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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13748.2-2026

Replacing GB/T 13748.2-2005

Methods for chemical analysis of magnesium and magnesium alloys - Part 2: Determination of tin, beryllium, copper, nickel and titanium content - Spectrophotometry

镁及镁合金化学分析方法 第2部分：锡、铍、铜、镍、钛含量的测定 分光光度法

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Foreword

The sample was processed into fragments with a thickness of no more than 1 mm.

6 Determination of Tin Content

6.1 Method Overview The sample was decomposed with nitric acid, and iron salt was added in the presence of ammonium chloride. The pH of the solution was adjusted to 7-8 with sodium hydroxide, and tin was separated by co-precipitation. Magnesium is dissolved and precipitated with sulfuric acid. In a sulfuric acid-citric acid medium, tetravalent tin ions react with catechol violet and hexadecyltrimethylammonium bromide to form a green precipitate. The absorbance of the colored complex was measured at a wavelength of 662 nm using a spectrophotometer. The mass of tin was then determined from the working curve, and the mass of tin was calculated. Quantitative fraction. Interference from ferric ions was eliminated using ascorbic acid.

6.2 Reagents or Materials Unless otherwise specified, only reagents confirmed to be of analytical grade and grade II water conforming to GB/T 6682 shall be used in the analysis.

6.2.1 Ammonium chloride, analytical grade.

6.2.2 Ammonia (rho=0.90g/mL).

6.2.3 Nitric acid (1 1).

6.2.4 Sulfuric acid (1 1).

6.2.5 Sulfuric acid-hydrogen peroxide. 100 mL of sulfuric acid (

1.0 mol/L) contains 2 to 3 drops of hydrogen peroxide (rho =

6.2.6 Sulfuric acid-citric acid mixture. Weigh 25.0g of citric acid (C6H8O7) and dissolve it in 400mL of water, then slowly add 30mL of sulfuric acid. (rho=1.84g/mL), dilute with water to 500mL and mix well.

6.2.7 Sodium hydroxide solution (200 g/L).

6.2.8 Ascorbic acid solution (20 g/L). Prepare as needed.

6.2.10 Hexadecyltrimethylammonium bromide solution (

0.3 g/L). Weigh

0.15 g of hexadecyltrimethylammonium bromide (C19H42BrN) and dissolve it in... Dilute 350 mL of anhydrous ethanol (rho =

0.79 g/mL) with water to 500 mL and mix well.

6.2.11 Iron solution (1 g/L). Weigh

0.484 g of ferric chloride hexahydrate (FeCl3·6H2O) into a beaker, add 6 mL of hydrochloric acid (1.1), Dissolve and dilute with water to 100 mL, then mix well.

6.2.12 Ammonium chloride washing solution (10g/L). Weigh 1g of ammonium chloride (6.2.1) and dissolve it in 100mL of water, then add 1mL of ammonia water (6.2.2).

6.2.13 Tin Standard Stock Solution (

0.1 mg/mL). Weigh 0.1000 g of metallic tin (wSn >= 99.9%) into a 150 mL beaker, add... 10 mL of sulfuric acid (rho =

1.84 g/mL) was heated until completely dissolved and white fumes were emitted. After cooling, 25 mL of sulfuric acid (rho =

1.84 g/mL) was added. Transfer sulfuric acid (1.9) to a 1000 mL volumetric flask, dilute to the mark with sulfuric acid (1.9), and mix well. 1 mL of this solution contains

0.1 mg of tin. Or Use certified standard solutions.

6.2.14 Tin Standard Solution (5.0 μg/mL). Transfer

25.00 mL of tin standard stock solution (6.2.13) to a 500 mL volumetric flask, and precipitate with sulfur. Dilute with acid (1 9) to the mark and mix well. 1 mL of this solution contains 5.0 μg of tin. Prepare as needed.

6.3 Test Procedure

6.3.1 Sample Weigh the sample according to Table 2 (Chapter 5), accurate to 0.0001g, and record it as m0.

6.3.2 Parallel Tests Perform the experiment twice in parallel and take the average value.

6.3.3 Blank Test A blank test was performed along with the sample.

6.3.4 Measurement

6.3.4.1 Place the sample in a 300mL beaker, add approximately 50mL of water, and add

35mL of nitric acid in portions (6.2.3). Cover with a watch glass and heat. Boil until the sample is completely dissolved for 5 minutes, rinse the petri dish and cup walls with water, and add water to 150 mL.

6.3.4.2 Add 5 mL of iron solution (6.2.11) and 10 g of ammonium chloride (6.2.1), and adjust with ammonia water (6.2.2) until ferric hydroxide precipitate initially appears (at this point, the solution...). Add 6 mL to 7 mL of ammonia water (6.2.2) to the solution (pH 6-7), add water to.200 mL, and boil for 1 to 2 minutes.

6.3.4.3 Remove and let cool slightly, then filter while still warm using rapid filter paper. Wash the beaker and precipitate 8 to 10 times each with hot ammonium chloride washing solution (6.2.12), then discard. Remove the filtrate. Dissolve the precipitate in 20 mL of hot sulfuric acid-hydrogen peroxide (6.2.5) in the original beaker, and wash with water 4-5 times. Dissolve in sodium hydroxide. Adjust the solution (6.2.7) until a brown precipitate initially appears, then adjust with sulfuric acid (6.2.4) until the precipitate just dissolves and one drop in excess, and heat until the test solution is clear.

6.3.4.4 Transfer the test solution into the corresponding volumetric flask (V0) according to Table 2, dilute with water to the mark, and mix well. Dispense the test solution (V1) into 50 mL containers according to Table 2. In a volumetric flask, add

10.0 mL of sulfuric acid-citric acid mixed acid (6.2.6) and

5.0 mL of ascorbic acid solution (6.2.8), mix well, and then add

3.0 mL of... Catechol purple solution (6.2.9),

3.0 mL hexadecyltrimethylammonium bromide solution (6.2.10) (mix gently after each addition of reagent), water Dilute to the mark, mix well, and let stand for 30 minutes.

6.3.4.5 Transfer a portion of the test solution into a 1cm cuvette, using the blank test solution accompanying the sample as a reference, and measure the solution at wavelength (4.1) on a spectrophotometer. The absorbance was measured at 662 nm, and the mass of tin (mSn) was obtained from the working curve.

6.3.5 Plotting the Working Curve

6.3.5.1 Transfer 0 mL,

5.00 mL of tin standard solution (6.2.14) to a set of... In a 50 mL volumetric flask, add

10.0 mL of a sulfuric acid-citric acid mixture (6.2.6) and

5.0 mL of ascorbic acid solution (6.2.8), respectively, mix well, and then divide into portions. Do not add

3.0 mL of catechol purple solution (6.2.9) or

3.0 mL of hexadecyltrimethylammonium bromide solution (6.2.10). (Each reagent should be