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Methods for chemical analysis of crude nickel hydroxide - Part 3: Determination of copper, cobalt, manganese, calcium, magnesium, zinc, iron, aluminum, lead, arsenic and cadmium contents - Inductively coupled plasma atomic emission spectrometry

粗氢氧化镍化学分析方法 第 3 部分：锌、铁、铝、铅、砷和镉量的测定 电感耦合等离子体原子发射光谱法

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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 3.

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 3: Determination of copper, cobalt, manganese, calcium, magnesium, zinc, iron, aluminum, lead, arsenic and cadmium contents - Inductively coupled plasma atomic emission spectrometry

1 Scope

This Part specifies the determination method of copper, cobalt, manganese, calcium, magnesium, zinc, iron, aluminum, lead, arsenic, and cadmium in crude nickel hydroxide.

This Part is applicable to the determination of copper, cobalt, manganese, calcium, magnesium, zinc, iron, aluminum, lead, arsenic, cadmium in crude nickel hydroxide. The determination range is shown in Table 1.

2 Method summary

The test samples are decomposed with hydrochloric acid, nitric acid, hydrofluoric acid, perchloric acid. The emission intensity of each element is measured on an inductively coupled plasma atomic emission spectrometer in a dilute hydrochloric acid medium; the mass fraction of each element is calculated according to the working curve method.

3.12 Calcium standard stock solution: Weigh 2.500 g of calcium carbonate (standard, pre-dried at 105 °C ~ 110 °C to constant weight, placed in a desiccator to cool to room temperature) in a 250 mL beaker; add 20 mL of hydrochloric acid (3.2); cover with a watch glass; heat to dissolve; boil to remove carbon dioxide; transfer to a 1000 mL volumetric flask after cooling; use water to dilute it to the mark; mix well. 1 mL of this solution contains 1 mg calcium.

3.13 Magnesium standard stock solution: Weigh 1.6582 g of magnesium oxide (W_{MgO} ≥ 99.95%, pre-calcined at 800 °C to constant weight, placed in a desiccator to cool to room temperature) in a 250 mL beaker; add a small amount of water to moisten; add 10 mL of hydrochloric acid (3.2); cover with a watch glass; dissolve completely at low temperature; transfer to a 1000 mL volumetric flask after cooling; use water to dilute it to the mark; mix well. 1 mL of this solution contains 1 mg of magnesium.

3.14 Standard stock solution of zinc: Weigh 0.1000 g of metallic zinc (w_{Zn} ≥ 99.95%); place it in a 400 mL beaker; add 20 mL of nitric acid (3.4); cover with a watch glass; dissolve completely at low temperature; rinse the watch glass and the wall of the beaker with water; heat and boil to expel nitrogen oxides. Remove and cool it; transfer to a 1000 mL volumetric flask; use water to dilute it to the mark; mix it. 1 mL of this solution contains 0.1 mg of zinc.

3.15 Standard stock solution of iron: Weigh 0.1430 g of ferric oxide (reference); place it in a 400 mL beaker; add 15 mL of hydrochloric acid (3.1); cover with a watch glass; heat at low temperature to dissolve completely; cool and transfer to a 1000 mL volumetric flask; use water to dilute it to the mark; mix it. 1 mL of this solution contains 0.1 mg of iron.

3.16 Aluminum standard stock solution: Weigh 0.1000 g of aluminum (w_{Al} ≥ 99.95%); place in a 400 mL beaker; add 50 mL of hydrochloric acid (3.2); cover with a watch glass; heat at low temperature to dissolve completely; cool and transfer to a 1000 mL volumetric flask; use water to dilute it to the mark; mix it. 1 mL of this solution contains 0.1 mg aluminum.

3.17 Lead standard stock solution: Weigh 0.1000 g of lead (w_{Pb} ≥ 99.95%); place in a 400 mL beaker; add 20 mL of nitric acid (3.4); cover with a watch glass; dissolve completely at low temperature; cool and transfer to a 1000 mL volumetric flask; use water to dilute it to the mark; mix it. 1 mL of this solution contains 0.1 mg lead.

3.18 Arsenic standard storage solution: Weigh 0.1320 g of arsenic trioxide (standard, pre-dried at 100 °C ~ 105 °C for 1 h, placed in a dryer and cooled to room temperature) in a 100 mL beaker; add 5 mL of sodium hydroxide solution (3.7); cover with a watch glass; heat at low temperature until completely dissolved; add 5 mL of water and 2 drops of phenolphthalein ethanol solution (3.8); neutralize with hydrochloric acid (3.2) until the red color just disappears; then add 2 mL in excess; transfer to a 1000 mL volumetric flask; use water to dilute it to the mark; mix well. 1 mL of this solution contains 0.1 mg of arsenic.

3.19 Cadmium standard stock solution: Weigh 0.1000 g of metallic cadmium ($w_{Cd} \geq$ 99.95%); place it in a 500 mL beaker; add 20 mL of nitric acid (3.3); cover with a watch glass; dissolve completely at low temperature; cool and transfer to a 1000 mL volumetric flask; use water to dilute it to the mark; mix it. 1 mL of this solution contains 0.1 mg of cadmium.

3.20 Copper, cobalt, manganese mixed standard solution: Pipette 20.00 mL of copper, cobalt, manganese standard stock solutions (3.9 ~ 3.11), respectively, into a 200 mL volumetric flask; add 10 mL of hydrochloric acid (3.2); use water to dilute it to the mark; mix it. 1 mL of this solution contains 100 μg of copper, cobalt, manganese, respectively.

3.21 Mixed standard solution of calcium and magnesium: Pipette 20.00 mL of calcium and magnesium standard stock solutions (3.12 and 3.13) into a 200 mL volumetric flask; add 10 mL of hydrochloric acid (3.2); use water to dilute it to the mark; mix well. 1 mL of this solution contains 100 μg of calcium and magnesium, respectively.

3.22 Mixed standard solution of copper, cobalt, manganese, calcium, magnesium: Pipette 10.00 mL of copper, cobalt, manganese mixed standard solution (3.20) and 10.00 mL of calcium and magnesium mixed standard solution (3.21), into a 100 mL volumetric flask; add 5 mL of hydrochloric acid (3.2); use water to dilute it to the mark; mix well. 1 mL of this solution contains 10 μg of copper, cobalt, manganese, calcium, magnesium, respectively.

3.23 Mixed standard solution of zinc, iron, aluminum: Pipette 20.00 mL of zinc, iron, aluminum standard stock solutions (3.14 ~ 3.16) into a 200 mL volumetric flask; add 10 mL of hydrochloric acid (3.2); dilute to the mark with water; mix it. 1 mL of this solution contains 10 μg of zinc, iron, aluminum, respectively.

3.24 Mixed standard solution of lead and arsenic: Pipette 20.00 mL of lead and arsenic standard stock solutions (3.17 and 3.18) into a 200 mL volumetric flask; add 10 mL of hydrochloric acid (3.2); dilute to the mark with water; mix it. 1 mL of this solution contains 10 μg of lead and arsenic, respectively.

3.25 Cadmium standard solution: Pipette 2.00 mL of cadmium standard stock solution (3.19) into a 200 mL volumetric flask; add 10 mL of hydrochloric acid (3.2); use water to dilute it to the mark; mix it. 1 mL of this solution contains 1 μg of cadmium.

4 Instrument

Inductively coupled plasma atomic emission spectrometer

- Under the best working conditions of the instrument, the relative standard deviation of the light intensity of each element does not exceed 2.5% when the standard solution with the lowest concentration (not the "zero" concentration standard solution) is used for 11 measurements.

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