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Methods for chemical analysis of tin - Part 1: Determination of copper, lead, zinc, cadmium, silver, nickel and cobalt contents - Flame atomic absorption spectroscopy

锡化学分析方法 第1部分:铜、铅、锌、镉、银、镍和钴含量的测定 火焰原子吸收光谱法

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1 Scope

This document describes methods for the determination of the copper, lead, zinc, cadmium, silver, nickel and cobalt contents of tin.

This document applies to the determination of the copper, lead, zinc, cadmium, silver, nickel and cobalt contents of tin. The determination range of each element is given in Table 1: copper from 0.000 10 % to 0.100 %; lead from 0.000 80 % to 0.050 0 %; zinc from 0.000 10 % to 0.003 0 %; cadmium from 0.000 10 % to 0.002 0 %; silver and nickel from 0.000 08 % to 0.010 0 %; and cobalt from 0.000 10 % to 0.007 0 %.

4 Principle

The test portion is dissolved in hydrochloric acid and nitric acid, and the bulk of the tin is driven off with a hydrochloric and hydrobromic acid mixture. In a mixed hydrochloric and nitric acid medium, using an air-acetylene flame, the absorbance of copper, lead, zinc, cadmium, silver, nickel and cobalt is measured separately at the recommended wavelength of each element on an atomic absorption spectrometer, and the content of each element is calculated by the working curve method.

5 Reagents

Unless otherwise stated, only reagents confirmed to be of guaranteed grade and water of

grade two as specified in GB/T 6682 are used in the analysis.

Hydrochloric acid of density 1.19 g/mL; nitric acid of density 1.42 g/mL; sulfuric acid 1 plus 1; a hydrochloric and hydrobromic acid mixture made from one volume of hydrochloric acid and one volume of hydrobromic acid of density 1.49 g/mL, mixed and prepared fresh; a hydrochloric and nitric acid mixture made from three volumes of hydrochloric acid and one volume of nitric acid, mixed and prepared fresh; hydrochloric acid 1 plus 1; nitric acid 1 plus 1; and nitric acid 2 plus 1.

Each stock standard solution is prepared by weighing 1.000 0 g of the metal, of mass fraction not less than 99.99 %, into a 250 mL beaker, adding 50 mL of acid, covering with a watch glass, heating gently until dissolution is complete and cooling; the solution is transferred into a 1 000 mL volumetric flask, 30 mL of acid is added, and it is diluted to the mark with water and mixed, giving a solution of which 1 mL contains 1 mg of the metal. Copper, lead, cadmium, silver and nickel are dissolved in nitric acid 1 plus 1 and made up with 30 mL of concentrated nitric acid; zinc and cobalt are dissolved in hydrochloric acid 1 plus 1 and made up with 30 mL of concentrated hydrochloric acid, and their solutions are transferred to plastic bottles for storage.

The mixed standard solution is prepared by first placing 150 mL of the hydrochloric and nitric acid mixture in a 1 000 mL volumetric flask, then transferring into it 20.00 mL each of the copper, silver, nickel and cobalt stock solutions, 10.00 mL each of the zinc and cadmium stock solutions and 60 mL of the lead stock solution, diluting to the mark with water and mixing; it is stored in a plastic bottle. One millilitre of this solution contains 20 micrograms each of copper, silver, nickel and cobalt, 10 micrograms each of zinc and cadmium, and 60 micrograms of lead.

6 Apparatus

An atomic absorption spectrometer fitted with hollow cathode lamps for copper, lead, zinc, cadmium, silver, nickel and cobalt. Any instrument that reaches the following figures under its best working conditions may be used.

Characteristic concentration, measured in a solution whose matrix matches that of the solution to be measured: not more than 0.020 micrograms per millilitre for copper, not more than 0.080 for lead, not more than 0.005 for zinc, not more than 0.007 for cadmium, not more than 0.010 for silver, not more than 0.020 for nickel and not more than 0.020 for cobalt.

Minimum stability: measuring the absorbance ten times with the standard solution of the highest concentration, the standard deviation shall not exceed 1 % of the mean absorbance; measuring the absorbance ten times with the standard solution of the lowest concentration, which is not the zero concentration standard solution, the standard deviation shall not exceed 0.5 % of the mean absorbance of the highest concentration standard

solution.

Linearity of the working curve: the calibration curve is divided into five equal concentration segments, and the ratio of the absorbance difference of the highest segment to the absorbance difference of the lowest segment shall be not less than 0.7.

