purity Standards, SC, USA) stock solution. In order to improve trapping efficiency a 1000 mg l^{-1} Ir (Accustandard) standard was used for coating the W-coil. For interference studies, Sb(III) (High Purity Standards), As(III), Te(IV), Se(IV) (Fisher Scientific) standards were prepared by appropriate dilutions of stock 1000 mg l^{-1} solutions. Certified reference materials, TMDA-61, trace element fortified water obtained from NWRI (Canada) and Dogfish Liver DOLT-3 from NRC (Canada), were used to test the accuracy of the proposed method.

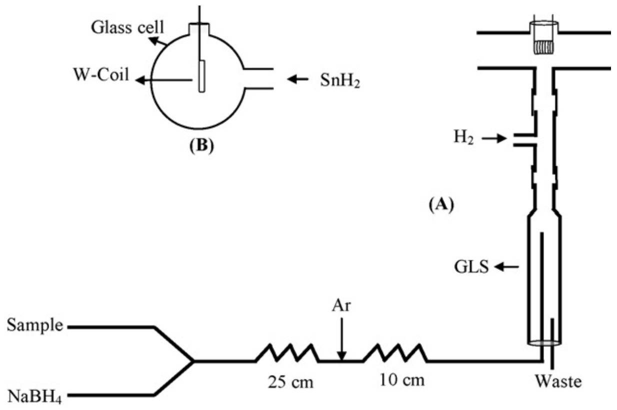

Fig. 1. Scheme of CF-HGAAS set up (A) and side view of the W-coil atomizer (B). GLS: gas liquid separator.

2.4. Sample preparation

0.2 g of DOLT-3 Dogfish Liver CRM was weighed into a PTFE beaker and 10 ml of concentrated HNO_3 was added and heated gently, till dryness. Then, 2 ml of concentrated HCl was added to the residue and heated to 85–90 °C for 20 min. After being cooled, the samples were diluted to 50 ml with deionized water and analyzed for tin. TMDA 61 trace element fortified water samples were analyzed for tin after dilution 25 times with 0.5 M HCl. Tap water and spring water samples were prepared in 0.5 M HCl. Mineral water was sonicated for 45 min for degassing; then, prepared in 0.5 M HCl.

2.5. Ir coating procedure

The coating was performed by manual injection of 25 μl aliquots of 1000 mg l^{-1} Ir stock solution on the W-coil and the temperature program shown in Table 1 was applied. The same procedure was repeated 4 times to increase deposited amount of iridium on the W-coil. During the coating procedure, H_2 and Ar flow rates were 175 and 150 ml min^{-1}, respectively.

2.6. General procedure

In on-line atomization mode, the acidified sample and $NaBH_4$ were pumped at a constant flow rate of 6 ml min^{-1} by a peristaltic pump. The generated stannane was transported to the W-coil atomizer which had been heated to the atomization temperature (1900 °C) in argon stream; a continuous signal was observed. The same continuous flow system was used with the flame heated quartz T-tube atomizer.

When the W-coil was used as a trapping surface, the coil was heated to trapping temperature (300 °C) and stannane was col-

Table 1
Temperature program for Ir coating.

	Time (s)	Temperature (°C)
Step 1	45	175
Step 2	30	600
Step 3	2	1300

Table 2
Interference studies, Sn concentration 5.0 ng ml^{-1} for W-coil trapping, 50.0 ng ml^{-1} for quartz T-tube atomizer and on-line W-coil atomizer.

		Concentration ratio (interferent/Sn)						
		0	10	50	100	200	300	400
		Relative signal (%)						
Te	Quartz T-tube	100	106	43	31	27	20	19
	W-coil on-line	100	91	82	78	88	73	81
	W-coil trapping	100	102	99	101	104	100	99
Sb	Quartz T-tube	100	112	88	68	45	32	24
	W-coil on-line	100	98	84	84	72	65	52
	W-coil trapping	100	100	81	65	56	56	54
Se	Quartz T-tube	100	22	11	9	10	12	8
	W-coil on-line	100	36	14	13	15	12	12
	W-coil trapping	100	101	78	49	45	22	26
As	Quartz T-tube	100	91	58	50	35	28	26
	W-coil on-line	100	97	76	63	49	40	39
	W-coil trapping	100	101	98	93	83	83	83

lected on the coil surface for a certain period. The pump was stopped and the connector between the power supply and the W-coil was switched off. The applied voltage was set to atomization temperature and the connector was switched on; a sharp transient signal was observed. Hydrogen gas was introduced to both the W-coil and quartz T-tube atomizer. The flow rates of hydrogen and argon gases were 175 ml min^{-1} and 125 ml min^{-1} respectively, in all measurements. Peak height absorbance values were measured throughout the experiments, except the trapping efficiency was calculated using peak areas of the signal.

