# Determination of antimony by using tungsten trap atomic absorption spectrometry ☆

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## ABSTRACT

An electrically heated tungsten coil was used as a trap in the determination of antimony. The technique consists of three steps. Initially, $SbH_3$ is formed by hydride generation procedure; then the analyte species in vapor form are transported to W-coil trap heated at 370 °C. Following the preconcentration step, the trap is heated to 895 °C; analyte species are revolatilized and transported to the flame-heated quartz atom cell where atomization and the formation of signal take place. The experimental parameters were optimized both for trap and no-trap studies. The most important experimental parameters are concentrations of HCl and $NaBH_4$ solutions, $H_2$ and Ar gas flow rates, and collection and revolatilization temperatures of W-coil. Accuracy was tested using a certified reference material, waste water EU-L-1. Limit of detection for the system is 16 ng $l^{-1}$ using a sample of 36 ml collected in 4.0 min. Enhancement factor in sensitivity was 17.

## 1. Introduction

The issues regarding antimony have been extensively studied due to high toxicity of this element. The concentration of antimony in earth's crust varies between 0.2 and 0.5 μg $g^{-1}$ in general. Different chemical forms of this element are possible such as chalcophile, occurring with sulfur and some heavy metals such as lead, copper, and silver. Although the oxidation states of Sb can be −III, 0, III and V, it is mostly found in two oxidation states (III and V) in environmental samples. These two forms exhibit pronounced differences in their analytical behavior, toxicity and mobility [1]; inorganic compounds of antimony are more toxic than its organic forms; toxicity of Sb(III) has been shown to be 10 times higher than that of Sb(V).

Typical concentrations of total dissolved antimony in non-polluted waters range from ng $l^{-1}$ to μg $l^{-1}$ [2]. Antimony and many of its compounds are also considered as pollutants of priority interest by the United States Environmental Protection Agency (USEPA) and European Union. The USEPA drinking water standards for maximum contaminant level goal (MCLG) and maximum con-taminant level (MCL) are both 6 μg $l^{-1}$ [3]. According to the European Union the admissible level for this element is 5 μg $l^{-1}$ in drinking water [4].

Among the techniques for determination of antimony, flame atomic absorption spectrometry (FAAS) and hydride generation atomic absorption spectrometry (HGAAS) require only a simple flame AAS instrument and some attachments; therefore these techniques in principle provide the most economical alternatives. Hydride genera-tion (HG) is one of the most popular and powerful techniques of vapor generation for chemical analysis [5]; although in principle HG is a sample introduction technique and thus can be coupled to any atomic detector including atomic fluorescence spectrometry (AFS), induc-tively coupled plasma optical emission spectrometry (ICPOES) and inductively coupled plasma mass spectrometry (ICPMS), HGAAS is the most often used one due to its simplicity and low cost.

Some recent applications for determination of antimony include diverse techniques such as AFS [6], electrothermal atomic absorption spectrometry (ETAAS) [7], ICPMS [8] and instrumental neutron activation analysis (INAA) [9] and stripping voltammetry [10].

Due to low concentration of Sb in body fluids and environmental samples, some preconcentration techniques are often required in order to improve sensitivity of simple AAS instruments that are available in most laboratories so that these systems can then be employed in economical antimony determination using FAAS or HGAAS. Chemical preconcentration on a microcolumn [11] and cloud point preconcentration [12,13] are few recent examples. A recent approach for sensitivity enhancement is trapping; applications in

literature are the use of hydride generation and integrated atom trap [14], molybdenum foil strip [15], quartz traps coupled to chemical hydride generation [16,17] and electrochemical hydride generation [18]. Another sort of trapping technique coupled to HGAAS [19] or cold vapor (CV) AAS [20] is the use of a tungsten coil for *in-situ* preconcentration.

Atom trapping provides relatively low limits of detection at ng $l^{-1}$ level. In the first application of a quartz trap HGAAS system for antimony, limit of detection was found to be 3.9 ng $l^{-1}$ for a sample of 8.0 ml collected in 120 s [16]; with improvements especially in use of an oxygen flow in collection step, limit of detection was 2.8 ng $l^{-1}$ for a sample of 20 ml collected in 300 s [17]. Using electrochemical hydride generation, sensitivity was somewhat lower and a detection limit of 53 ng $l^{-1}$ was obtained for a sample of 6.0 ml collected in 120 s; sensitivity was improved by a factor of 18 as compared with electrochemical hydride generation [18].

