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Methods for chemical analysis of nickel - Part 11: Determination of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, bismuth contents - Inductively coupled plasma mass spectrometry

镍化学分析方法 第 11 部分：镁、铝、锰、钴、铜、锌、镉、锡、锑、铅、铋含量的测定 电感耦合等离子体质谱法

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Foreword

GB/T 8647 "*Methods for chemical analysis of nickel*" is divided into 11 parts:

- Part 1: Determination of iron content - Sulfosalicylic acid spectrophotometric method;
- Part 2: Determination of aluminium content - Electrothermal atomic absorption spectrometric method;
- Part 3: Determination of silicon content - Molybdenum blue spectrophotometric method;
- Part 4: Determination of phosphorous content - Molybdenum blue spectrophotometric method;
- Part 5: Determination of magnesium content - Flame atomic absorption spectrometric method;
- Part 6: Determination of cadmium, cobalt, copper, manganese, lead and zinc contents - Flame atomic absorption spectrometric method;
- Part 7: Determination of arsenic, antimony, bismuth, tin and lead contents - Electrothermal atomic absorption spectrometric method;
- Part 8: Determination of sulphur content - Infra-red absorption method after high frequency induction furnace combustion;
- Part 9: Determination of carbon content - Infra-red absorption method after high frequency induction furnace combustion;
- Part 10: Determination of arsenic, cadmium, lead, zinc, antimony, bismuth, tin, cobalt, copper, manganese, magnesium, silicon, aluminium, iron contents - Atomic emission spectrometric method;
- Part 11: Determination of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, bismuth contents - Inductively coupled plasma mass spectrometry.

This Part is Part 11 of GB/T 8647.

This Part was drafted in accordance with the rules given in GB/T 1.1-2009.

This Part was proposed by China Nonferrous Metals Industry Association.

This Part shall be under the jurisdiction of the National Technical Committee on Nonferrous Metals of Standardization Administration of China (SAC/TC 243).

Methods for chemical analysis of nickel - Part 11: Determination of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, bismuth contents - Inductively coupled plasma mass spectrometry

1 Scope

This Part of GB/T 8647 specifies the methods for the determination of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, and bismuth contents in nickel.

This Part applies to the determination of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, and bismuth contents in nickel. The determination range is 0.00010 % ~ 0.0050 %.

2 Method summary

Dissolve the sample in nitric acid, on an inductively coupled plasma mass spectrometer, using the working curve method and the method of online addition of an internal standard mixed solution of scandium, rhodium, and rhenium, determine the contents of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, and bismuth in the test solution.

3 Reagents or materials

Unless otherwise specified, the water used in the analysis is grade 1 water or water of equivalent purity. All test instruments are thoroughly rinsed with hot nitric acid and then rinsed with water.

3.1 Nitric acid (ρ = 1.42 g/mL), MOS grade.

3.2 Nitric acid (1 + 1).

3.3 Standard stock solution: Use valid, certified single-element standard stock solutions of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, bismuth, scandium, rhodium, and rhenium, with a mass concentration of 1000 μg/mL.

3.4 Mixed standard solution A: Respectively transfer 1.00 mL of each standard stock solution (3.3) of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, and bismuth into a 100 mL volumetric flask containing 10 mL of nitric acid (3.2), dilute to the mark with water, and mix thoroughly. 1 mL of this solution contains 10 μg of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, and bismuth respectively.

3.5 Mixed standard solution B: Respectively transfer 1.00 mL of each mixed standard solution A (3.4) into a 100 mL volumetric flask containing 10 mL of nitric acid (3.2), dilute to the mark with water, and mix thoroughly. 1 mL of this solution contains 100 ng of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, and bismuth respectively.

3.6 Internal standard mixed standard solution: Use the standard stock solution of scandium, rhodium, and rhenium (3.3) with a mass concentration of 1000 μg/mL, dilute sequentially before use, so that 1 mL of the diluted solution contains 10 ng of scandium, rhodium, and rhenium respectively. The medium is nitric acid (2 + 98).

3.7 Argon ($w_{Ar} \geq 99.99$ %).

4 Instrumentation

4.1 Inductively coupled plasma mass spectrometer, with an instrument mass resolution of not less than 0.8 amu ± 0.1 amu.

4.2 The recommended mass numbers for isotope determination of magnesium, aluminum, manganese, cobalt, copper, zinc, cadmium, tin, antimony, lead, bismuth, scandium, rhenium, and rhodium are shown in Table 1.

5 Sample

The sample shall be in the form of crumbs or powder.

6 Test procedure

6.1 Test material

Weigh 1.00 g of the sample, to the nearest 0.0001 g.

6.2 Parallel test

Perform two tests in parallel.

6.3 Blank test

Perform a blank test along with the test material.

6.4 Determination

6.4.1 Place the test material (6.1) in a 100 mL polytetrafluoroethylene or quartz beaker, add 20.0 mL of nitric acid (3.2), and heat at low temperature until the test material is completely dissolved. Remove the beaker, cool down and transfer to a 100 mL plastic volumetric flask, dilute to the mark with water, and mix thoroughly.

6.4.2 Take 10.00 mL of the test solution (6.4.1) into a 100 mL plastic volumetric flask, add 2.0 mL of nitric acid (3.1), dilute to the mark with water, and mix thoroughly.

6.4.3 Under the selected instrument operating conditions, according to the isotope mass numbers recommended in Table 1, using the working curve method and the method of online addition of the internal standard mixed standard solution (3.6), determine the test solution (6.4.2) and the blank test solution (6.3) simultaneously with the series standard solutions. The instrument automatically calculates the mass concentration of each determined element in the test solution based on the working curve.

6.5 Plotting of working curve

6.5.1 Respectively transfer 0 mL, 1.00 mL, 5.00 mL, 10.00 mL, and 20.00 mL of mixed standard solution B (3.5) and 0.50 mL of mixed standard solution A (3.4) to a set of 100 mL plastic volumetric flasks, add 2.0 mL of nitric acid (3.1), dilute to the mark with water, and mix thoroughly.

6.5.2 On an inductively coupled plasma mass spectrometer, under the selected instrument operating conditions, according to the isotope mass numbers recommended in Table 1, using the method of online addition of the internal standard mixed standard solution (3.6), determine the series standard solutions (6.5.1). The instrument automatically plots the working curve. The correlation coefficient of the working curve for each element shall be above 0.999, otherwise the determination shall be repeated, or the series standard solutions shall be re-prepared for determination.

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