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Methods for chemical analysis of lead and lead alloys - Part 18: Determination of silver, copper, bismuth, arsenic, antimony, tin, zinc, iron, cadmium, nickel, magnesium, aluminum, calcium, selenium and tellurium contents - Inductively coupled plasma mass spectrometry

铅及铅合金化学分析方法 第18部分:银、铜、铋、砷、锑、锡、锌、铁、镉、镍、镁、铝、钙、硒和碲含量的测定 电感耦合等离子体质谱法

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Warning Warning notice

The warning printed at the head of the document reads: persons using this document should have practical experience of work in a formal laboratory. The document does not indicate all the possible safety problems, and it is the responsibility of the user to take suitable safety and health measures and to make sure that the conditions comply with the relevant national regulations.

1 Scope

The document describes a method for determining the contents of silver, copper, bismuth, arsenic, antimony, tin, zinc, iron, cadmium, nickel, magnesium, aluminium, calcium, selenium and tellurium in lead and lead alloys.

It applies to the determination of those elements in lead ingots, secondary lead, secondary

lead-antimony alloy, secondary lead-calcium alloy, lead-calcium alloy ingots for battery grids, lead-antimony alloy ingots for battery grids, lead alloy ingots for cable sheathing, lead anode plates for electrodeposition, and lead and lead-antimony alloy bars and wire.

Table 1 gives the elements and their mass fraction determination ranges: 0.0001 percent to 0.010 percent for silver, copper, arsenic, antimony, tin, zinc, iron, cadmium, nickel, magnesium, aluminium, selenium and tellurium; and 0.0010 percent to 0.010 percent for bismuth and calcium. Footnote a to Table 1 states that when the tin content of the lead or lead alloy is greater than 0.010 percent and not greater than 2.0 percent the determination range for nickel is 0.0010 percent to 0.010 percent, and that the document does not apply when the tin content is greater than 2.0 percent.

2 Normative references

GB/T 6682 Water for analytical laboratory use - Specification and test methods; GB/T 17433 Basic terms of chemical analysis of metallurgical products.

Dated references apply in the edition cited; undated references apply in their latest edition, including any amendments.

3 Terms and definitions

The terms and definitions given in GB/T 17433 apply to the document; no further terms are defined.

4 Principle

The test portion is dissolved in dilute nitric acid or in nitric acid with tartaric acid and measured directly on an inductively coupled plasma mass spectrometer. The mass concentration of each element to be determined is calculated by the working curve method and the result is expressed as a mass fraction. Drift of the instrument and matrix effects on the determination are corrected by the internal standard method.

5 Reagents

Unless otherwise stated, only reagents confirmed to be of guaranteed grade or better are used in the analysis.

5.1 Water complying with grade 1 or grade 2 of GB/T 6682. 5.2 High purity lead with a mass fraction of lead not less than 99.999 percent. 5.3 Tartaric acid. 5.4 Nitric acid of density 1.4 g/mL. 5.5 Hydrochloric acid of density 1.18 g/mL. 5.6 Hydrofluoric acid, 40 percent. 5.7 Hydrogen peroxide, 30 percent. 5.8 Nitric acid diluted 1 to 1. 5.9 Nitric acid diluted 1 to 4. 5.10 Hydrochloric acid diluted 1 to 1.

5.11 Single element standard stock solutions at a mass concentration of 1 mg/mL, either

bought as certified standard solutions or prepared as described in 5.12 to 5.29.

5.12 to 5.26 give the preparation of the standard stock solutions of silver, copper, bismuth, arsenic, antimony, tin, zinc, iron, cadmium, nickel, magnesium, aluminium, calcium, selenium and tellurium. The general pattern for the metals is to weigh 0.5000 g of the metal at a purity of not less than 99.99 percent into a 250 mL beaker, add 50 mL of nitric acid (5.8), dissolve with heating, cool, transfer to a 500 mL volumetric flask, dilute to the mark with water and mix, giving a solution in which 1 mL contains 1 mg of the element.

Departures from that pattern: for bismuth (5.14) a further 25 mL of nitric acid (5.4) is added in the volumetric flask before dilution; for antimony (5.16) the metal is dissolved with 25 mL of nitric acid (5.4) and 3 g of tartaric acid (5.3); for tin (5.17) 0.5000 g is weighed into a 100 mL polytetrafluoroethylene beaker, dissolved with heating in 20 mL of nitric acid (5.8) and 5 mL of hydrofluoric acid (5.6) and transferred to a 500 mL plastic volumetric flask; for aluminium (5.23) 50 mL of hydrochloric acid (5.10) is used in place of nitric acid; and for calcium (5.24) 2.4972 g of calcium carbonate of primary reference grade, dried beforehand at 105 degrees C to 110 degrees C for 1 h and cooled in a desiccator, is weighed into a 250 mL beaker, wetted with 20 mL of water, dissolved by slowly adding 40 mL of nitric acid (5.8) with heating, boiled, cooled to room temperature and transferred to a 1000 mL volumetric flask.

