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YS

**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 252.4-2007

**Methods for chemical analysis of nickel matte. Determination of
copper content. Sodium thiosulphate titrimetric method**

高镍钨化学分析方法铜量的测定硫代硫酸钠滴定法

(English Translation)

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Foreword

YS/T 252 "Methods for chemical analysis of nickel matte" is divided into five parts:

YS/T 252.1 Methods for chemical analysis of nickel matte - Determination of nickel content - Dimethylglyoxime gravimetric method
 YS/T 252.2 Methods for chemical analysis of nickel matte - Determination of iron content - Sulfosalicylic acid photometric method
 YS/T 252.3 Methods for chemical analysis of nickel matte - Determination of cobalt content - Flame atomic absorption spectrometric method
 YS/T 252.4 Methods for chemical analysis of nickel matte - Determination of cobalt content - Sodium thiosulphate titrimetric method
 YS/T 252.5 Methods for chemical analysis of nickel matte - Determination of sulfur content - Combustion-neutralization titrimetric method
 this Part is Part 4.

This Part replaces YS/T 252.4-1994 "Determination of copper content by sodium thiosulphate titrimetric method ". Compared with YS/T 252.4-1994, this Part mainly has the following changes:

- ADJUST the measurement range from 6.00% ~ 30.00% to 6% ~ 55%;
- ADJUST the sample amount from 0.200 g to 0.400 g;
- ADJUST the concentration of sodium thiosulfate standard solution from 0.02 mol/L to 0.01 mol/L;
- ADD the repeatability limits, supplementing quality assurance and control terms.

This Part was proposed by AND shall be under the jurisdiction of the National Technical Committee for Standardization of Nonferrous Metals.

Responsible drafting organization of this Part: Jinchuan Group Co., Ltd.

Participated drafting organization of this Part: Guangzhou Nonferrous Metals Research

Institute, Beijing General Research Institute of Mining and Metallurgy.

1 Scope

This standard specifies the method for determination of copper content in high nickel matte.

This standard is applicable to the determination of copper content in high nickel matte.

Measuring range: 6% ~ 55%.

2 Method summary

The sample is decomposed with hydrochloric acid and nitric acid-potassium chlorate saturated solution. The ammonium bifluoride is used to mask the iron and the solution is controlled at pH3.5 ~ pH4. Potassium iodide is added, to cause the copper to precipitate copper iodide and precipitate an equal amount of iodine. Use the starch solution as an indicator; use sodium thiosulfate standard titration solution to indirectly determine the amount of copper.

3 Reagents

3.1

Ammonium bifluoride.

3.2

Potassium iodide.

3.3

Ammonia water (ρ 0.90 g/mL).

3.4

Hydrochloric acid (ρ 1.19 g/mL).

3.5

Nitric acid (ρ 1.42 g/mL).

3.6

Nitric acid (1 + 1).

3.7

Sulfuric acid (1 + 1).

3.8

Urea saturated solution.

3.9

Potassium sulfate solution (100 g/L).

3.10

Nitric acid-potassium chlorate saturated solution

Dissolve potassium chlorate in nitric acid (3.5) until saturated.

3.11

Starch solution (5 g/L).

3.12 Copper standard solution: Accurately weigh 1.0000 g of metallic copper (mass

fraction of copper $\geq 99.95\%$). Place it in a 400 mL beaker. Add 20 mL of nitric acid (3.6). Dissolve at low temperature and evaporate to a viscous state. Cool it. Add 5 mL of sulfuric acid (3.7). Heat until a lot of white smoke comes out. Cool it slightly. Add water to boil to dissolve the salts. Cool to room temperature. Pipette it into a 1000 mL volumetric flask. Use water to make the volume reach to the mark. 1 mL of this solution contains 1.0 mg of copper.

3.13 Sodium thiosulfate standard titration solution [$c(\text{Na}_2\text{S}_2\text{O}_3) \sim 0.01 \text{ mol/L}$]:

3.13.1

Preparation

Weigh 2.5 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$). Dissolve it in newly boiled water and cool it to room temperature. Add 0.1 g of anhydrous sodium carbonate. Dissolve it and transfer it to a 1000 mL brown bottle. Use water to make the volume reach to the mark. Calibrate after standing for one week.

3.13.2 Calibration: Pipette 25.00 mL of copper standard solution (3.12) into a 300 mL

triangular beaker. Add ammonia water (3.3) dropwise, until it turns dark blue. Proceed the subsequent steps according to 4.3.3.

Calculate the titration coefficient of sodium thiosulfate standard titration solution for copper, according to formula (1):

Where:

F_{Cu} - Titration coefficient, the unit volume of sodium thiosulfate standard titration solution is equivalent to the mass of copper, in milligrams per milliliter (mg/mL);

ρ - The mass concentration of the copper standard solution, in milligrams per milliliter (mg/mL);

V1 - The volume of the copper standard solution dispensed, in milliliters (mL);

V2 - The volume of the sodium thiosulfate standard titration solution consumed when titrating the copper standard solution, in milliliters (mL).

Take the average of four results. The difference in the volume of the standard titration solution, which is consumed during the four calibrations, shall not be greater than 0.10 mL; otherwise, the calibration will be re-calibrated.

Note: The sodium thiosulfate standard titration solution must be recalibrated every one week.