The aim of this study was to develop a sensitive analytical method for determination of Sb using a W-coil trap coupled to a chemical HGAAS system. One of the previous applications using a W-coil atom trap coupled to HGAAS has been used in determination of bismuth [19]. The method for Sb involves generation of stibine in an acidic medium by reaction with tetrahydroborate (III); then the volatile analyte species are preconcentrated on a W-coil trap that was previously heated to an optimized collection temperature. At the end of the collection period, collected analyte species are revolatilized by heating the trap to a higher temperature under an optimized mixture of argon and hydrogen gases; the species are transported to an externally heated quartz atomizer; finally, a transient signal is obtained in less than 0.5 s.

## 2. Experimental

### 2.1. Instrumentation

An ATI Unicam 929 atomic absorption spectrometer equipped with a deuterium background correction system was used. A Philips data coded hollow cathode lamp was operated at 15 mA; the analytical line at 231.2 nm was selected. The experimental set-up is similar to the one reported previously [19]; the differences will be noted below. A quartz T-tube heated by a stoichiometric air/acetylene flame was used as atomizer. A Gilson Minipuls 3 (Villers Le Bell, France) 4-channel peristaltic pump was used in all continuous flow (CF) analyses. Tygon peristaltic pump tubing of 0.8 mm i.d. (Ismatec, Germany) was used for pumping the analyte and reductant solutions were mixed in a 3-way PTFE connector (Cole Parmer Instrument Co. USA). Flow rates of Ar and $H_2$ gases were measured by two separate flow meters (Cole Parmer Instrument Co. USA). For the separation of Sb hydride from the liquid phase, a laboratory-made cylindrical gas liquid separator (GLS) was used. Tygon tubing having 40 mm of length and 6.0 mm of i.d. (Masterflex, Cole Parmer Instrument Co.) was used between GLS and the inlet arm of the quartz T-tube atomizer. Quartz atomizer consisted of a horizontal arm with dimensions of 115 mm in length, 15 mm in o.d. and 13 mm in i.d.; inlet arm was 110 mm in length, 10 mm in o.d. and 7.0 mm in i.d. Another smaller quartz tube was inserted and fixed in the inlet arm with dimensions of 6.0 mm in o.d. and 4.0 mm in i.d.; it extended between the open end of the inlet arm and a point that was 5.0 mm away from the W-coil. The W-coil used for trapping was obtained from a 15 V, 150 W projector bulb (Halogen Photo optic Lamp Xenophot, Osram, Germany) and placed in the inlet arm of the quartz T-tube, 50 mm away from the connection point to the horizontal arm. Tungsten wire was shaped into a flattened coil to occupy a cross sectional area of 3.4×4.5 mm. In order to insert and hold the W-coil in its place, a somewhat different design was employed as compared with the previous one [19]. This new configuration allows a very rapid and convenient change of W-coil when needed [21]. The coil temperature was manually controlled by a variable potential power supply (Variac)

and a 750 W transformer connected to mains electricity (220 V ac) through a power switch. The input voltage of the variable potential power supply was 220 V. The temperature of W-coil was measured by a ceramic coated Ni–Cr thermocouple. The temperature measurements were done by contacting the ceramic tip of the thermocouple to the center of the coil.

### 2.2. Reagents

All reagents used throughout the study were at least analytical reagent grade. The standard solutions of Sb were prepared by making necessary dilutions from 1000 mg $l^{-1}$ Sb (Aldrich) of stock standard solution. In order to obtain the optimum acidification of reagents, appropriate amount of analytical grade 65% (w/w) HCl was used. Reductant solutions were prepared daily from the powder sodium tetrahydroborate (III) (Merck, min. Purity 98%). For the stabilization of the $NaBH_4$ solution, NaOH (Carlo Erba, Milano, Italy) was used. Dilutions were made using 18 MΩ cm de-ionized water obtained from a Milli-Q water purification system (Millipore, Bedford, MA, USA). Certified reference material waste water EU-L-1 was purchased from SCP Science, St. Laurent, Canada.