5.27 Scandium standard stock solution: 0.3835 g of scandium sesquioxide of mass fraction not less than 99.99 percent, ignited beforehand at 800 degrees C for 1 h and cooled in a desiccator, is placed in a 100 mL beaker, 25 mL of nitric acid (5.4) is added, hydrogen peroxide (5.7) is added dropwise until dissolution is complete, the solution is boiled and cooled, transferred to a 250 mL volumetric flask, a further 25 mL of nitric acid (5.4) is added and the solution is diluted to the mark; 1 mL contains 1 mg of scandium.

5.28 Caesium standard stock solution: 1.2671 g of caesium chloride of spectroscopic purity, dried at 105 degrees C for 2 h and cooled in a desiccator, is dissolved in water in a 250 mL beaker, transferred to a 1000 mL volumetric flask and diluted to the mark; 1 mL contains 1 mg of caesium. 5.29 Rhenium standard stock solution: 0.5000 g of rhenium of mass fraction not less than 99.99 percent is dissolved with heating in 50 mL of nitric acid (5.8), cooled, transferred to a 500 mL volumetric flask, a further 25 mL of nitric acid (5.4) is added and the solution is diluted to the mark; 1 mL contains 1 mg of rhenium.

5.30 Silver standard solution: 1.00 mL of the silver standard stock solution (5.12) is transferred to a 1000 mL volumetric flask, 50 mL of nitric acid (5.4) is added and the solution is diluted to the mark and mixed; 1 mL contains 1 microgram of silver. The solution is kept in the dark.

5.31 Mixed standard solution: 1.00 mL of each of the single element standard stock solutions of copper, bismuth, arsenic, antimony, tin, zinc, iron, cadmium, nickel, magnesium, aluminium, calcium, selenium and tellurium (5.13 to 5.26) is transferred to a 1000 mL

plastic volumetric flask, 50 mL of nitric acid (5.4) is added and the solution is diluted to the mark and mixed; 1 mL contains 1 microgram of each of those elements. Where commercial standard stock solutions are used for the preparation, elements that interfere chemically with one another or that produce a precipitate are to be prepared in separate groups and a suitable medium chosen.

5.32 Mixed internal standard solution A: 1.00 mL each of the scandium, caesium and rhenium standard stock solutions (5.27 to 5.29) is transferred to a 1000 mL volumetric flask, 50 mL of nitric acid (5.4) is added and the solution is diluted to the mark; 1 mL contains 1 microgram each of scandium, caesium and rhenium. 5.33 Mixed internal standard solution B is prepared in the same way from 0.20 mL of each stock solution; 1 mL contains 200 nanograms of each of the three elements.

5.34 Argon of volume fraction not less than 99.99 percent. 5.35 Helium of volume fraction not less than 99.999 percent. 5.36 Hydrogen of volume fraction not less than 99.999 percent.

6 Apparatus

An inductively coupled plasma mass spectrometer fitted with a collision or reaction cell is used, with argon (5.34) as the carrier gas and helium (5.35), hydrogen (5.36) or another gas recommended by the instrument manufacturer as the collision or reaction cell gas. In the collision or reaction mode the spectral interferences of the argon-chloride, argon-oxide, argon and argon-dimer ions are removed. The mass resolution is to be not greater than 0.8 u.

Table 2 gives the recommended isotope mass numbers and the internal standard element for each element: silver, mass 107 or 109, internal standard caesium or rhenium; copper, 63, scandium or caesium; bismuth, 209, caesium or rhenium; arsenic, 75, scandium or caesium; antimony, 121 or 123, caesium or rhenium; tin, 118, caesium or rhenium; zinc, 66, scandium or caesium; iron, 56, scandium or caesium; cadmium, 111, caesium or rhenium; nickel, 58 or 60, scandium or caesium; magnesium, 24, scandium or caesium; aluminium, 27, scandium or caesium; calcium, 40 or 44, scandium or caesium; selenium, 78 or 80, scandium or caesium; tellurium, 128, caesium or rhenium. The internal standard elements themselves are measured at mass 45 for scandium, 133 for caesium and 187 for rhenium. The note to Table 2 states that arsenic, iron, calcium and selenium are determined in the collision or reaction mode.

7 Samples

The sample is to be in the form of chips whose largest edge is not greater than 3 mm.

8 Test procedure