

Short communication

Direct atomic absorption spectrometry determination of tin, lead, cadmium and zinc in high-purity graphite with flame furnace atomizer[☆]

A. Zacharia^{a,*}, S. Gucer^b, B. Izgi^b, A. Chebotarev^a, H. Karaaslan^b

^a *I.I.Mechnikov Odessa National University, Department of Analytical Chemistry, str.Marazlievskaya, 1A/8, 65014 Odessa, Ukraine*

^b *Uludag University, Science and Art Faculty, Department of Chemistry, 16059 Bursa, Turkey*

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Abstract

This work described methodology of Sn, Pb, Cd and Zn impurities determination in high-purity graphite at direct atomic absorption spectrometry (AAS) with flame furnace (FF) atomizer. It was evidence that quality of AAS measurements are depended from sample amount, its homogeneity, particle size, as well as calibration procedure and operation parameters of FF atomizer. Prior to analysis the method has been developed and optimized with respect to the furnace heating temperature and flame composition of FF atomizer. Conditions of absorption peak areas (Q_A) formation to each element were studied on the basis of contribution into its value some of individual parameters of analytes, including mass-transporting process from increasing mass of graphite samples into gas phase. Because particle size and homogeneous distribution of analyte in powdered materials has an enormous influence on accuracy and precision of measurement results, graphite as well as appropriate series of powdered reference standards was previously ground and investigated. Graphite samples to be analyzed and standard reference materials with mass from 0.025 to 0.200 g was previously briquetted as pellet and insert on corresponding hole in furnace. The characteristic mass (g_0) of Sn, Pb, Cd and Zn were 0.35, 0.1, 0.008 and 0.025 ng, respectively, and relative standard deviation (S_r) not more than 20%.

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1. Introduction

Graphite, carbon and some of their compounds are one of the important materials for modern technology. Their application in various fields of high-technology and industry required powerful, rapid and reliable methods for determination of trace components. The direct analysis of solid and powdered materials including graphite and corresponding materials (various

carbides, natural coals, etc.) by so-called solid sampling technique (SoS) atomic absorption spectrometry is now one of the most promising analytical methods. The advantages of the SoS technique have been reviewed and compared to conventional procedures with regard to aspects of risk of contamination, minimization of losses in the pre-treatment operation steps and incomplete atomization of elements to be determined from solid matrix [1–4]. But the main drawbacks of SoS can be given as follows: (i) unrepresentative mass (<10 mg) of solids to be analyzed, (ii) non-homogenous distribution of analytes in the whole sample, (iii) high background level and (iv) calibration problems. Slurry sampling (SIS) was considered by some to have certain advantages over SoS that are summarized in review [4], where has been noted that slurry analysis of solids samples does not have advantages over direct solid sampling and the that most probable critical factor is the need for maintaining the stability of the slurry until sample injection. Only few authors report about direct SoS AAS for graphite, coal and similar materials analysis [5–12]. It has been noted [9] that determination of Cd in coal

Abbreviations: FF, flame furnace atomizer; Q_A , peak area; g_0 , characteristic mass; AAS, atomic absorption spectrometry; ESA, emission spectral analysis

[☆] The aim of this work therefore was to investigate analytical possibility of FF AAS technique for direct determination of Sn, Pb, Cd and Zn impurities in increasing mass (0.025–0.200 g) of high-purity graphite. The investigation is taking into account questions of sample amount, its homogeneity, particle size, as well as calibration procedure.

* Corresponding author. Tel.: +380 8048 7253976.

E-mail addresses: anz@real.ua (A. Zacharia), sgucer@uludag.edu.tr (S. Gucer).

using SoS graphite furnace AAS with conventional equipment turned out to be difficult, particularly as calibration against aqueous standards was not possible, necessitating the use of certified reference materials (CRM) for that purpose.

So, to find the simplest and most reliable calibration technique for coal analysis by SIS-ET-AAS the authors [10] proposed to use as chemical modifier Ru or Pd/Mg to Cd and Pb determination. In this work they noted that when coal samples were analyzed the excessively high background absorption at low pyrolysis temperature only allowed reliable measurements Pb analyte signal above 650 °C, where the background signal was low. When Pb is determined in solid coal samples without a modifier, losses start to become significant at pyrolysis temperature >750 °C. In this procedure aqueous and solid coal standards were used and in the case of Cd and Pb determination solid standards were chosen.

