

Simultaneous determination of hydride forming (As, Bi, Ge, Sb, Se, Sn) and Hg and non-hydride forming (Ca, Fe, Mg, Mn, Zn) elements in sonicate slurries of analytical samples by microwave induced plasma optical emission spectrometry with dual-mode sample introduction system

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Abstract

A slurry sampling method for the simultaneous determination of hydride forming (As, Bi, Ge, Sb, Se, Sn) and Hg and non-hydride forming (Ca, Fe, Mg, Mn, Zn) elements, without total sample digestion has been developed using the commercial dual-mode sample introduction system (MSIS) coupled with microwave induced plasma optical emission spectrometry (MIP-OES) from biological and environmental reference materials and real samples. The main advantage of this system is its simultaneous determination of elements that form volatile vapor species and elements that do not, without any instrumental changes. Optimization of reaction, nebulization and instrumental conditions was performed to characterize the new system. Slurry concentration up to 4% m/v (particles < 100 μm) prepared in 10% HNO₃ containing 100 μL of decanol, by application of ultrasonic agitation, was used with calibration by the standard addition technique. An ultrasonic probe was used to homogenize the slurry in the quartz cup just before its introduction into the reaction/nebulization system; the multimode sample introduction system (MSIS) combines the benefits of nebulization and vapor generation in a single device. Detection limits (LOD, 3σ_{blank}, peak area) of 0.07, 0.29, 0.25, 0.10, 0.12, 0.14, 0.11, 0.28, 0.42, 0.02, 0.21 and 0.34 μg g⁻¹ were obtained for As, Bi, Ge, Sb, Se, Sn, Hg, Ca, Fe, Mg, Mn and Zn, respectively. The relative standard deviations were *ca.* 10%, adequate for slurry analysis. To test the accuracy, six certified reference materials were analyzed with the analyte concentrations mostly in the μg g⁻¹ level. Measured concentrations are in satisfactory agreement with certified values for the biological reference materials (LUTS-1, DOLT-2) and environmental reference materials (PACS-1, GWB 07302, NIST 2710, NBS 1633b), all adequate for slurry sampling. The method was successfully applied to the determination of the elements in real samples (coal fly ash, lake sediment, sewage). The method requires small amounts of reagents and reduces contamination and losses.

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1. Introduction

The chemical vapor generation (CVG) of hydrides and mercury is currently a very popular sample introduction technique used in analytical atomic spectrometry [1–6]. However, as a rule, CVG atomic spectrometric techniques require complete digestion of the samples prior to analysis. The digestion step is very critical, which increases both analysis time and the risk of sample contamination and/or losses of the analyte. A

simple alternative to circumvent the digestion of samples is the slurry sampling (nebulization), which combines the advantages of the solid and liquid sampling (presentation) [7].

The method of hydride generation (HG) directly from slurried samples has recently been reviewed by Matusiewicz [8], in which important aspects of the slurry sampling (SS) in relation to analytical atomic spectrometry was presented. The ability of the microwave induced plasma optical emission spectrometry (MIP-OES) technique to determine several elements simultaneously may be advantageously used in this context and seems to be an effective approach. Slurry nebulization has also proved to be useful for sample introduction into the MIP [9,10]. It