The interferences were investigated by using a constant concentration of the analyte in the presence of a successively increasing amount of the interfering element. The oxidation states of the interfering elements were As(III), Sb(III), Se(IV), and Te(IV). The results are presented in Table 2 as relative signal % versus ratios of interfering element to analyte concentrations. The absorption measured with analyte solution was normalized to 100%.

3. Results and discussion

3.1. Optimization studies

The effects of: the concentrations of $NaBH_4$ and HCl, gas flow rates for Ar and H_2, trapping and atomization temperatures, coating mass of Ir and trapping period, on the absorbance signal of tin were studied. A concentration of 5.0 ng ml^{-1} Sn and 30 s trapping period were used for the optimization studies except for the optimization of trapping time.

3.2. Optimization of $NaBH_4$ and HCl concentration

The concentration of $NaBH_4$ was studied in the range of 1.0–2.5% (w v^{-1}). The signal increased significantly up to 1.5% and reached a plateau. Therefore 1.5% was used throughout the experiments. NaOH in the reducing agent was the main source of blank signal during the preliminary studies. Its concentration was decreased to 0.02% (w v^{-1}) and no significant signal was observed from the blank solution even for longer trapping periods.

HCl concentration was studied in the range of 0.2–2.0 mol l^{-1}. The absorbance signal increased up to 0.4 mol l^{-1} and reached a plateau between 0.4 and 1.0 mol l^{-1} HCl concentrations. At concentrations higher than 1.0 mol l^{-1} HCl, significant decrease in absorbance signal was observed. Therefore, 0.5 mol l^{-1} HCl was selected as an optimum concentration.

3.3. Optimization of gas flow rates

Hydrogen gas is necessary in order to prevent oxidation of the W-coil. Oxidation of the W-coil could be observed visually after a few cycles of trapping and atomization when flow rates lower than 75 ml min^{-1} were applied. Therefore, hydrogen gas flow rate was studied in the range of 75–250 ml min^{-1}.The absorbance signal significantly increased up to 150 ml min^{-1} and stayed constant up to 250 ml min^{-1}. A flow rate of 175 ml min^{-1} was used throughout the experiments.

Flow rate of Ar gas was studied in the range of 50–250 ml min^{-1}. The maximum absorbance signal was obtained for flow rates between 100 and 150 ml min^{-1}; 125 ml min^{-1} was selected as an optimum value.

3.4. Optimization of trapping and atomization temperatures

The trapping temperature was studied between 100 and 450 °C and during the optimization, atomization temperature was set to 1900 °C. An important observation here was that the generated stannane could be trapped on the surface of the W-coil even at low temperatures (100 °C). Increasing the trapping temperature up to 300 °C increased the absorbance signal significantly as shown in Fig. 2. At temperatures higher than 350 °C, a sharp decrease in signal was observed due to partial release of the analyte from the trapping surface. The highest signal was observed at 300 °C, therefore, this value was selected as an optimum trapping temperature. In order

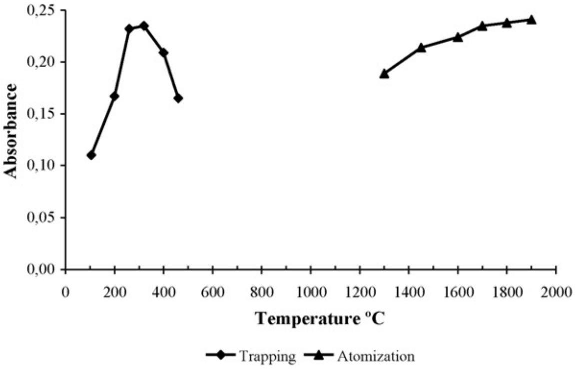

Fig. 2. Optimization of trapping and atomization temperatures using Ir-coated W-coil. Trapping time 30 s, 5.0 ng ml^{-1} Sn; atomization temperature was 1900 °C as the trapping temperature was varied; trapping temperature was 300 °C as the atomization temperature was varied.

Table 3
Analytical figures of merits for Sn determination with three modes of atomization.