### 2.3. Procedure

Optimized parameters were used as explained later in the text. The optimizations were made in several cycles until the result obtained did not differ from the last optimized value. During any optimization, as the value of the parameter in question was varied, all the other parameters were in their optimized values. The experiments were carried out in continuous flow mode for both trap and no-trap studies. For hydride generation without trap, the signal was obtained in the usual manner in CF mode. The generated gaseous antimony hydride was separated from the liquid phase by a laboratory-made cylindrical GLS and was transferred to a quartz T-tube using the Ar gas as the carrier. The continuous signal was recorded. In trap experiments, initially, the carrier gas mixture was sent through the system. Then, the trap was brought to its optimum collection temperature and the pumps were activated to start the flow of carrier and reductant solutions. The transferred stibine was trapped on the W-coil atom trap placed in the inlet arm of the quartz T-tube. At the end of the collection period, the power to heat the trap was discontinued, the pumps were deactivated and hydrogen flow was increased to its optimum value for revolatilization stage. Under the optimum gas flow, the trap temperature was brought to the optimum value for revolatilization and a transient signal was obtained with a base width that is less than 0.5 s.

## 3. Results and discussions

### 3.1. W-coil system

For the antimony determination resistively heated W-coil was used as a trap medium in this study. Each coil can be used for at least 100 firings under optimum parameters. $H_2$ flow in the system directly affects the number of firings with repeatable signals. If the flow rate of the $H_2$ is lower then 38 ml min$^{-1}$ in the collection and revolatilization step, the life-time of W-coil becomes shorter. The presence of $H_2$ gas is necessary for both affecting a more efficient revolatilization and protection of the W-coil against oxidation. The coil should be heated as fast as possible at the revolatilization stage to obtain relatively sharp signals. For this purpose, a power switch was connected in front of the variable potential power supply; the switch was turned off prior to rapid heating, voltage was selected and then the switch was turned on to assure the highest heating rate. Although the chemical nature of the revolatilized analyte species is not known, heating of quartz T-tube atomizer was necessary.

## 3.2. Optimization of system

The optimization of the experimental parameters were carried out using both CF-HGAAS and W-trap HGAAS. In the optimization studies, one parameter was varied while the others were kept constant. Optimization cycles were repeated until stable and repeatable signal was obtained. Optimizations of reaction and stripping coil lengths, concentrations of carrier and reductant solutions were common in both CF-HGAAS and W-trap HGAAS systems. The optimum HCl and NaBH$_4$ concentrations were determined in no-trap studies in continuous flow mode by using 20.0 µg l$^{-1}$ Sb as a standard solution.

### 3.2.1. Optimization of lengths of reaction and stripping coils

Reaction coil consists of the tubing between the merging point of sample and reductant solutions and the point where argon is introduced as the purge gas; as the continuation of the flow system, stripping coil is the tubing between the Ar introduction point and the GLS. Whenever necessary, a hydrogen flow was added to Ar gas stream using a T-junction. In the optimization study, length of reaction coil was varied between 20 and 40 cm while length of stripping coil was varied between 25 and 50 cm. The optimum lengths for reaction coil and stripping coils were found to be 30 cm and 35 cm, respectively. The analyte signal decreased with the lengths both below and above the optimum values for both coils.

### 3.2.2. Optimization of concentration of reductant solution

For the production of antimony hydride species, NaBH$_4$ was used as a reductant solution. The effect of NaBH$_4$ concentration on SbH$_3$ production was investigated by using continuous flow. In order to find the optimum NaBH$_4$ concentration in the reductant solution, concentration was varied between 0.8 and 1.4% (w/v); 1.2% (w/v) NaBH$_4$ was found as the optimum reductant solution using the changes in the analytical signal with respect to reductant concentration for no-trap studies. In this optimization, it was observed that the signal increased up to a concentration of 1.2% (w/v) and decreased for higher values. The decrease can be explained by the dilution effect, since more H$_2$ was generated at higher concentrations of reductant. When the theoretical hydrogen flow rates generated by mixing the HCl and reductant solutions for 1.2 and 1.4% NaBH$_4$ were calculated, the dilution by generated gases corresponded to a value of 7.6% where the observed signal decrease was 8.0%; therefore the match was reasonable.

