



Ultratrace determination of tin by hydride generation in-atomizer trapping atomic absorption spectrometry



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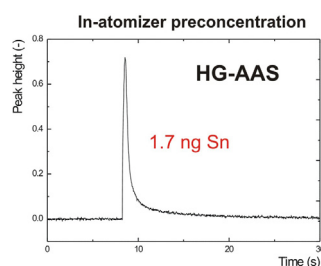
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HIGHLIGHTS

- In-atomizer trapping HG-AAS was optimized for Sn.
- A compact quartz trap-and-atomizer device was employed.
- Generation, preconcentration and atomization steps were investigated in detail.
- Hundred percent preconcentration efficiency for tin was reached.
- Routine analytical method was developed for Sn determination (LOD of 0.03 ng mL⁻¹ Sn).

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 8 July 2013

Received in revised form 1 October 2013

Accepted 10 October 2013

Available online 19 October 2013

Keywords:

Hydride generation

Tin preconcentration

In-atomizer trapping

Trap-and-atomizer device

Stannane

ABSTRACT

A quartz multiatomizer with its inlet arm modified to serve as a trap (trap-and-atomizer device) was employed to trap tin hydride and subsequently to volatilize collected analyte species with atomic absorption spectrometric detection. Generation, atomization and preconcentration conditions were optimized and analytical figures of merit of both on-line atomization as well as preconcentration modes were quantified. Preconcentration efficiency of $95 \pm 5\%$ was found. The detection limits reached were 0.029 and 0.14 ng mL⁻¹ Sn, respectively, for 120 s preconcentration period and on-line atomization mode without any preconcentration. The interference extent of other hydride forming elements (As, Se, Sb and Bi) on tin determination was found negligible in both modes of operation. The applicability of the developed preconcentration method was verified by Sn determination in a certified reference material as well as by analysis of real samples.

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1. Introduction

Natural processes as well as antropogenic activities are both the sources of metals and their compounds to be evolved into the environment [1]. Since exposure to heavy metals is potentially harmful their monitoring in environmental, biological and clinical samples even at trace levels is nowadays required by law and other regulations in many cases. As a consequence development of sensitive analytical methods for metal determination/speciation at

ultratrace levels becomes a challenging task. Tin is possibly an essential element for animals, but no specific role for tin in human health has been identified. Toxicity of tin compounds strongly depends on their nature with organotins belonging to the most toxic pollutants in aquatic ecosystems [1]. Alkyltin salts have been widely used in material science and agriculture as antifouling agents and fungicides, e.g. the use of organotins as antifouling paints on large ships caused global contamination of harbor sediments. Four different types of target organ toxicity have been described for organotin compounds—neuro-, hepato-, immuno- and cutaneous toxicity [1].

Spectrometric analytical methods are widely used as detectors for trace and ultratrace determination of metals. Hydride

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generation atomic absorption spectrometry (HG-AAS) [2,3] is due to its simplicity, selectivity and sensitivity a powerful and favorite analytical technique for determination of many clinically/toxicologically important analytes such as As, Se, Sb, Bi, Pb and also Sn. Analyte conversion to the gaseous hydride has two advantages—firstly, analyte is separated from the matrix and secondly, analyte can be preconcentrated from the gaseous phase in order to decrease the limit of detection (LOD). Possibility of efficient analyte preconcentration by in-atomizer hydride trapping combined with inexpensive atomic absorption detector gives thus an alternative to conventional approaches to elemental and speciation analysis based on the liquid sampling ICP-MS which is, despite its high investment and operation costs, the most frequent and straightforward technique in the field of ultratrace element analysis. Inherent disadvantage of HG-AAS is that the additional step, hydride generation performed in hydride generators, has to be included to the measurement scheme.

Although in-situ trapping of hydrides in a graphite furnace (GF) [4,5] is the most common approach to hydride trapping with AAS detection, also quartz and metal preconcentration devices [3,5,6] have been designed and successfully employed for ultratrace determination of hydride forming elements with detection by AAS.

Among them, a compact quartz trap-and-atomizer device [7,8] has been shown a powerful tool for determination of hydride forming elements at ultratrace levels by HG-AAS. This device is actually the multiple microflame quartz tube atomizer (multi-atomizer) [9,10] with its inlet arm modified to serve as the trap. Whereas oxygen rich atmosphere is used to reach efficient analyte trapping in the first step of the preconcentration procedure, hydrogen containing atmosphere is used to rapidly volatilize the trapped species in the second stage. Routine analytical methods were developed based on this universal device. As a consequence, procedures for ultratrace determination of As, Sb, Bi, Se and Pb were reported [7,8,11]. Complete (100%) preconcentration efficiency was found for in-atomizer collection of stibine [8], bismuthine [8] and plumbane [11]. High preconcentration yields, 50% and 70%, were reached for arsine and selenium hydride preconcentration [7], respectively, in the same trap-and-atomizer device. These facts indicate the potential of this device to be employed also for preconcentration of other hydride forming elements.

