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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 21933.1-2008

Ferronickel - Determination of nickel content - The dimethylglyoxime gravimetric method

镍铁 镍含量的测定 丁二酮肟重量法

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1 Scope

Warning: persons using this part shall have practical experience of normal laboratory work. This part does not purport to address all of the safety problems that may arise. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with the conditions laid down by the relevant national regulations.

This part of GB/T 21933 specifies the dimethylglyoxime gravimetric method for the determination of the nickel content of ferronickel.

It applies to nickel contents from 15 % to 60 % expressed as a mass fraction.

2 Normative references

The following documents become provisions of this part of GB/T 21933 through reference in the text. For dated references, subsequent amendments, excluding corrections, or revisions do not apply; the parties to agreements based on this part are nevertheless encouraged to investigate the possibility of applying the most recent editions. For undated references, the latest edition applies.

GB/T 12805 Laboratory glassware - Burettes · GB/T 12806 Laboratory glassware - One-mark volumetric flasks · GB/T 12808 Laboratory glassware - One-mark pipettes · GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions (GB/T 6379.1-2004, ISO 5725-1:1994, IDT) · GB/T 6379.2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method (GB/T 6379.2-2004, ISO 5725-2:1994, IDT) · GB/T 11415-1989 Laboratory sintered (porous) filters - Porosity grading, classification and designation

3 Principle

The test portion is decomposed with nitric acid and the silica is dehydrated with perchloric acid and filtered off. In an ammoniacal tartrate medium the nickel is precipitated with an alcoholic solution of dimethylglyoxime. After a double precipitation the precipitate is dried at 150 degrees Celsius and weighed, and the nickel remaining in the filtrate is determined by atomic absorption spectrometry.

4 Reagents and materials

Unless otherwise stated, only reagents confirmed to be of analytical grade and distilled water, or water of equivalent purity, are used.

4.1 Hydrochloric acid, density 1.19 g/mL. 4.2 Ammonia solution, density 0.925 g/mL. 4.3 Nitric acid, density 1.42 g/mL. 4.4 Perchloric acid, density 1.67 g/mL. 4.5 Hydrofluoric acid, density 1.13 g/mL. 4.6 Acetic acid, density 1.05 g/mL.

4.7 Hydrochloric acid (1+1). 4.8 Hydrochloric acid (1+9). 4.9 Nitric acid (1+1). 4.10 Acetic acid (1+1). 4.11 Hydrofluoric acid (1+1). 4.12 Dimethylglyoxime, 10 g/L in ethanol. 4.13 Tartaric acid solution, 500 g/L.

5 Apparatus

Ordinary laboratory glassware and the following.

5.1 Filtering crucible of sintered porous glass with a pore size of 10 micrometres to 20 micrometres, of grade P16 as specified in GB/T 11415-1989, or a commercial G3 or G4 crucible. 5.2 Glass beaker of 600 mL. 5.3 Pipettes of 50 mL and 100 mL, class A to GB/T 12808. 5.4 Volumetric flasks of 200 mL and 1 000 mL, class A to GB/T 12806. 5.5 Polytetrafluoroethylene beaker of 600 mL, for samples of high silicon content.

6 Sampling and sample preparation

6.1 The laboratory sample is taken and prepared by an agreed procedure; in case of dispute the relevant national standards apply.

6.2 The laboratory sample is normally in the form of granules, drillings or millings and needs no further preparation. 6.3 If the sample has been contaminated with grease during grinding or drilling it is washed with analytical grade acetone and dried in air. 6.4 If the particle size of the laboratory sample varies widely, the test portion is taken after reduction.

7 Procedure

Warning 1: perchloric acid fumes are a strong oxidising agent and may cause an explosion on contact with organic material. All fuming operations shall be carried out in a fume cupboard suitable for the use of perchloric acid. Warning 2: hydrofluoric acid is a strongly corrosive acid, severely irritant to the skin and capable of causing burns that heal with difficulty. On contact with the skin, wash immediately with water and seek medical treatment.

7.1 Test portion. Weigh 4.0 g of the sample to the nearest 0.001 g. 7.2 Blank test. A blank test is carried out with the test portion.

7.3 Preparation of the crucible. A hot mixture of 20 mL of hydrochloric acid, 10 mL of nitric acid and 30 mL of water is filtered through the crucible, which is then washed free of acid with warm water, dried in an oven at 150 degrees Celsius for two hours, cooled in a

desiccator for 60 minutes and weighed rapidly.

7.4 Preparation of the test solution. The test portion is placed in a 600 mL beaker with 25 mL of water and 50 mL of nitric acid (1+1), covered and dissolved at low temperature as completely as possible. Where the silicon content of the ferronickel exceeds 1 % by mass, a polytetrafluoroethylene beaker is used and the sample is treated with hydrofluoric and perchloric acid. Forty millilitres of perchloric acid are then added, the solution is heated to dense perchloric fumes and refluxed for 20 minutes; the silica is filtered off and the filtrate collected in a 1 000 mL flask.

7.5 Determination. An aliquot containing about 60 mg to 120 mg of nickel is taken - 100 mL where the nickel content is below 30 %, 50 mL where it is above - and diluted to 300 mL. Ten millilitres of tartaric acid solution are added and ammonia is added with stirring until the colour turns from yellow to blue-green; acetic acid is then added slowly to bring the pH between 4 and 5 and the solution is heated to 60 degrees Celsius. Dimethylglyoxime is added with stirring, about 4 mL for every 10 mg of nickel with an excess of 20 mL, then ammonia to a pH of about 10; the mixture is stirred for 30 seconds and left to stand for 30 minutes.

The precipitate is filtered on medium-speed paper and washed five times with warm water at 40 to 50 degrees Celsius, the filtrate being retained. The first precipitate is redissolved and the nickel is precipitated a second time with 2 mL of tartaric acid solution and an excess of 5 mL of dimethylglyoxime. The precipitate is collected in the weighed crucible, dried at 150 degrees Celsius for two hours, cooled for 60 minutes and weighed under the same conditions as the empty crucible. The combined filtrates are evaporated, made up to 200 mL and the residual nickel is determined by atomic absorption spectrometry in accordance with Annex A; that residue shall not exceed 0.2 % by mass of the nickel in the original sample.

8 Expression of results

8.1 Calculation. The nickel content as a mass fraction is calculated by formula (1) from the mass of the test portion, the mass of the empty crucible, the mass of the crucible with the nickel dimethylglyoxime precipitate, the aliquot ratio of the test solution and the correction for the nickel found in the combined filtrate; the factor 20.32 is one hundred times the factor for converting nickel dimethylglyoxime to nickel.

8.2 Precision. The method was tested internationally in 7 countries by 20 laboratories and 39 analysts on 8 samples with nickel contents between 21 % and 41 %, each laboratory providing the results of two independent analysts.

The statistical results of Table 1, calculated in accordance with GB/T 6379, give for nickel between 15 % and 30 % a repeatability r of 0.13, a within-laboratory reproducibility of 0.19 and a between-laboratory reproducibility R of 0.24; for nickel between 31 % and 45 %, 0.19,

0.33 and 0.35 respectively. The report and the statistical analysis of the interlaboratory test are given in Annex B.

9 Test report

The test report shall include the identification of the test sample, of the laboratory and of the date of analysis; the degree of compliance with this part; the analytical result and how it is expressed; any anomaly observed during the determination; and any operation not included in this part, or optional, that may have influenced the result.