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

YS/T 252.1-2007

**Methods for chemical analysis of nickel matte. Determination of
nickel content. Dimethylglyoxime gravimetric method**

高镍钨化学分析方法镍量的测定丁二酮肟重量法

(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 1.

This Part replaces YS/T 252.1-1994 "Determination of nickel content by dimethylglyoxime gravimetric method". Compared with YS/T 252.1-1994, this Part mainly has the following changes:

- ADJUST the measurement range from 45.00% ~ 70.00% to 20% ~ 80%;

- CHANGE the acidity of the solution from pH7 ~ pH8 to pH8 ~ pH9 during precipitation;
- CANCEL Appendix A: Determination of nickel amount by EDTA titration method;
- ADD the determination of nickel content in the filtrate by dimethylglyoxime gravimetric method;
- 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: Beijing General Research Institute of Mining and Metallurgy, Guangzhou Nonferrous Metals Research Institute.

1 Scope

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

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

Measuring range: 20% ~ 80%.

2 Method summary

The sample is decomposed with hydrochloric acid or nitric acid-potassium chlorate saturated solution. In the ammonia medium, nickel and diacetyl oxime form a red nickel diacetyl oxime precipitate, which is separated from other impurity elements. The precipitate is filtered, washed, and dried to constant weight; the amount of nickel is calculated. The amount of nickel in the mixed filtrate is extracted with chloroform under pH6.5, separated from other impurities, measured using diacetyl oxime spectrophotometry and then corrected.

3.9 Nitric acid-potassium chlorate saturated solution: Dissolve potassium chlorate in

nitric acid (3.4) until saturated.

3.14 Dimethylglyoxime-sodium hydroxide solution (10 g/L): Dissolve 15 g of sodium

hydroxide in 100 mL of water. After cooling to room temperature, dissolve 1 g of dimethylglyoxime in the solution. Mix well.

3.16 Nickel standard stock solution:

Weigh 0.1000 g of metallic nickel (the mass fraction of nickel is $\geq 99.95\%$). Place it in a 400 mL beaker. Add 10 mL of nitric acid (3.5). Cover it with a watch glass. Dissolve it completely at low temperature. Add 2 mL of sulfuric acid (3.6). Continue heating until a large amount of white smoke is gone. Cool down. Rinse the watch glass and cup wall with water. Heat to dissolve the salts. Cool to room temperature. Transfer to a 1000 mL volumetric flask. Use water to make the volume reach the mark. 1 mL of this solution contains 100 μg of nickel.

3.17 Nickel standard solution:

Pipette 10.00 mL of nickel standard storage solution (3.16) into a 200 mL volumetric flask. Use water to make the volume reach the mark. 1 mL of this solution contains 5 μg of nickel.

5.4.1 Collect the filtrate from 5.3.3. Cool it. Transfer it into a 500 mL or 1000 mL volumetric flask depending on the volume. Use water to make the volume reach to the mark.

5.4.2 Dispense 100.00 mL of the mixed filtrate (5.4.1) into a 250 mL separatory funnel.

Use hydrochloric acid (3.2) and ammonia water (3.7) to adjust the pH of the solution to about 6.5. Add 15 mL of chloroform (3.8). Shake for 2 minutes. Let it stand for 5 minutes. After layering, move the organic phase into another separatory funnel. Add 15 mL of chloroform (3.8) to the aqueous phase, to repeat the extraction once. Combine the organic phases. Discard the aqueous phase.

5.4.3 Add 15 mL hydrochloric acid (3.3) to the organic phase (5.4.2). Shake for 1 minute.

Let it stand for stratification. Transfer the organic phase into another separatory funnel. Add 15 mL of hydrochloric acid (3.3) for back extraction once. Combine the aqueous phase in a 100 mL beaker.

5.4.4 Heat the solution (5.4.3) in the beaker at low temperature, to evaporate to a volume

of about 2 mL. Remove to cool it. Add 2 mL of nitric acid (3.4). Continue heating and evaporate to dryness. Cool it. Add hydrochloric acid (3.2) and heat slightly to dissolve the salts. After cooling, transfer it into a 50 mL volumetric flask.

5.4.5 Add 5 mL of persulfate lock solution (3.15) and 5 mL of dimethylglyoxime-sodium hydroxide solution (3.14). Mix well. Let it stand for 10 minutes. Use water to make the volume reach to the mark.

5.4.6 Pipette part of the test solution (5.4.5) into a 1 cm cuvette. Use the reagent blank

as a reference, to measure the absorbance at the wavelength of 510 nm on the spectrophotometer. Find out the corresponding nickel amount from the working curve.

5.4.7 Drawing of working curve:

Pipette 0.00 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL, 5.00 mL of nickel standard solutions (3.17). Place them in a set of 50 mL volumetric flasks, respectively. Proceed the following steps in accordance with (5.4.5). Using the reagent blank as a reference, measure the absorbance at the wavelength of 510 nm with a spectrophotometer. Draw a working curve, using the nickel amount as the abscissa and the absorbance as the ordinate.

Note: The nickel content in the filtrate shall not exceed 0.20% of the nickel content in the original sample.