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should be noted here, that only very recently have Matusiewicz and Ślachciński [11] proposed a method for the determination of As, Sb, Se, Sn and Hg in analytical reference materials by HG-MIP-OES using slurry sampling. On the other hand, Santos *et al.* [12] proposed a method for the determination of As, Hg, Sb, Se and Sn in sediments by on-line chemical vapor generation inductively coupled plasma optical emission spectrometry (ICP-OES) using slurry sampling. In addition, Ribeiro *et al.* [13] proposed a method for the determination of As, Hg, Sb, Se and Sn in environmental and geological reference materials, as acidified slurries, by flow injection coupled to a hydride generation system and determination by inductively coupled plasma mass spectrometry (ICP-MS). The minor disadvantage of these systems is the need for separate MIP-OES (or ICP-OES) analysis to determine the other elemental constituents of the sample. Normally, introduction of analytes/samples by CVG and pneumatic nebulization (NEB) are performed separately by using different sample introduction systems. A survey of the literature [14–25] indicates that a few papers describe analytical methods suitable for determination of hydride and non-hydride forming elements simultaneously from the solution, but, as far as we are aware, has not been applied for analysis of slurried samples, using the dual sample introduction systems. Wolnik *et al.* [14] described a tandem nebulization system in which a concentric Meinhard nebulizer is mounted in a Teflon spray chamber, joined to a cross-flow nebulizer and fitted to a spray chamber connected to the ICP torch. Hutton and Preston [15] described a simple modification to the nebulizer assembly that allows either sensitive hydride or conventional solution analysis to be performed with no downtime. Huang *et al.* [16,17] developed a nebulizer hydride generator systems in which large droplets of the acid sample aerosol from the pneumatic nebulizers were trapped by the impact wall of a hydride generators, and reacted with reductant. Fengzhou *et al.* [18] developed and integrated a nebulizer-hydride generator system in which the spray chamber for pneumatic nebulization was modified for hydride generation. Pohl and Żyrnicki [19] employed a Jobin Yvon hydride generation system (CMA) without a gas–liquid phase separator for the determination of As, Bi, Se, Sn and non-hydride forming elements by the ICP-OES technique. McLaughlin and Brindle [20] reported a multi-mode sample introduction system (MSIS) in which hydride formation takes place at the outlet of the two capillaries, located in a modified cyclonic spray chamber, that transport the sample and sodium borohydride solutions. The MSIS system provides convenient means of determining elements, either by nebulization or vapor generation, or simultaneously in the same device. Rojas *et al.* [21] used a modified V-groove with one additional channel to introduce the hydride solution. Asfaw and Wibetoe [22,23] applied a commercial MSIS for the determination of As, Bi, Sb, Se, introduced as the hydride, simultaneously with the other non-hydride forming elements (Ca, Cd, Co, Cu, Mg, Ni, K, P, Pb, S, Zn). Zoltan *et al.* [24] studied a modified cross-flow-type nebulizer coupled with a conventional Scott double-pass spray chamber. In this case, they adapted two new channels into the nebulizer in order to introduce the hydride and acid solutions separately. Finally, Gómez *et al.* [25] explored a dual

nebulizer sample introduction system for simultaneous determination of volatile element hydrides and other elements. The system consists of two pneumatic nebulizers connected to the conventional spray chamber of the instrument *via* a simple adaptor. This configuration permits hydride generation but still allows other elements to be determined by pneumatic nebulization. To our knowledge, no paper(s) have been published for the simultaneous determination of hydride and non-hydride forming elements, using slurry sampling chemical vapor generation/pneumatic nebulization microwave induced plasma optical emission spectrometry with dual-mode sample introduction system.

In this paper we will describe, for the first time, the development of a simultaneous multi-element SS-CVG/NEB-MIP-OES method (by using a continuous mode system) for the quantitative determination of As, Bi, Ge, Sb, Se, Sn, Hg and Ca, Fe, Mg, Mn, Zn in biological and environmental certified reference materials and real samples prepared as slurries, using a dual-mode sample introduction system. It also describes the MIP-OES instrumental operating parameters, as well as the simultaneous CVG and NEB experimental parameters and an evaluation of the main analytical figures of merit, and presents details of the analytical procedure including preparation of slurry samples for the determination of volatile hydrides and elemental mercury as well as other elements. Finally, accuracy was checked by the analysis of six certified reference materials (LUTS-1, DOLT-2, PACS-1, GWB 07302, NIST 2710, NBS 1633b).

2. Experimental

2.1. Instrumentation

The measurements were performed with a simultaneous MIP-OES spectrometer equipped with a microwave plasma excitation source, which includes a TE₁₀₁ integrated microwave resonator with a microwave power supply. The majority of the OES system and microwave plasma cavity assembly used for simultaneous multi-element analysis was essentially the same as described previously; [11] this will not be discussed again here.

Table 1
Instrumental parameters for He-MIP-OES

Nominal microwave frequency/MHz	2450
Microwave cavity	TE ₁₀₁ rectangular
Microwave power/W	260
Plasma torch	Quartz tube, 2.5 mm i.d.
Plasma viewing mode	Axial
Plasma supporting gas flow rate/ml min ⁻¹	350
Integration time/s	0.1
Determination	Simultaneous
Working wavelengths/nm	As 228.811; Bi 223.061 Ge 303.907; Sb 217.582 Se 196.026; Sn 242.949 Hg 253.652 Ca 393.336; Fe 238.204 Mg 285.213; Mn 257.611 Zn 213.857

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