TRACE IMPURITIES DETERMINATION IN LEAD BY SPARK-SOURCE MASS SPECTROMETRY

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Very low concentrations of many elemental impurities are determined in a lead matrix by spark-source mass spectrometry using photoplate detection. In order to obtain semiquantitative results, at least one element (antimony) should be accurately determined by an independent technique such as atomic absorption spectroscopy in order to be used as an internal standard.

INTRODUCTION

Lead is a heavy, malleable and very resistant to corrosion metal. It may be hardened by alloying it with small amounts of antimony, tin, copper, arsenic, bismuth, cadmium and sodium, being all of them of industrial importance.

The metal and its alloys are used in solders, pigments, pipe, construction, cable covering, bullets, batteries.

Because of its high density (11.34 g cm$^{-3}$), atomic number and low cost is commonly used as a radiation shield.
It is used in different forms such as sheet, flat and rectangular bricks, in plastics containing up to 90% by weight of Pb for X-ray protection, leaded glass, containers for shipping radioisotopes and lead casks for storing radioactive materials. A purity of 99.5% is suitable for X and γ-ray shields but might be unacceptable for radiation shielding in the presence of neutrons, being necessary in the presence of them, a high purity lead¹.

Spark-source mass spectrometry /SSMS/ has been reported to be a useful analytical technique for the examination of high purity metals, semiconductors and non-conducting powders.

The method is extremely sensitive; the sample does not require any chemical pretreatment, thereby eliminating troublesome blank values. One of its most important advantages is the simultaneous detection of theoretically all elements with comparable sensitivity, at concentration levels below 1 μg.g⁻¹, due to the very high ionization energy used in the spark plasma.

EXPERIMENTAL

The mass spectrometer used was a radiofrequency spark source double focussing instrument with Mattauch-Herzog geometry, Consolidated Electrodynamics Corporation /C.E.C./ 21-110 C.

Photographic detection was performed with Ilford Q-2 /33 cm x 5 cm/ ion-sensitive plates.

27 exposures were recorded on the plate in the range of 8x10⁻¹⁴ to 1x10⁻⁷ C. The plates were developed using a Kodak D-19 developer.