In addition, NaOH was used for the stabilization of NaBH$_4$. Concentration of NaOH was varied between 0.2 and 0.4% (w/v). Concentration of NaOH was optimized in no-trap studies and then checked also in W-trap HGAAS system. It was found that concentration of NaOH has a significant effect on the analytical signal of antimony. The highest signal for antimony was obtained using the reductant solution containing 0.3% (w/v) of NaOH while the other parameters were kept at their optimum found values.

### 3.2.3. Optimization of concentration of carrier solution

In this study, HCl was used as carrier solution. In order to find the optimum HCl concentration, HCl concentration in the analyte solution was varied between 0.075 and 0.200 mol l$^{-1}$. The optimum value for HCl was found as 0.100 mol l$^{-1}$, somewhat in the middle of the plateau. Higher and lower values resulted in a gradual decrease in the analytical signal. When the concentration of HCl exceeds 0.100 mol l$^{-1}$, the signal decreased. The effect of sample acidity was also checked in W-trap HGAAS system and found to be as same as in the no-trap system.

### 3.2.4. Optimization of flows and composition of gases in the collection and revolatilization stages

In the hydride generation system different carrier gases introduced between the reaction and the stripping coils are used to separate the volatile species from the liquid phase and furthermore transport these species from GLS to externally heated T-tube atomizer. In this study a
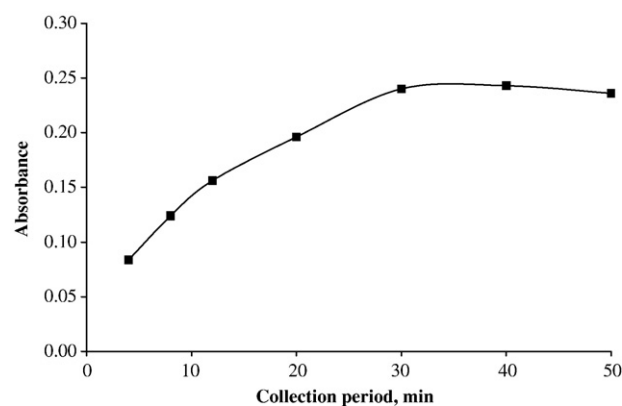


**Fig. 1.** Variation of signal with collection period. Percent standard deviation was 4.00 or lower ($N=3$).

mixture of Ar and H$_2$ was applied in not only the collection but also the revolatilization steps. In general, the flow rate of H$_2$ was kept constant at an optimized value where the oxidation of the W-coil was minimized or eliminated. Here, H$_2$ was used not only to prevent oxidation of the W-coil by creating a reducing environment around the coil but also to improve the revolatilization efficiency of antimony species from the W-coil.

In the collection step, the optimum flow rates of Ar and H$_2$ were found to be 75 and 38 ml min$^{-1}$, respectively, but these flow rates were optimized as 75 and 429 ml min$^{-1}$ for the revolatilization stage. While the flow rate of Ar was the same in collection and revolatilization steps, the optimum H$_2$ flow rate was found to be higher in the revolatilization step. If the flow rate of hydrogen was kept at the same level in both the collection and revolatilization steps, antimony signal decreased to the half of its optimized value. The flow rate of Ar gas was varied between 50 and 400 ml min$^{-1}$ during the optimization. The optimum value was found to be 75 ml min$^{-1}$; signal decreased for lower and higher values.

### 3.2.5. Optimization of flow rates of carrier and reductant solution

The efficiency of the production of antimony hydride species is directly related with flow rates of carrier and reductant solutions. In this study, the flow rate of HCl that was used as the carrier solution was varied between 4.0 and 10.0 ml min$^{-1}$ and the optimum value was found to be 9.0 ml min$^{-1}$. The signal increased as the flow rate was increased until the value of 9.0 ml min$^{-1}$ is reached and stayed constant afterwards. The flow rate of NaBH$_4$ solution was varied between 1.0 and 4.0 ml min$^{-1}$; the optimum result was found as 2.2 ml min$^{-1}$. For the higher and lower flow rates of NaBH$_4$, the peak height of Sb signal sharply decreased.