Parameter	W-coil (trapping)	W-coil	Quartz T-tube
LOD, ng ml^{-1}	0.065	1.3	1.2
Characteristic concentration, ng ml^{-1}	0.049	1.1	1.0
Linear range, ng ml^{-1}	0.5–4.0	10–120	10–120
Calibration equation[a]	$y = 0.0922[\mathrm{Sn}] + 0.001$	$y = 0.0034[\mathrm{Sn}] + 0.0007$	$y = 0.0036[\mathrm{Sn}] + 0.0008$
Sample volume	6.0 ml	–	–
Trapping time	60 s	–	–

[a] y is absorbance, [Sn] is concentration of Sn in ng ml^{-1}.

to determine the optimum atomization temperature, the trapping temperature was set to 300 °C and the atomization temperature was varied between 1350 and 1950 °C. Absorbance increased up to 1700 °C and stayed constant. Therefore, 1900 °C was taken as an atomization temperature. The trapped tin species were stable on the W-coil surface for at least 5 min; the same signal was observed when atomization was achieved just after the trapping period or 5 min later.

It is known that coating the trapping surface with Pt, Pd, Ir, Rh improves the trapping efficiency [8]. Although, stannane can be trapped and atomized on uncoated W-coil surface, Ir was preferred as a coating element since the signal increased 2.5 fold as compared to the uncoated W-coil when it was coated with 50 μg Ir. The effect of coating mass was investigated in the range of 0–200 μg. The absorbance signal was practically the same for 50, 100, 150 and 200 μg of Ir; a 100 μg iridium coating was used for the rest of the experiments in order to assure the signal stability for a longer time.

3.5. *Analytical figures of merit*

Analytical figures of merit were calculated using peak height of the absorbance signal; the results are shown in Table 3. The calculated LOD value using 60 s trapping period was 0.065 ng ml^{-1}. The enhancement factors for the LOD value were found as 20 and 18 when compared to W-coil on-line atomization and quartz T-tube atomizer, respectively. The effect of trapping period on the signal was investigated between 30 and 120 s. Increase in the absorbance signal was linear with the trapping period in the studied range. It is possible to use higher trapping periods than 60 s in order to achieve lower LOD values since the blank values were insignificant but this would in turn increase the analysis time. The trapping efficiency of the method was calculated as 44% by comparing the peak areas of the signals obtained by using flow injection W-coil without trapping (60 ng ml^{-1} Sn, 250 μl loop volume) and in situ trapping with the W-coil (6 ml of 2.5 ng ml^{-1} Sn).

3.6. *Determination of Sn in water samples*

The reliability of the proposed method for tin determination in water samples was examined by analyzing tap water, spring water and mineral water samples. High sensitivity provided by the W-coil in situ trapping method allows the determination of tin in water samples. The samples were analyzed by using 60 s trapping period. Tin concentrations were successfully determined in water samples except in tap water sample was around detection limit therefore, it was not quantified. Recovery from the spiked samples were also studied and the results obtained are shown in Table 4.

3.7. *Determination of Sn in CRM's*

Accuracy of the proposed method was checked by analyzing certified reference materials; TMDA-61 fortified water and DOLT-3 Dogfish Liver. External calibration was used for the determination of tin in both CRMs. The results were in agreement with the certified values as shown in Table 5.

Table 4
Determination of Sn in water samples using W-coil in situ trapping mode, collection time was 60 s.

	Found (ng ml^{-1})	Spiked (ng ml^{-1})	Found (ng ml^{-1})
Tap water	ND	0.5	0.60 ± 0.03
Spring water	0.63 ± 0.03	0.5	1.08 ± 0.05
Mineral water	1.70 ± 0.12	1.0	2.61 ± 0.10

Results are average value ± standard deviation ($n = 5$).

3.8. *Interference studies*

The interference effect of the hydride-forming elements, As(III), Sb(III), Te(IV) and Se(IV) was investigated and the results were compared with the quartz T-tube atomizer and W-coil on-line atomization.

During interference studies with the quartz T-tube atomizer, hydrogen gas was introduced at the same flow rate (175 ml min^{-1}) in order to have similar atmospheres in both atomizers. Memory effects due to the deposition of interferent species on the surface of the quartz T-tube atomizer were eliminated by pumping a blank solution and by measuring the interferent-free analyte standard solution. No memory effect was observed with the W-coil atomizer. A 50 ng ml^{-1} Sn concentration was used for on-line atomization modes throughout the interference studies.

The magnitude of interferences from As(III), Sb(III) and Te(IV) was reduced significantly using the W-coil on-line atomization mode as compared to the quartz tube atomizer. Although all of the experimental parameters were the same, the atomization temperature was the main difference [25]. The highest interference was observed from selenium for all atomization modes.

