

Determination of tellurium by hydride generation with *in situ* trapping flame atomic absorption spectrometry[☆]

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Abstract

The analytical performance of coupled hydride generation — integrated atom trap (HG-IAT) atomizer flame atomic absorption spectrometry (FAAS) system was evaluated for determination of Te in reference material (GBW 07302 Stream Sediment), coal fly ash and garlic. Tellurium, using formation of H₂Te vapors, is atomized in air–acetylene flame-heated IAT. A new design HG-IAT-FAAS hyphenated technique that would exceed the operational capabilities of existing arrangements (a water-cooled single silica tube, double-slotted quartz tube or an “integrated trap”) was investigated. An improvement in detection limit was achieved compared with using either of the above atom trapping techniques separately. The concentration detection limit, defined as 3 times the blank standard deviation (3σ), was 0.9 ng mL⁻¹ for Te. For a 2 min *in situ* pre-concentration time (sample volume of 2 mL), sensitivity enhancement compared to flame AAS, was 222 fold, using the hydride generation — atom trapping technique. The sensitivity can be further improved by increasing the collection time. The precision, expressed as RSD, was 7.0% (*n*=6) for Te. The designs studied include slotted tube, single silica tube and integrated atom trap-cooled atom traps. The accuracy of the method was verified using a certified reference material (GBW 07302 Stream Sediment) by aqueous standard calibration curves. The measured Te contents of the reference material was in agreement with the information value. The method was successfully applied to the determination of tellurium in coal fly ash and garlic.

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1. Introduction

Tellurium is regarded as rare, non-essential element, usually occurring at low concentration levels, and it is known to be toxic to humans (Te affect various organs) [1]. It has found many applications in technological processes resulting in local enrichment and release [2]. Tellurium must therefore be determined in a wide range of matrices, e.g., biological, clinical, geological, semiconductor, and/or metallurgical samples. The determination of Te is a difficult task because of the very low

concentration and/or amount of the element. Therefore, in most cases a pre-concentration stage is essential before the element can be determined.

A very important step in many modern analytical methods is the sample introduction into the instrument. In order to avoid several problems of the determination procedure, the formation of volatile phases from sample solution is a widely used technique for separating the analyte from the sample matrix. Thus, some possible interferences are eliminated and the analyte is concentrated, and the sensitivity is increased. The volatile phase must be formed by fast quantitative reaction and be stable enough for transport to the instrument. Several volatile compounds are used in analytical chemistry: organometallic compounds, chelate compounds, inorganic salts and covalent hydrides.

It is well established that significant improvements in the limits of detection of flame atomic absorption spectrometry (FAAS) and graphite furnace atomic absorption spectrometry (GF-AAS) may be achieved by chemical vapor generation, mostly hydride

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generation (HG) and in-atomizer (silica or graphite tube) trapping (pre-concentration) as reviewed [3,4] for the determination of As, Bi, Ge, In, Pb, Sb, Se, Sn, Te and Tl (as well as Hg).

Hydride generation combined with AAS detection (HG-AAS) is one of the most powerful analytical methods for the determination of Te. Initially, AAS using different atomization devices was the most employed technique [5–13]. Kobayashi and Imaizumi [5] described the generation of Te(IV) with atomization in a flame AAS. Zhang et al. [6] developed a method for the determination of Te by HG-AAS using electrically heated quartz tube furnace under sub-atmospheric pressure. Hall et al. [7] studied the performance of hydride generation quartz tube AAS in the determination of Te in geological reference materials. Tamari [8] described the application of HG-FAAS with coprecipitation method for the determination of Te(IV). Körez et al. [9] suggested a separation pre-concentration method using a mercapto-modified silica microcolumn for the determination of trace amount of Te (IV) by HG-AAS using a flame-heated quartz tube. Ha et al. [10] developed a method for the determination of Te in urine by HG-AAS with derivative signal processing. A method based on flow-injection HG-AAS for the determination of Te in nickel-based alloys and pure copper [11] and in lead and lead alloy [12] was described. Kaplana et al. [13] described a procedure for the determination of total Te in garlic that combines HG-AAS with pre-concentration of the analyte by coprecipitation.