Optimizations of concentrations and flow rates for HCl and reductant solutions were carried out by keeping all parameters constant while and only the tested parameter was varied.

### 3.2.6. Optimization of collection and revolatilization trap temperatures

In order to obtain a high trapping efficiency W-coil trap temperature should be carefully optimized. In this study the trap temperature for collection was varied between 103 and 539 °C. The trapping efficiency of W-coil increases sharply as the temperature increases up to approximately 370 °C. Increasing the trapping temperature resulted in a decrease of the analytical signal owing to lowered trapping efficiency. Hence, 370 °C was found as the optimum trap temperature for efficient preconcentration of Sb species on W-coil. Revolatilization trap temperature was varied between 625 and 1076 °C while the collection temperature was kept constant at its optimized value. The optimum temperature for revolatilization was 895 °C at which the highest signal was obtained. There is no need for waiting to cool the W-coil atom trap after revolatilization step or heat for the activation of W-coil before each
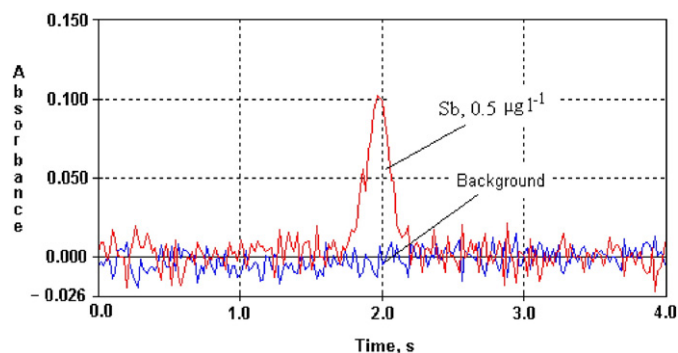
**Fig. 2.** Sb signal obtained using 0.50 μg l$^{-1}$ Sb standard, 9.0 ml min$^{-1}$ of sample flow rate after 4.0 min collection period.

collection cycle because the trap cools down very rapidly in only a few seconds due to the continued gas flow. In order to protect the W-coil, hydrogen flow should be on throughout the experiment.

### 3.2.7. Selection of collection period of Sb in W-trap studies

In the W-trap studies, collection period of Sb was the one of the most critical stages. Using 0.50 μg l$^{-1}$ of Sb during analyte collection, this period was varied between 4.0 and 50.0 min. The change of signal with increasing collection period is shown in Fig. 1. In order to obtain the signals that are used for calibration and analytical figures of merit, a collection period of 4.0 min was selected. The signals became larger with increasing collection period until 30 min after which a plateau was reached. After selecting the optimum values for W-coil trap system, a signal shown in Fig. 2 was obtained for 0.50 μg l$^{-1}$ Sb standard; 36.0 ml of sample were collected in 4.0 min using a sample flow rate of 9.0 ml min$^{-1}$.

Optimized analytical parameters and specifications for the W-coil atom trap HGAAS and no-trap methods are given in Table 1.

### 3.3. Analytical figures of merit

#### 3.3.1. Calibration plots and linearity

Calibration plots with and without using W-coil trap were constructed. The linear portion of calibration without trap included Sb concentrations of 5.0–25 μg l$^{-1}$ using peak height values. In this linear range, linearity equation was found as $y = 0.0093x + 0.0077$ and correlation coefficient ($R^2$) was found as 0.9986; $y$ is absorbance value for CF mode and $x$ is concentration in μg l$^{-1}$. In the W-trap studies,

**Table 1**
Hydride generation, collection and revolatilization conditions for the W-trap HGAAS method for Sb determination

| Parameters | Results |
|---|---|
| Carrier solution | 0.1 mol l$^{-1}$ HCl, 9.0 ml min$^{-1}$ |
| Reductant solution | 1.2% (w/v) NaBH$_4$ (Stabilized in 0.3% (w/v) NaOH), 2.2 ml min$^{-1}$ |
| Length of reaction coil | 30 cm |
| Length of stripping coil | 35 cm |
| Carrier gas for HGAAS | 75 ml min$^{-1}$ Ar |
| Collection carrier gas | 75 ml min$^{-1}$ Ar, 38 ml min$^{-1}$ H$_2$ |
| Revolatilization carrier gas | 75 ml min$^{-1}$ Ar, 429 ml min$^{-1}$ H$_2$ |
| Collection period | 4.0 min |
| Collection trap temperature | 370 °C |
| Revolatilization trap temperature | 895 °C |
| Peristaltic pump | Gilson Minipuls 3 |
| On-line trap for preconcentration | Resistively heated W-coil |
| Heating system for trap | Power switch, Variac (0–220 V ac), transformer (750 W) |
| Atomizer | Flame heated quartz T-tube |

