

Determination of antimony in lead–zinc concentrates and other smelter products by atomic absorption spectrometry after extraction with di-isopropyl ether and reductive stripping

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Received 15 May 1995; accepted 10 July 1995

Abstract

Several papers have appeared in the literature describing the determination of antimony, where antimony(V) is extracted into the organic phase and the organic solutions directly analyzed by atomic absorption spectrometry (AAS). This paper describes a procedure where antimony from the organic solution is reductively stripped into an aqueous phase and analyzed for antimony by AAS. The advantage of the method for a routine process control laboratory is highlighted.

Keywords: Antimony; Lead–zinc concentrates; Smelter products; Atomic absorption spectrometry

1. Introduction

Determination of antimony by atomic absorption spectrometry (AAS) has been fairly comprehensively covered in the literature [1–6]. Donaldson [1] developed several methods for the determination of small amounts of antimony in copper, nickel, lead, zinc and molybdenum concentrates and related materials. These methods involve preliminary separation of antimony(III) from the matrix element by co-precipitation with hydrous ferric and lanthanum oxides. The precipitate is dissolved in dilute hydrochloric acid and the antimony determined either by AAS or, if the sample contains $<100 \mu\text{g g}^{-1}$ of antimony, by the spectrophotometric iodide method after further separation from iron, lead and other coextracted elements by chloroform extraction as antimony xanthate. Although these methods

yield accurate results for antimony, they are time-consuming, particularly the spectrophotometric method, and the AAS method is not applicable to samples with high lead and iron contents.

Methods have also been employed by several workers in which antimony is extracted by organic solvents from hydrochloric acid solutions and the antimony content determined by aspirating the organic phase into the flame [3–6].

Although analyzing the organic solutions helps to achieve increased sensitivity leading to determination of smaller quantities [2], a few problems are faced in using this technique. The liquid trap should be drained of the water, and filled with organic solvent. While feeding the organic solution, the fuel content has to be kept low to avoid a sooty flame and the flame tends to flare violently unless the fuel and air are tightly controlled. This leads to unstable conditions and fluctuating results. Especially in

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a process control laboratory, the analyst would feel more comfortable feeding aqueous solutions for quick analysis. Recently a paper [7] was published from our laboratories wherein thallium was determined in our process solutions and in zinc and cadmium metals by first extracting thallium into isopropyl ether and then stripping back to the aqueous phase using a sodium sulphite solution.

We have now also worked out a procedure for the determination of antimony on similar lines and applied it to lead and zinc smelter products. The procedure involves suitable decomposition of the sample and taking up the salts in 7.5 M hydrochloric acid. Sodium nitrite is added to oxidise antimony(III). The chloro-complex of antimony(V) is extracted with isopropyl ether, antimony is stripped from the organic phase to the aqueous phase with sodium sulphite solution and the stripped solution is analyzed for antimony.

2. Experimental

2.1. Apparatus

An Electronic Corporation of India model 4103-B atomic absorption spectrometer is used under the following conditions: wavelength, 217.6 nm; spectral band width, 0.2 nm; current, 10 μ A; flame, air–acetylene.

2.2. Reagents

All chemicals used were of analytical grade.

2.2.1. Standard antimony solution

Pure antimony metal (1 g) is heated with 10 ml of concentrated sulphuric acid in a 250 ml beaker until dense white fumes are evolved. Hydrazine sulphate (1 g) is added after cooling and the solution heated again until dense white fumes are evolved. The beaker is then cooled in ice water. 100 ml of 2 M hydrochloric acid is added and the mixture allowed to attain room temperature. The contents of the beaker are transferred to a 1 l standard flask and made up to the mark with 2 M hydrochloric acid. The solution contains 1 mg ml⁻¹ of antimony and is suitably diluted with 7.5 M hydrochloric acid to obtain a 100 μ g ml⁻¹ solution.

2.2.2. Sodium sulphite solution (0.5% w/v)

Anhydrous sodium sulphite (0.5 g) is dissolved in 100 ml of water.

2.2.3. Sodium nitrite solution (25% w/v)

Sodium nitrite (25 g) is dissolved in 100 ml of water.

2.3. Procedures

2.3.1. Calibration

To 30 ml of 7.5 M hydrochloric acid in each of six 125 ml separating funnels 0.0, 1.0, 2.0, 3.0, 4.0 and 5.0 ml of antimony working standard (0–500 μ g) solutions are added from a semimicroburette. Sodium nitrite (1 ml) followed immediately by 20 ml of isopropyl ether are added to each separating funnel. The separating funnels are shaken gently for 1 min and the layers are allowed to separate. The lower aqueous layers are transferred to another set of separating funnels containing 10 ml of isopropyl ether and once again shaken for 1 min. The lower aqueous layers are discarded and the organic layers added to the first set of separating funnels.

Sodium sulphite (20 ml) is added to each separating funnel containing isopropyl ether and shaken for 3 min. The layers are allowed to separate.

The aqueous layers are collected in 50 ml volumetric flasks. The organic layers are further shaken with a small portion (10 ml) of sodium sulphite solution and the aqueous portions collected in the same volumetric flasks. The solutions are made up by the addition of the required quantity of water. The aqueous solutions are then analyzed by AAS for antimony. The absorbance of the blank (zero added antimony), if any, is deducted from the absorbance values of the standards.

2.4. Sample treatment

Depending upon the antimony content, 0.5–1.0 g of sample is placed in a 250 ml beaker. Potassium bisulphate (1 g) and concentrated sulphuric acid (10 ml) are added and the beaker is heated until dense fumes are evolved (1 h). The beaker is cooled and the walls are washed down with a small quantity of water (10 ml). Hydrazine sulphate (1 g) is added and the beaker is heated again until dense white fumes are evolved. The beaker is cooled in the water, 20 ml of 7.5 M hydrochloric acid is

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