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Replacing GB/T 223.23~223.24-1994

Iron, steel and alloy - Determination of nickel content - The dimethylglyoxime spectrophotometric method

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Issued by: General Administration of Quality Supervision, Inspection and Quarantine;

Standardization Administration Committee.

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Foreword

This Part of GB/T 223 is the integral revision of GB/T 223.23-1994 "Methods for Chemical Analysis of Iron, Steel and Alloy - The Dimethylglyoxime Spectrophotometric Method for the Determination of Nickel Content" and GB/T 223.24-1994 "Methods for Chemical Analysis of Iron, Steel and Alloy - The Extraction Separation - The Dimethylglyoxime Spectrophotometric Method for the Determination of Nickel Content".

This Part replaced GB/T 223.23-1994 and GB/T 223.24-1994.

Compared with GB/T 223.23-1994 and GB/T 223.24-1994, the main changes of this Part are listed as follows:

- modified the name to "Iron, Steel and Alloy - Determination of Nickel Content - The Dimethylglyoxime Spectrophotometric Method";
- merged the previous two standards into one standard, including two analysis methods;
- added the explanation content for reagents and water in the analysis, and revised the expression method for solution concentration;
- added the expression for safety notice and specimen quantity weighing;
- revised the result calculation formulae and quantity units in formulae;
- revised and specified the explanation for precision function formula.

Appendix A of this Part is informative.

This Part was proposed by China Iron and Steel Association.

This Part shall be under the jurisdiction of National Technical Committee on Iron and Steel of Standardization Administration of China.

The drafting organizations of this Part: Baoshan Iron & Steel Co., Ltd. Special Steel Branch, China Iron & Steel Research Institute Group and Tianjin Special Steel Factory.

Main drafters of this Part: Wang Yujuan, Cui Qiuhong and Guo Yunshan.

Versions of standard substituted by this Standard are:

- GB 223.23-1982, GB 223.23-1994;
- GB 223.24-1982, GB 223.24-1994.

Iron, steel and alloy - Determination of nickel content - The dimethylglyoxime spectrophotometric method

WARNING: the personal using this Part shall have the practical experience of regular laboratory working. This Part does not point out all possible safety problems. The user is responsible to adopt the proper safety and health measures and ensure the condition meeting the requirements of the national relevant regulations.

1 Scope

This Part of GB/T 223 specifies the determination of nickel content with dimethylglyoxime direct photometric method and extraction separation - dimethylglyoxime spectrophotometric method.

The method one of this Part is applicable to the determination of nickel content with mass fraction 0.030% ~ 2.00% in pig iron, iron powder, carbon steel and alloy steel; the method two of this Part is applicable to the determination of nickel content with mass fraction 0.010% ~ 0.50% in pig iron, carbon steel, alloy steel and precious alloy.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Part of GB/T 223. For dated reference, subsequent amendments to (excluding correction to), or revisions of, any of these publications do not apply. However, all parties coming to an agreement according to this standard are encouraged to study whether the latest edition of these documents is applicable. For undated references, the latest edition of the normative document is applicable to this Part.

GB/T 6379.1, *Accuracy (Trueness and Precision) of Measurement Methods and Results - Part 1: General Principles and Definitions*

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 20066, *Steel and Iron-Sampling and Preparation of Samples for the Determination of Chemical Composition*

3 Method One -- Diacetyldioxime Directly Photometric Method

3.1 Principles

Dissolve sample by acid, perchloric acid smokes and oxidize chromium to hexavalence, screen iron with sodium tartrate; in the strongly alkaline medium, take ammonium persulfate as the oxidizer; nickel and dimethylglyoxime generate red complex compound, and measure the absorbance.

The manganese content larger than 1.5 mg, copper content larger than 0.2mg and cobalt content larger than 0.1 mg in the color reagent may interfere with the determination.

3.2 Reagents and materials

Unless otherwise specified, during the analysis, only confirmed analytical reagent and distilled water or purity-equivalent water may be used.

3.2.1 Ethanol, more than 95% (volume fraction).

3.2.2 Perchloric acid with ρ 1.67 g/mL.

3.2.3 Nitric acid with ρ 1.42 g/mL, and dilute to 2+3.

3.2.4 Mixed acid of hydrochloric acid-nitric acid; mix one hydrochloric acid (ρ is about 1.19g/mL), one nitric acid (ρ is about 1.42g/mL) and two waters.

