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Methods for chemical analysis of aluminium and aluminium alloys -

Part 1: Determination of mercury content

铝及铝合金化学分析方法

第1部分: 汞含量的测定

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Foreword

GB/T 20975 "Methods for chemical analysis of aluminium and aluminium alloys" is divided into 31 parts:

- Part 1: Determination of mercury content;
- Part 2: Determination of arsenic content;
- Part 3: Determination of copper content;
- Part 4: Determination of iron content Orthopenanthroline photometric method;
- Part 5: Determination of silicon content;
- Part 6: Determination of cadmium content Flame atomic absorption spectrometric method;
- Part 7: Determination of manganese content Potassium periodate spectrophotometric method;
- Part 8: Determination of zinc content;
- Part 9: Determination of lithium content Flame atomic absorption spectrometric method;
- Part 10: Determination of tin content;
- Part 11: Determination of lead content;
- Part 12: Determination of titanium content;
- Part 13: Determination of vanadium content N-benzoyl-Nphenylhydroxylamine spectrophotometric method;
- Part 14: Determination of nickel content;
- Part 15: Determination of boron content;
- Part 16: Determination of magnesium content;
- Part 17: Determination of strontium content Flame atomic absorption spectrometric method;
- Part 18: Determination of chromium content;

Methods for chemical analysis of aluminium and aluminium alloys -

Part 1: Determination of mercury content

1 Scope

This Part of GB/T 20975 specifies the method for determination of mercury content in aluminium and aluminium alloys.

This Part applies to the determination of mercury content in aluminium and aluminium alloys. All coexisting elements in aluminium and aluminium alloys do not interfere with the determination of mercury. The measurement range of Method 1 is: >0.0001%~0.010%. The measurement range of Method 2 is: 0.000001%~0.0001%.

2 Method 1: Cold atomic absorption spectrometric method

2.1 Method principle

The test portion is dissolved with hydrochloric acid-nitric acid mixed acid; the divalent mercury in the solution is reduced to mercury with stannous chloride; and on a cold atomic absorption mercury analyzer, at a wavelength of 253.7 nm, the absorbance of mercury vapor is measured, to calculate the mass fraction of mercury.

2.2 Reagents and materials

Unless otherwise stated, in the analysis, only identified guaranteed reagents and Grade 1 water are used.

- **2.2.1** Nitric acid (ρ =1.42 g/mL).
- **2.2.2** Hydrochloric acid (ρ =1.19 g/mL).
- **2.2.3** Mixed acid: Nitric acid (2.2.1), hydrochloric acid (2.2.2), and water are mixed in a volume ratio of (1+3+4).

- **2.2.4** Potassium dichromate solution (25 g/L): WEIGH 25 g of potassium dichromate, DISSOLVE in water; TRANSFER to a 1000 mL volumetric flask; USE water to dilute to volume, and MIX well.
- **2.2.5** Nitric acid-potassium dichromate solution: WEIGH 0.5 g of potassium dichromate and DISSOLVE in water; ADD 50 mL of nitric acid (2.2.1); TRANSFER to a 1000 mL volumetric flask; USE water to dilute to volume, and MIX well.
- **2.2.6** Stannous chloride solution (200 g/L): WEIGH 20 g of stannous chloride (SnCl₂ · H₂O); DISSOLVE it in 20 mL of hydrochloric acid (2.2.2); TRANSFER to a 100 mL volumetric flask; USE water to dilute to volume, and MIX well.
- **2.2.7** Standard stock solution of mercury (100 μ g/mL): WEIGH 0.1354 g of mercury bichloride [w(HgCl₂) \geq 99.95%] which has been thoroughly dried in a silica gel drier; and ADD 5 mL of nitric acid (2.2.1) and a small amount of water. After micro-heat dissolution, TRANSFER to a 1000 mL volumetric flask; USE nitric acid-potassium dichromate solution (2.2.5) to dilute to volume, and MIX well. 1 mL of this solution contains 100 μ g of mercury.
- **2.2.8** Mercury standard solution (10 μ g/mL): PIPETTE 10.00 mL of standard stock solution of mercury (2.2.7) in a 100 mL volumetric flask; USE nitric acid-potassium dichromate solution (2.2.5) to dilute to volume, and MIX well. 1 mL of this solution contains 10 μ g of mercury. Prepared when used.
- **2.2.9** Mercury standard solution (0.1 μ g/mL): PIPETTE 2.00 mL of mercury standard solution (2.2.8) in a 200 mL volumetric flask; USE nitric acid-potassium dichromate solution (2.2.5) to dilute to volume, and MIX well. 1 mL of this solution contains 0.1 μ g of mercury. Prepared when used.

2.3 Instrument

Cold atomic absorption mercury analyzer.

2.4 Sample

The sample is processed into chips having a thickness of no more than 1 mm.

2.5 Analytical procedures

2.5.1 Test portion

Sample (2.4) is weighed according to Table 1, accurate to 0.0001 g.

- **3.2.6** Hydrochloric acid (5+95).
- **3.2.7** Potassium borohydride solution (4 g/L): WEIGH 1.0 g of potassium borohydride in a 300 mL beaker; USE sodium hydroxide solution (5 g/L) to dilute to 250 mL, and MIX well. Prepared when used.
- **3.2.8** Standard stock solution of mercury (1.0 mg/mL).
- **3.2.9** Mercury standard solution A (10 μ g/mL): PIPETTE 1.00 mL of standard stock solution of mercury (3.2.8) into a 100 mL volumetric flask; ADD 10.0 mL of nitric acid (3.2.4); USE water to dilute to volume, and MIX well. 1 mL of this solution contains 10 μ g of mercury. Prepared when used.
- **3.2.10** Mercury standard solution B (0.1 μ g/mL): PIPETTE 1.00 mL of mercury standard solution A (3.2.9) into a 100 mL volumetric flask; ADD 10.0 mL of nitric acid (3.2.4); USE water to dilute to volume, and MIX well. 1 mL of this solution contains 0.1 μ g of mercury. Prepared when used.
- **3.2.11** Mercury standard solution C (0.01 μ g/mL): PIPETTE 10.00 mL of mercury standard solution B (3.2.10) into a 100 mL volumetric flask; USE hydrochloric acid (3.2.6) to dilute to volume, and MIX well. 1 mL of this solution contains 0.01 μ g of mercury. Prepared when used.

3.3 Instrument

Atomic fluorescence spectrometer, with mercury hollow cathode lamp.

3.4 Sample

The sample is processed into chips having a thickness of no more than 1 mm.

3.5 Analytical procedures

3.5.1 Test portion

WEIGH 1.00 g of sample (3.4), accurate to 0.0001 g.

3.5.2 Number of determinations

The determination is performed twice independently, and the average is taken.

3.5.3 Blank test

A blank test is carried out along with the test portion (3.5.1).

3.5.4 Determination

3.5.4.1 PLACE the test portion (3.5.1) in a 400 mL beaker; ADD 30 mL of mixed

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