In [11], at the SoS AAS determination of 14 common impurities, including Pb and Zn, in graphite and silicon carbide, the authors used platform-technique and a sample portion between 0.1 and 8.0 mg. No-significant gas-phase matrix interferences, background signals for any elements or memory effects were observed. When silicon carbide was analyzed by heating about 5 mg of this material at 2400, 2500, 2600 and 2700 °C for 20 s and weighing the residue, mass losses of 13, 26, 60 and 68%, respectively, were found. When applying a temperature of 2600 °C the matrix remains quantitatively on the platform were physically and chemically unchanged. For elements atomized at temperature lower than 2400 °C no significant background signals were observed. Authors of this work point out that with graphite analysis small differences between the transient behaviors in atomization from the graphite sample and compared to aqueous solutions were not significant and no background signals or memory effects were observed. For calibration 10 μ l aliquots of aqueous standard solution were used.

Baxter and Frech [11] examined different SIS AAS calibration techniques with respect to accuracy and found that a necessary condition for the calibration is that the matrix be identical as far as possible in both the solid samples and the calibration standard.

To improve SIS AAS analysis construction of several corresponding laboratory manufacturing devices were proposed and successfully used at the analytical practice [13–15] and only one type of special commercial instrument for solid—SM 30 Zeeman GF AAS (Grün Analysengeräte, Wetzlar, FRG). As was noted [10], one of the major reasons why direct SoS has been abandoned in favor of SIS was the availability of commercial accessories for the latter technique, whereas the former technique was not supported by any major instrument manufacturer.

Several authors [16–19] pointed out some of advantages of the flame furnace (FF) atomizer over conventional atomizers such as: (i) simplicity of construction and exploitation; (ii) satisfactory sensitivity determination of high and medium volatility elements; (iii) not so significant differences in comparison with commercial variants of HGA-type atomizers exposed to different sorts of interferences; (iv) possibility of AAS analysis of a representative mass of solid and powdered materials; (v) simplicity of reproduction (without losses) when inserting solid or

powdered samples into the furnace and removing them from the furnace (without remains).

Unfortunately, till now FF atomizer is not widely used in direct AAS analysis because it is not constructed commercially.

The aim of this work was to investigate analytical possibility and peculiarity of the FF AAS technique for direct solid sampling technique at determination of Sn, Pb, Cd and Zn impurities (≥ 0.005 ppm) in representative mass (≥ 0.100 g) of high-purity graphite.

The investigation takes into account questions of sample mass, its homogeneity, particle size, and the character of calibration procedure.

2. Experimental

2.1. Instruments, FF AAS system and sample preparation

An ATI-Unicam 929 Model AAS equipped with deuterium background correction was used in the absorption mode with the parameters of air-acetylene flame and graphite furnace mentioned below. Hollow cathode lamps were used as radiation sources for Sn, Pb, Cd and Zn determinations. The general scheme of the FF atomizer was adapted from L'vov design [16].

Graphite furnaces were prepared from high-purity carbon rod and before analysis was heated up to 600 °C for 1.0–1.5 h, impregnated for 3 h by 30% (v/v) zirconium oxychloride solution and then dried and purified by electrically heating at 2500 °C for 20–25 s in an argon atmosphere with the FF atomizer.

2.2. Analytical procedure

Graphite samples to be analyzed as well as standard reference materials with mass from 0.025 to 0.200 g were previously briquetted as pellet (diameter 6.0 mm) and inserted into the corresponding hole in graphite furnace. This technique allows with very simple tools the sampling of solid and powdered materials without losses and removing their remains from furnace after the operation cycle. Emission spectral analysis (ESA) was additionally used at pre-investigations at condition that have been detailed before [19].

All graphite samples were previously ground and powdered for 60–90 min with agate mortar and particle size distribution was obtained by photo sedimentation analysis.

The appropriate aqueous calibration standards were prepared by dilutions of stock solution in 0.01 mol l⁻¹ nitric acid and series of calibration graphite standards samples – by mechanical dilutions (at 5–10 times) from sample to sample with high-purity graphite its general certificate reference material (CRM) with content of Sn, Pb, Cd, Zn – 250 ppm. High-purity grade materials were used for this investigation.

Peak areas of absorption values (Q_A) were used throughout.

In order to optimize and develop the FF AAS method, the general operation parameters of graphite temperature, its timing and air-acetylene flame composition (last characterized by relative contents of oxidizer) were investigated.

The conditions of Q_A formation of each element were studied on the basis of contribution to its value some of individual param-

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