The aim of this work was to find optimum conditions for stannane preconcentration and to develop and validate a routine analytical procedure for ultratrace determination of tin by in-atomizer preconcentration HG-AAS. It should be noted that the developed preconcentration method can be used for the determination of both inorganic and organic Sn species, provided suitable treatment (conversion to hydride active species by (photo)chemical reaction) precedes the HG step for organotin compounds.

2. Experimental

2.1. Reagents

All reagents were of analytical reagent grade or higher purity. Deionized water ($<0.1 \mu\text{S cm}^{-1}$, Ultrapure, Watrex, USA) was used to prepare solutions. Working Sn standards were prepared fresh daily from 1 mg mL^{-1} Sn stock solution (BDH Laboratory Reagents Ltd., England) by dilution in 0.1 mol L^{-1} HCl (Merck, Germany). The blank was 0.1 mol L^{-1} HCl. The reductant was 1.5% (m/v) solution of NaBH_4 (Sigma Aldrich, Germany) in 0.4% (m/v) KOH (Merck, Germany) filtered after preparation and stored frozen.

For interference studies, the As(III), Sb(III), Bi(III), and Se(IV) 1 mg mL^{-1} stock solutions (BDH Laboratory Reagents Ltd., England) were used to prepare the working solutions in 0.1 mol L^{-1} HCl and

containing always 1 ng mL^{-1} Sn as analyte and between 1.00 to 1000 ng mL^{-1} of the interferent. Hydrofluoric acid (38% (m/v), p.a., Spolchemie, Ústí nad Labem, Czech Republic) and nitric acid (65%, p.a., Lach-Ner, Czech Republic) were used to clean the trap-and-atomizer device.

A certified reference material TMRain-04 (simulated rain sample for trace elements produced by Environment Canada, Canada) with defined Sn content was employed to verify accuracy of the developed analytical method. A sample of mineral water was bought in a local supermarket.

Compressed gases Ar (99.996%), H_2 (99.95%) and O_2 (99.5%) were produced by SIAD Czech, Ltd., Czech Republic.

2.2. Instrumentation

A GBC model SavantAA atomic absorption spectrometer (GBC Scientific Instrument, Australia) was employed without background correction. A Photron Sn superlamp (Photron Ltd., Australia) operated at 286.3 nm analytical line with 0.5 nm spectral bandpass and a lamp current of 15 mA (boost current 10 mA).

2.3. Hydride generator and atomizers

An in-house made, continuous flow hydride generation system similar as described in refs. [7,8,11] with a 3 mL inner volume gas-liquid separator (GLS) with a forced outlet was used (see Ref. [12] for detailed GLS description). Gas flows of Ar, H_2 and O_2 were controlled by mass flow controllers (Omega Engineering, USA). See Table 1 for summary of liquid and gas flow rates employed.

The commercial Perkin Elmer externally heated quartz tube atomizer (Quartz Cell 2 for FI-MHS) without end windows, termed further as conventional QTA, was used when explicitly stated. It was actually a plain T-tube with the horizontal arm (aligned in the optical path of the spectrometer) length of 160 mm, i.d. of 7 mm and o.d. of 14 mm. The commercial heating unit with temperature control produced by RMI (Lázně Bohdaneč, Czech Republic) was used to heat the horizontal arm to 950°C . The length and i.d. of the inlet arm was 90 and 1 mm, respectively. A deactivated fused silica capillary (Supelco, Germany, 0.53 mm i.d.) was centered within the inlet arm of the QTA with the downstream end of the capillary aligned in the T-junction of the inlet and optical arm. The capillary served to introduce the flow of oxygen into the optical arm of QTA. Gases from the gas liquid separator (analyte hydride, H_2 as a product of reductant decomposition and carrier Ar) were introduced through the inlet arm of the QTA.

Otherwise the trap-and-atomizer device was employed. See Ref. [7] for a detailed description. The horizontal arm of the device was made of two concentric tubes: the inner (optical) one was evenly perforated with 14 holes. The same heating unit as specified above was employed to heat the horizontal arm to 950°C . A flow of gas (outer gas) can be introduced from the sides into the cavity between both tubes of the horizontal arm and then passed through the holes into the optical tube. A continuously flowing outer gas, consisting of air, was used for the duration of each experiment. If explicitly stated hydrogen was employed as outer gas instead of air. The length of the inlet arm of the trap-and-atomizer device was 100 mm with the i.d. of 2 mm and o.d. of 4 mm.

The inlet arm of the trap-and-atomizer device, resistively heated independently of the horizontal arm by a laboratory power supply source PS 3065-10 B (E-A, Elektro-Automatik GmbH, Viersen, Germany), served as the trap. Resistive heating was realized by a 40 cm long canthal wire coil ($4.17 \Omega \text{ m}^{-1}$, 0.65 mm in diameter, length of the coil 6 cm). A deactivated fused silica capillary (Supelco, Germany, 0.53 mm i.d.) was centered within the inlet arm.

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