3.2.5 Sodium tartrate solution, 300 g/L.

3.2.6 Sodium hydroxide solution, 100 g/L.

3.2.7 Dimethylglyoxime solution, 10 g/L, prepare with ethanol (3.2.1).

3.2.8 Ammonium persulfate solution, 40 g/L.

3.2.9 Nickel standard solution

3.2.9.1 Nickel stock solution, 100 μg/mL. Weigh 0.1000 g of pure nickel (mass fraction above 99.99%) into 150-mL conical flask, add 20 mL of nitric acid (3.2.3), cool to room temperature after heating and dissolution, transfer into 1000-mL volumetric flask, dilute with water to the scale, and mix well.

3.2.9.2 Nickel standard solution, 10.0 μg/mL. Shift 25.00 mL of nickel stock solution (3.2.9.1) into 250-mL volumetric flask, add 5 mL of nitric acid (3.2.3), dilute with water to the scale, and mix well.

4.2.1 Trichloromethane.

4.2.2 Ethanol, more than 95% (volume fraction).

4.2.3 Perchloric acid with ρ 1.67g/mL.

4.2.4 Ammonia water with ρ 0.90g/mL.

4.2.5 Ammonia water with ρ 0.90g/mL, and dilute to 1+30.

4.2.6 Nitric acid with ρ 1.42g/mL, and dilute to 2+3.

4.2.7 Nitric acid with ρ 1.42g/mL, and dilute to 1+20.

4.2.8 Mixed acid of hydrochloric acid-nitric acid: mix one hydrochloric acid (ρ is about 1.19 g/mL), one nitric acid (ρ is about 1.42 g/mL) and two waters.

4.2.9 Ammonium citrate solution, 200 g/L.

4.2.10 Bromothymol blue solution, 1 g/L. Weigh 0.1 g of bromothymol blue, add 1 mL of sodium hydroxide solution (4.2.13) and 50 mL of water to dissolve, and then dilute with water to 100 mL, mix well.

4.2.11 Dimethylglyoxime solution, 10 g/L. Prepare with ethanol (4.2.2).

4.2.12 Sodium tartrate solution, 300 g/L.

4.2.13 Sodium hydroxide solution, 100 g/L.

4.2.14 Ammonium persulfate solution, 40 g/L.

4.2.15 Nickel standard solution

4.2.15.1 Nickel stock solution, 100 μg/mL. Weigh 0.1000 g of pure nickel (mass fraction above 99.99%) into 150-mL conical flask, add 20 mL of nitric acid (4.2.6), cool to room temperature after heating and dissolution, transfer into 1000-mL volumetric flask, dilute with water to the scale, and mix well.

4.2.15.2 Nickel standard solution, 10.0 μg/mL. Shift 50.00 mL of nickel standard solution (4.2.15.1) into 500-mL volumetric flask, add 10 mL of nitric acid (4.2.6), dilute with water to the scale, and mix well.

4.3 Instruments and equipment

In the analysis, only the general laboratory instruments and equipment as well as the spectrophotometer are used.

4.4 Sampling and sample preparation

original conical flask.

4.5.3.6 Evaporate the water phase to about volume 5mL, and transfer into 50-mL volumetric flask after cooling.

4.5.3.7 Add 2 mL of Sodium tartrate solution (4.2.12), 5 mL of sodium hydroxide solution (4.2.13), 2 mL of dimethylglyoxime solution (4.2.11) and 5 mL of ammonium persulfate solution (4.2.14), mix well after adding one reagent, dilute with water to the scale, and mix well.

4.5.3.8 After placing for 15min, transfer partial solution into 2-cm absorption vessel, measure the absorbance at wave length 540nm on the spectrophotometer taking the water as the reference, subduct the absorbance of blank solution, search out the corresponding nickel mass (μg) from the calibration curve.

4.5.4 Drawing of calibration curve

Respectively transfer 0, 2.00 mL, 4.00 mL, 6.00 mL, 8.00 mL and 10.00 mL of nickel standard solution (4.2.15.2) into 50-mL volumetric flask, carry out according to 4.5.3.7 and 4.5.3.8 and measure the absorbance. Subduct the absorbance of reagent blank. Draw the calibration curve taking the nickel mass as the lateral coordinate and the absorbance as the longitudinal coordinate.

4.6 Result calculation

The nickel content shall be calculated by mass fraction w_{Ni}; the value shall be expressed with % and shall be calculated according to Formula (2):

$$w_{Ni} = \frac{m_1 \times V}{m \times V_1 \times 10^6} \times 100 \qquad \cdots\cdots(2)$$

where,

V_1 - the value respectively adopting the test solution volume, in milliliters (mL);

V - the value of total test solution volume, in milliliters (mL);

m_1 - the value of nickel mass obtained from the calibration curve, in micrograms (μg);

m - the value of specimen mass, in grams (g).

4.7 Precision

The precision test of this Part is carried out for 6-level nickel content in 8

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