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Methods for chemical analysis of fire smelting nickel substrate material - Part 1: Determination of nickel content - Dimethylglyoxime spectrophotometric method and dimethylglyoxime gravimetric method

火法冶炼镍基体料化学分析方法

第 1 部分：镍量的测定 丁二酮肟分光光度法和丁二酮肟重量法

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Methods for chemical analysis of fire smelting nickel substrate material - Part 1: Determination of nickel content - Dimethylglyoxime spectrophotometric method and dimethylglyoxime gravimetric method

1 Scope

This Part of YS/T 953 specifies the dimethylglyoxime spectrophotometric method and dimethylglyoxime gravimetric method for the determination of nickel content in the fire smelting nickel substrate material.

This Part applies to the determination of nickel content in the fire smelting nickel substrate material. The determination range of Method 1 is 0.50%~5.00%. The determination range of Method 2 is 1.00%~25.00%.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 20066 Steel and iron - Sampling and preparation of samples for the determination of chemical composition

3 Method 1 - Dimethylglyoxime spectrophotometric method

3.1 Method summary

USE hydrochloric acid and nitric acid to dissolve the test portion. USE potassium sodium tartrate as a masking agent; in an alkaline medium of sodium hydroxide, use ammonium persulfate as an oxidant; nickel and dimethylglyoxime form a soluble wine-red complex. At the 460 nm wavelength of the spectrophotometer, measure the absorbance and calculate the nickel content.

industrial standards.

3.4 Analytical procedures

3.4.1 Mass of test portion

WEIGH 0.10 g of sample (3.3), accurate to 0.0001 g.

3.4.2 Number of determinations

Perform two determinations independently and take the average.

3.4.3 Blank test

WEIGH 0.090 g of pure iron (w_{Ni}≤0.005%); do a blank test along with the test portion.

3.4.4 Determination

3.4.4.1 Dissolution of test portion

PLACE the test portion (3.4.1) in a 250 mL polytetrafluoroethylene beaker; ADD 10 mL of hydrochloric acid (3.2.1), 10 mL of nitric acid (3.2.2); HEAT at low temperature to almost no reaction; then add 3~5 drops of hydrofluoric acid (3.2.3) in portions until completely dissolved. ADD 2 mL of perchloric acid (3.2.4); HEAT it to emit perchloric acid fumes; REMOVE it and cool it slightly. TRANSFER the solution to a 200 mL beaker and continue heating until the fumes nearly out. REMOVE it and slightly cool it; ADD 20 mL of hydrochloric acid (3.2.5); HEAT to dissolve the salts and then boil slightly for 5 min. REMOVE and cool to room temperature; TRANSFER to a 200 mL volumetric flask; USE water to dilute to the mark and mix well (if there is residue, use quick filter paper to dry-filter).

3.4.4.2 Measurement

TAKE 10.00 mL of test solution (3.4.4.1) into a 100 mL volumetric flask; ADD in turn 5 mL of potassium sodium tartrate solution (3.2.7), 10 mL of sodium hydroxide solution (3.2.6), 5 mL of ammonium persulfate solution (3.2.8), 5 mL of alkaline dimethylglyoxime solution (3.2.9). Every time a reagent is added, it needs to be mixed. After standing at room temperature for 20 min, use water to dilute to the mark and mix well.

PIPETTE part of the above solution into a 1 cm cuvette; USE the reagent blank (3.4.3) color developing solution made along with the test portion as a reference; at the 460 nm wavelength of the spectrophotometer, measure the absorbance. FIND the corresponding nickel content from the working curve.

filtrate to about 250 mL. While stirring, use aqua ammonia (4.2.5) to adjust the pH value to about 4.5; USE hot water at 60 °C~80 °C to dilute to about 300 mL.

4.4.4.3 Precipitation

While stirring, slowly add dimethylglyoxime solution (4.2.9) to the test solution (4.4.4.2). ADD about 0.4 mL of dimethylglyoxime solution (4.2.9) for every 1 mg of nickel, with an excess of 20 mL (the volume shall not exceed 1/3 of the volume of the test solution, so as not to dissolve part of the precipitate in ethanol). Then, while stirring, slowly use aqua ammonia (4.2.5) to adjust the pH value of the solution to 8~9. After fully stirring, let it stand for 30 min to make the precipitate agglomerate.

4.4.4.4 Filter, dry, weigh

TRANSFER the precipitate (4.4.4.3) to a G4 glass sand plate funnel, which was previously dried at 145 °C to a constant weight; USE a vacuum pump for suction filtration; thoroughly wash the beaker. USE cold water to wash the precipitate 6 times (the suction filtration speed should not be too fast; do not let the precipitate be dry).

The crucible and the precipitate are dried in an oven at 145 °C for 2 h; then naturally cooled in a desiccator for 30 min; and then quickly weighed.

4.5 Calculation of analysis results

The nickel content is calculated by the mass fraction w_{Ni} of nickel. The value is expressed in %, calculated according to formula (2):

$$w_{Ni} = \frac{(m_2 - m_1) \times 0.203\ 2}{m} \times 100\% \qquad \cdots\cdots (2)$$

Where:

m_1 - The mass of empty crucible, in grams (g);

m_2 - The total mass of crucible and precipitation of nickel dimethylglyoxime, in grams (g);

m - Mass of test portion weighed, in grams (g);

0.2032 - The conversion factor of nickel dimethylglyoxime to nickel.

The calculation result is kept to two decimal places.

4.6 Precision

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