**Table 2**
Analytical figures of merit obtained in trap and no-trap studies

| | With trap | No-trap |
|---|---|---|
| LOD, ng l$^{-1}$ | 16 | 220 |
| LOQ, ng l$^{-1}$ | 53 | 733 |
| $C_o$, ng l$^{-1}$ | 26 | 436 |
| $m_o$, pg | 936 | – |

linear range included Sb concentrations of 0.10–2.0 μg l$^{-1}$; best line equation was found to be $y = 0.1394x + 0.0056$ and correlation coefficient ($R^2$) was found as 0.9975; $y$ is absorbance for peak height measurement and $x$ is concentration in μg l$^{-1}$.

#### 3.3.2. Precision

RSD values for W-trap were equal to or better than 3% and 9% for peak height and area measurements, respectively, using 0.5 μg l$^{-1}$ Sb in the collection mode. As shown in Fig. 2, half width of signal was about 0.2 s; the peaks were extremely sharp to allow the use of peak height.

#### 3.3.3. Accuracy

Certified reference material waste water EU-L-1was analyzed to find the accuracy of the improved method for Sb. The results were in good agreement with the certified values. Found value was 56.3 ± 6.2 μg l$^{-1}$ while certified value is given as a range of 50–70 μg l$^{-1}$. Direct calibration method was used; standard addition technique was not necessary.

#### 3.3.4. LOD, LOQ, $C_o$ and $m_o$

Three and 10 times the standard deviation of blank values were used in the LOD and LOQ calculations, respectively. In W-coil trap studies, 4.0 min collection period was applied for the LOD and LOQ calculations. In calculations of characteristic concentration, $C_o$, for W-trap and no-trap studies, 0.5 μg l$^{-1}$ of Sb and 10.0 μg l$^{-1}$ Sb were used, respectively. Characteristic mass $m_o$ for W-trap system was calculated using 0.5 μg l$^{-1}$ of Sb in W-trap system, collecting 36 ml in 4.0 min. Peak height values were used in all calculations. Analytical figures of merit mentioned above are shown in Table 2. Using $C_o$ values, an enhancement factor of 17 was found for the present conditions. According to the data in Fig. 1, enhancement can be better until a collection period of 30 min is reached, although the improvement in enhancement will not be in a linear fashion with respect to collection period.

When the overall performance is compared with the trap system for bismuth [19], it seems that the enhancement in sensitivity obtained for Sb is lower. This difference is partly due to the differences in conventional hydride systems for two elements. In terms of characteristic concentrations, sensitivities are 290 and 436 ng l$^{-1}$ for Bi and Sb, respectively; this causes an advantage for Bi by a factor of 1.50. However, the difference in enhancement is larger than this figure, indicating that trap efficiencies for two elements are different. We preferred not to carry out any experiments regarding efficiencies both in cases of Bi and Sb since final gas flows and compositions in both cases are different for conventional hydride and trap systems; therefore the signals cannot be directly compared to calculate efficiencies.

## 4. Conclusion

A sensitive, accurate and reliable method was developed for Sb determination in trace levels using W-trap HGAAS. A simple flame AAS instrument, a peristaltic pump and a simple flow system are all needed to obtain sensitivity at high ng l$^{-1}$ level. All experimental parameters that influence the collection/volatilization efficiency were optimized in order to have the best sensitivity in W-coil trap HGAAS system. The importance of excess H$_2$ flow rate in revolatilization stage should be

highlighted. For the parameters chosen, enhancement in sensitivity was found to be 17; this figure can be better for longer collection times as depicted in Fig. 1.

## Acknowledgments

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