Reports on the hydride trapping of Te and atomization in a graphite furnace are few [14–17]. In 1984, Andreae [14] first described the hydride generation and *in situ* trapping (in the graphite tube) system for the GF-AAS determination of Te(IV). Doidge et al. [15] improved the determination of Te with the use of hydride generation and *in situ* trapping (in graphite tube) using palladium as the enhancement element. Yoon et al. [16] improved the sensitivity for determining Te, using a hydride generation and *in situ* trapping technique for GF-AAS. Ni et al. [17] described the application of GF-AAS with *in situ* pre-concentration in a graphite furnace coated with silver for the determination of tellurium.

In situ trapping permits a significant enhancement in sensitivity for batch and continuous hydride generation approaches used in the ultra-trace determination of volatile hydride species. Due to its importance, *in situ* trapping, which allows the coupling of hydride generation to integrated atom trap (IAT) system [18–20], was chosen for this study. Korkmaz et al. [21] investigated the nature of re-volatilization from atom trap surfaces in flame by AAS. Analytes Au, Bi, Cd, Mn and Pb were trapped on a water-cooled, U-shaped silica trap or a slotted silica tube trap and re-volatilized by organic solvent aspiration. They concluded that, although heating was not necessarily associated with re-volatilization, direct contact between the flame and the active silica surface was required. Recently [22], the analytical performance of three trap systems (water-cooled U-shaped silica trap, water-cooled U-shaped silica trap combined with slotted silica tube and slotted tube trap) for flame AAS were evaluated for determination of Cd and Pb in waters. Guo and Guo [23] reported SeH₂ collection at gold wire heated to 200 °C situated in a quartz tube atomizer (atomic fluorescence spectrometry (AFS) or AAS detection) with separate inlet for argon. A successful trapping of PbH₄ in a bare

Table 1

Optimized operating conditions for determination of tellurium by IAT-FAAS with continuous-flow hydride generation

Parameter	Tellurium
FAAS system	
Wavelength (nm)	214.3
Spectralband pass (nm)	0.3
Lamp current (mA)	10
Flame type ^a	Air-C ₂ H ₂
Flame conditions ^b	Lean
Silica tube obscuration (%)	ca.25
Coolant water (L min ⁻¹)	ca. 2
Window of measurements (s)	15
Read time (s)	7
Signal measurement	Peak area absorbance
HG-IAT system	
NaBH ₄ concentration ^c [% (m/v)]	2.0
NaBH ₄ solution flow rate (mL min ⁻¹)	1.0
HCl concentration (mol L ⁻¹)	3.0
Thiourea [% (m/v)]	1.5
Sample/HCl flow rate (mL min ⁻¹)	1.0
Carrier air flow rate (mL min ⁻¹)	100
PVC/PTFE peristaltic tubes	40 cm × 1.0 mm i.d. × 2.5 mm o.d.
Trapping time (s)	120
Trapping temperature (°C)	<100
Atomization temperature ^d (°C)	ca. 1330

^a Nebulizer uptake rate, 5 mL min⁻¹.

^b Air flow rate 475 L h⁻¹, acetylene flow rate 50 L h⁻¹ (fuel-lean flame); 10 cm slot burner.

^c In 0.5% (m/v) NaOH solution.

^d Ref. [30].

quartz tube was announced by Korkmaz et al. [24], who also suggested that the same trap could be used also for other hydrides. Recently, a preliminary evaluation of quartz tube trap for collection of SbH₃ and for volatilization of trapped analyte with subsequent atomization in a multiple microflame quartz tube atomizer for AAS was presented [25]. Kratzer and Dĕdina extended their investigation of stibine trapping in quartz tube traps [25] to stibine collection (and subsequent analyte atomization) in conventional quartz tube atomizers [26]. They employed the simplest possible experimental arrangement; just the commercially available externally heated quartz tube atomizer without any trap or additional heating device. A modification of the externally heated quartz tube atomizer, making possible *in situ* trapping of bismuthine and subsequent analyte atomization for Bi was described [27].

This work has been mainly aimed at improving analytical performance of tellurium conventional hydride generation flow system by combining this approach for *in situ* trapping technique with an integrated atom trap system for flame AAS that is applicable to determination of Te in reference material and in real analytical samples. The hydride generation technique brings the FAAS method closer to the detection limits of HG-GF-AAS (*in situ* trapping technique) [14,16,17].

2. Experimental

2.1. Apparatus

A Carl Zeiss Jena (Jena, Germany) Model AAS3 flame atomic absorption spectrometer (components described in Ref.

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