The recommended wavelengths of the elements are given in Table 2: copper 324.7 nm, lead 283.3 nm, zinc 213.9 nm, cadmium 228.8 nm, silver 328.1 nm, nickel 232.0 nm and cobalt 240.7 nm.

7 Samples

The sample is in the form of chips not more than 1 mm thick.

8 Test procedure

Test portion. The sample is weighed to 0.000 1 g in the amount given in Table 3. That table pairs the determination range of each element with the mass to be weighed, the volume to which the solution is made up, the volume of hydrochloric and nitric acid mixture to be added back and, for the highest ranges of copper and lead, the aliquot to be taken, the volume in which it is measured and the further addition of acid mixture; the individual values of that table are not reproduced here.

Duplicate tests. Two determinations are made in parallel and the mean is taken.

Blank test. A blank test is carried out alongside the test portion.

Determination. The test portion is placed in a 300 mL beaker; 15 mL of hydrochloric acid 1 plus 1 and 2 mL to 3 mL of nitric acid are added and the mixture is heated gently until dissolution is complete, then taken off the heat and cooled slightly. One millilitre of sulfuric acid 1 plus 1 and 5 mL of the hydrochloric and hydrobromic acid mixture are added and the solution is heated until sulfuric acid fumes appear, then taken off and cooled slightly. This step is repeated two to three times until the bulk of the tin has been driven off and the sulfuric acid fumes have gone; if no obvious salts remain on evaporation to dryness, 1 mL of sulfuric acid 1 plus 1 is added and the step repeated. The residue is cooled to room temperature, the hydrochloric and nitric acid mixture is added back as given in Table 3, the beaker is covered with a watch glass and heated to boiling on a hot plate, then cooled to room temperature; an aliquot is taken and diluted as given in Table 3, or the solution is transferred into a volumetric flask of the corresponding volume, diluted to the mark with water and mixed.

Using an air-acetylene flame, the absorbance of the blank solution and of the test solution is measured at the recommended wavelength of each element on the atomic absorption spectrometer at the same time as the series of standard solutions, the zero being set with water, and the content of the corresponding element is read from the working curve.

Working curve. Volumes of 0 mL, 0.50 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL and 6.00 mL of the mixed standard solution are transferred into a set of 100 mL volumetric flasks, 15 mL of the hydrochloric and nitric acid mixture is added, and the flasks are diluted to the mark with water and mixed. Under the same conditions as for the test solution, and with the zero set with water, the absorbance of the series of standard solutions is measured, the absorbance of the zero concentration solution of the series is subtracted, and the working curve is drawn with the mass concentration of the corresponding standard solution on the horizontal axis and the absorbance on the vertical axis.

9 Processing of test data

The content of the element sought in the test solution is expressed as the mass fraction of that element and is calculated from the mass concentration of the element in the test solution read from the working curve, the mass concentration of the element in the blank solution read from the working curve, the volume to which the test solution was made up, the volume in which the test solution was measured, the volume of the aliquot taken, and the mass of the test portion, all in micrograms per millilitre, millilitres and grams as appropriate.

Where the mass fraction is less than 0.001 0 %, it is expressed to five decimal places; where it is 0.001 0 % or more but less than 0.100 %, to four decimal places; and where it is 0.100 % or more, to three decimal places.

10 Precision

Repeatability. Under repeatability conditions, the absolute difference between the values of two independent test results obtained within the range of mean values given in Table 4 shall not exceed the repeatability limit r, and cases in which it does exceed r shall not be more than 5 %. The repeatability limit r is obtained from the data of Table 4 by linear interpolation or extrapolation.

Reproducibility. Under reproducibility conditions, the absolute difference between the values of two independent test results obtained within the range of mean values given in Table 5 shall not exceed the reproducibility limit R, and cases in which it does exceed R shall not be more than 5 %. The reproducibility limit R is obtained from the data of Table 5 by linear interpolation or extrapolation.

Table 4 and Table 5 pair, for each of the seven elements, a series of mass fraction levels with the corresponding repeatability limit or reproducibility limit; the individual values are not reproduced here. Annex A reports the statistical data of the interlaboratory test from which the precision data come: the precision was established by twelve laboratories on five or six different levels of test material, each laboratory determining the copper, lead, zinc, cadmium, silver, nickel and cobalt content of each level seven times independently under