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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 223.23-2026

**Iron, steel and alloy-Determination of nickel
content-Spectrophotometric method and gravimetric method**

钢铁及合金 镍含量的测定 分光光度法和重量法

(English Translation)

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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.

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.

3.2.9.1 Nickel stock solution, 100 μ g/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 μ g/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.

3.3 Instruments and equipment

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

3.4 Sampling and sample preparation

The sampling and sample preparation shall be carried out according to GB/T 20066 or proper national standards.

3.5 Analysis procedures

Warning. when generally existing ammonia, nitrous acid smog or organic substance, the emission of perchloric acid smoke may cause explosion.

3.5.1 Specimen quantity

Weigh the sample by Table 1 according to the nickel content (mass fraction) to the accuracy of 0.0001 g.

Table 1

Nickel content (mass fraction) / %	Specimen quantity / g
0.03 ~ 0.10	0.50
>0.10 ~ 0.50	0.20
>0.50 ~ 2.00	0.10

3.5.2 Blank test

Carry out the blank test together with the specimen.

3.5.3.1 Place the specimen (3.5.1) into 150-mL conical flask, add

5mL~10mL of nitric acid (3.2.3) or hydrochloric acid-nitric acid mixed acid (3.2.4), add 3mL~5mL of perchloric acid (3.2.2) after heating and dissolution, evaporate to emit perchloric acid smoke and oxidize chromium to hexavalence, slightly cool.

3.5.3.2 Add a small amount of water to dissolve salt, transfer into 100-mL

volumetric flask after cooling (transfer into 50-mL volumetric flask when the mass fraction of nickel is 0.03%~0.10%), dilute with water to the scale, and mix well. The sediment shall be filtered.

Take two parts of 10.00 mL of (take 5.00 mL when the mass fraction of nickel is 1.00%~2.00%) test solutions and place them into 50-mL volumetric flask respectively according to 3.5.3.2.1 and 3.5.3.2.2.

3.5.3.2.1 Color reagent. add 10 mL of sodium tartrate solution (3.2.5), 10

mL of sodium hydroxide solution (3.2.6), 2 mL of dimethylglyoxime solution (3.2.7) and 5 mL of ammonium persulfate solution (3.2.8), mix well after adding one reagent, dilute with water to the scale, and mix well.

3.5.3.2.2 Reference solution. add 10 mL of sodium tartrate solution (3.2.5), 10 mL of sodium hydroxide solution (3.2.6), 2 mL of ethanol (3.2.1) and 5 mL of ammonium persulfate solution (3.2.8), dilute with water to the scale, and mix well.