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

YS/T 252.5-2007

**Methods for chemical analysis of nickel matte. Determination of
sulfur content. Combustion-neutralization titrimetric method**

高镍钨化学分析方法硫量的测定燃烧-中和滴定法

(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 5.

This Part replaces YS/T 252.5-1994 "Determination of sulfur content by sodium thiosulphate titrimetric method ". Compared with YS/T 252.5-1994, this Part mainly has the following changes:

- CHANGE the analysis method from barium sulfate gravimetric method to

combustion-neutralization titration method;

- ADD 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: Beijing General Research Institute of Mining and Metallurgy, Daye Nonferrous Metals Co., Ltd.

1 Scope

This Part specifies the method for determination of sulfur content in high nickel matte.

This Part is applicable to the determination of sulfur content in high nickel matte.

Measuring range: 15% ~ 25%.

2 Method summary

The sample uses copper wire as flux and burns in air flow at 1250 °C ~ 1300 °C, to convert sulfur into sulfur dioxide. It is absorbed and oxidized into sulfuric acid by hydrogen peroxide. Methyl red-methylene blue solution is used as a mixed indicator. Use sodium hydroxide standard titration solution to make titration, until the solution changes from purple to bright green, which is taken as the end point.

3.2 Acid absorption solution (potassium dichromate-sulfuric acid solution): Heat and

dissolve 5 g of potassium dichromate with a small amount of water. After cooling, add

3.6 Methyl red-methylene blue mixed indicator: Weigh 0.3 g of methyl red and 0.25 g

of methylene blue. Dissolve in 500 mL of absolute ethanol.

3.7 Hydrogen peroxide absorption solution (prepared when used): Take 50 mL of hydrogen peroxide. Use water to dilute it to 1 L. Add 10 mL of mixed indicator (3.6). Use sodium hydroxide solution (3.8) to make adjustment, until bright green appears.

3.8.1 Preparation: Prepare sodium hydroxide into a saturated solution. Place it in a

plastic bottle until the solution is clear. Pipette 32 mL of supernatant. Use carbon dioxide-free water to dilute it to 10 L. Mix well.

3.8.2 Calibration: Weigh four sets of 0.300 g (accurate to 0.0001 g) of potassium

hydrogen phthalate (standard reagent), that has been dried at 100 °C ~ 105 °C for 2 hours. Place it in a 300 mL Erlenmeyer flask. Use 60 mL of hot water, which does not contain carbon dioxide, to dissolve it. After cooling, add 2 drops of phenolphthalein indicator (3.5). Use sodium hydroxide standard titration solution to make titration, until it shows light red, which is taken as end point.

Do a blank test along with the calibration.

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

Where:

FS - Titration coefficient, the mass of sulfur equivalent to 1.00 mL of sodium hydroxide standard titration solution, in milligrams per milliliter (mg/mL);

m1 - The mass of potassium hydrogen phthalate, in grams (g);

V1 - During calibration, the volume of sodium hydroxide standard titration solution consumed in titrating potassium hydrogen phthalate solution, in milliliters (mL);

V0 - During calibration, the volume of sodium hydroxide standard titration solution consumed by titrating the blank test solution, in milliliters (mL);

204.2 - Molar mass of potassium hydrogen phthalate [M(C₈H₅KO₄)], in grams per mole (g/mol);

16.03 - Molar mass of sulfur [M(1/2S)], in grams per mole (g/mol).

Take the average of four results. The range of the four calibration results is not greater than 0.0060 mg/mL; otherwise, the measurement will be repeated.

4.1 Tubular combustion furnace: The maximum temperature 1350 °C, common temperature 1300 °C, equipped with resistance furnace temperature controller (0 °C ~ 1600 °C).

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