The magnitude of interferences decreased with the proposed method, in situ trapping on Ir-coated W-coil surface; Te(IV) interference was completely eliminated in the studied concentration range and interference due to As(III) was reduced significantly (Table 2). Moreover, at low concentrations of interferent/analyte ratios, Se(IV) interference was reduced as compared to the W-coil on-line atomization. However, the interference effect of Sb was relatively higher with the trapping mode. As mentioned in our previous study [24], the reason for this is the fact that trapping temperatures of stibine and stannane are very close to each other. Therefore, most probably stibine is also preconcentrated during the trapping period resulting in a more effective suppressing of the analytical signal with relatively higher concentrations of Sb.

The main reason for the reduced interferences with the trapping mode was the use of diluted solutions and partial separation of interfering species during the trapping period. A series of experiments with in situ trapping mode were performed using 2 and

Table 5
Determination of Sn in CRMs ($n = 3$).

CRM	Certified value	Found
Fortified water TMDA 61, μg l^{-1}	59.6 ± 10.2	57.6 ± 1.9
Dogfish Liver, DOLT-3, μg g^{-1}	0.4[a]	0.41 ± 0.02

[a] Information value.

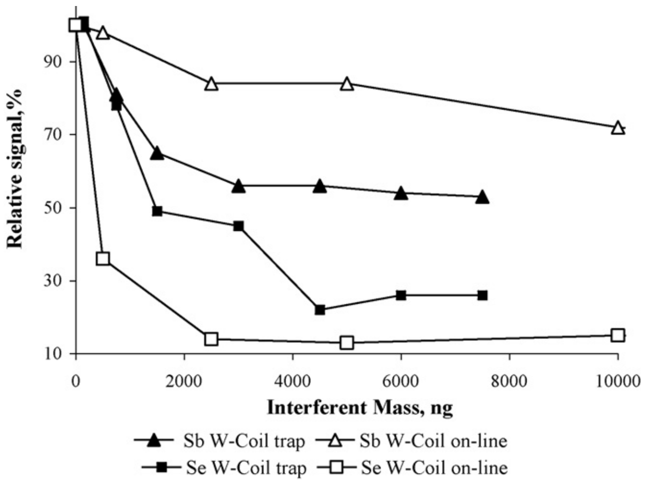

Fig. 3. Comparison of the interference effect of antimony and selenium in terms of interferent mass on Sn signal using the W-coil atomizer with trap and on-line atomization modes. Trapping time 30 s, 5.0 ng ml^{-1} Sn; for in situ trap, 50 ng ml^{-1} Sn and a 0.5 ml sample loop was used for flow injection on-line atomization mode. The atomization temperature was 1900 °C in both cases.

5 ng ml^{-1} Sn concentrations while keeping the interferent/analyte ratios constant. The recovered signals were significantly higher for 2 ng ml^{-1} tin for all interfering elements because the interference magnitude depends on the total mass of the interferent transported to the atomizer.

A second series of experiments were performed using flow injection and on-line atomization in order to introduce a definite amount of interfering elements to the W-coil atomizer. The concentrations of interferent were selected such as to keep the total masses of the interfering species the same for both in situ trapping and on-line atomization. The recovered signals were significantly improved for Te, As and Se while Sb suppressed the analyte signals more in the trapping mode. Fig. 3 shows the interference effect for trapping and on-line atomization modes for Sb and Se in term of mass of interfering element. These results can be attributed to the partial elimination of the interfering species except for Sb during the trapping period.

4. Conclusion

A novel method for the determination of tin has been developed by using an Ir-coated W-coil. The W-coil can either be used as an on-line atomizer or as a trapping surface. The LOD values obtained with on-line atomization using the W-coil was comparable to the quartz T-tube atomizer. Improvement in sensitivity was achieved by trapping the analyte on the W-coil surface. Iridium coating enhances the absorbance signal 2.5 times when compared to uncoated W-coil and the same coating can be used at least 250 consecutive measurements without any loss of sensitivity. The enhancement factor for LOD value for 60 s trapping period was found as 18 with respect to quartz T-tube atomizer.

The performance of the in situ trapping method was verified by analyzing water samples. The accuracy of the method was also tested with CRM's. The results obtained were in good agreement with certified or information values.

The interferences are decreased by using W-coil as an on-line atomizer compared to a quartz tube atomizer. The interference effect of tellurium hydride was completely eliminated in the trapping mode and decreased for arsenic, antimony and selenium hydride. This can be attributed to partial elimination of interfering species during the trapping period and the decrease in the total mass transported to the atomizer since diluted solutions were used. Another advantage of the W-coil atomizer over the quartz tube atomizer was that memory effect in the presence of high concentrations of the interfering species was eliminated.

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