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Methods for chemical analysis of crude nickel hydroxide - Part 1: Determination of nickel content - Dimethylglyoxime gravimetric

粗氢氧化镍化学分析方法 第 1 部分：镍量的测定 丁二酮肟重量法

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Foreword

YS/T 1229-2018 "Methods for chemical analysis of crude nickel hydroxide" is divided into 4 parts:

- Part 1: Determination of nickel content - Dimethylglyoxime gravimetric method;
- Part 2: Determination of cobalt content - Flame atomic absorption spectrometric method;
- Part 3: Determination of copper, cobalt, manganese calcium, magnesium, zinc, iron, aluminum, lead, arsenic and cadmium contents - Inductively coupled plasma atomic emission spectrometry;
- Part 4: Determination of chlorine content - Turbidimetric method.

This Part is Part 1.

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

This Part was proposed by AND shall be under the jurisdiction of the National Technical Committee for Standardization of Nonferrous Metals (SAC/TC 243).

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Methods for chemical analysis of crude nickel hydroxide - Part 1: Determination of nickel content - Dimethylglyoxime gravimetric

1 Scope

This Part specifies the determination method of nickel content in crude nickel hydroxide.

This Part is applicable to the determination of nickel content in crude nickel hydroxide. The determination range is 20.00% ~ 50.00%.

2 Method summary

After the sample is dissolved in acid, nickel and dimethylglyoxime generate red precipitate in a slightly ammonia solution. The precipitate is filtered, washed, and dried at 120 °C to a constant weight; then it is weighed. The nickel content in the mixed filtrate is extracted with chloroform at pH6.5, separated from other impurities, corrected by dimethylglyoxime spectrophotometry.

3 Reagents

Unless otherwise specified, all reagents used are analytically pure reagents; the water used is distilled water or deionized water.

3.1 Ammonium bifluoride.

3.2 Nitric acid (ρ = 1.42 g/mL).

3.3 Sulfuric acid (ρ = 1.84 g/mL).

3.4 Hydrochloric acid (1 + 1).

3.5 Ammonia water (1 + 1).

3.6 Nitric acid (1 + 1).

3.7 Chloroform.

3.8 Ammonium acetate solution (200 g/L), filter before use.

3.9 Sodium thiosulfate solution (200 g/L), filter before use.

3.10 Potassium sodium tartrate solution (200 g/L), filter before use.

3.11 Ethanol (1 + 4).

3.12 Dimethylglyoxime ethanol solution (10 g/L), filter before use.

3.13 Dimethylglyoxime-sodium hydroxide solution (10 g/L): Dissolve 15 g of sodium hydroxide in 100 mL of water; cool to room temperature; dissolve 1 g of dimethylglyoxime in the solution; mix well.

3.14 Nickel standard stock solution: Weigh 1.0000 g of metallic nickel (≥ 99.99%); place in a 400 mL beaker; add 20 mL of nitric acid (3.6); cover with a watch glass; dissolve completely at low temperature; add 2 mL of sulfuric acid (3.3); continue heating until a large amount of white smoke disappears; cool it. Rinse the watch glass and the wall of the cup with water; heat to dissolve the salts; cool to room temperature; transfer to a 1000 mL volumetric flask; dilute to scale with water; mix it. 1 mL of this solution contains 1 mg nickel.

3.15 Nickel standard solution: Pipette 5.00 mL of nickel standard stock solution (3.14) to a 1000 mL volumetric flask; dilute to mark with water; mix it. 1 mL of this solution contains 5 μg of nickel.

4 Instruments

4.1 Electric constant temperature drying oven.

4.2 Glass crucible G4 (filter plate pore size 5 μm ~ 15 μm), 30 mL.

4.3 Spectrophotometer.

5 Specimen

5.1 The sample particle size is not greater than 0.154 mm.

5.2 The analysis sample is dried in advance according to the conditions agreed upon by the supplier and the buyer. It is taken out of the oven and immediately placed in a desiccator. It is weighed immediately after cooling to room temperature.

6 Analytical procedures

6.1 Sample

Weigh 1.00 g of specimen, accurate to 0.0001 g.

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