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

GB/T 30072-2025

Replacing GB/T 30072-2013

**Ferronickel - Determination of nickel content - EDTA titrimetric
method**

镍铁 镍含量的测定 EDTA滴定法

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part 1: Structure and Drafting Rules of Standardization Documents". Drafting.

This document replaces GB/T 30072-2013 "Determination of Nickel Content in Iron by EDTA Titration Method" and is consistent with GB/T 30072-2013.

Aside from structural adjustments and editorial changes, the main technical changes are as follows.

- a) The measurement range has been changed (see Chapter 1, Chapter 1 of the 2013 edition);
- b) The conditions for the ammonium fluoride reagent have been changed (see 5.9, 4.1 of the 2013 edition);
- c) The conditions for the hydroxylamine hydrochloride reagent have been changed (see 5.10, 4.2 of the 2013 edition);
- d) The preparation of EDTA standard solutions has been modified (see 5.16, 4.13 in the 2013 edition);
- e) The sample quantity has been changed (see 8.1, 7.1 in the 2013 edition);

- f) The recommended amount of EDTA standard solution has been increased (see 8.3);
- g) The determination and representation of analytical results have been improved (see 9.2);
- h) The precision has been changed (see Chapter 10, Chapter 9 in the 2013 edition);
- i) A flowchart of the test results acceptance procedure has been added (see Appendix A).

Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents.

This document was proposed by the China Iron and Steel Association.

This document is under the jurisdiction of the National Technical Committee on Standardization of Pig Iron and Ferroalloys (SAC/TC318).

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This document was first published in 2013, and this is its first revision.

Determination of nickel content in iron by EDTA titration Warning---Personnel using this document should have practical experience working in a formal laboratory. This document does not address all possible safety issues.

Users are responsible for taking appropriate safety and health protection measures and complying with the conditions stipulated in relevant national regulations.

1 Scope

This document specifies the method for determining the nickel content in ferronickel using EDTA titration.

This document applies to the determination of nickel content in ferronickel, with a determination range (mass fraction) of 5.00% to 92.00%.

2 Normative references

GB/T 6379.1

GB/T 6379.2

GB/T 6682

GB/T 8170

GB/T 12806

GB/T 12808

GB/T 24585

GB/T 25050

GB/T 25051

3 Terms and Definitions

This document does not contain any terms or definitions that need to be defined.

4 Principles

The sample was prepared with nitric acid-hydrochloric acid; for samples with high silicon content, hydrofluoric acid was added as a dissolving agent. Perchloric acid caused fuming and decomposition. Fluoride was used as a masking agent in a slightly acidic solution.

Iron, aluminum, titanium, and sodium hexametaphosphate mask manganese. Excess EDTA is added in an acetate-sodium acetate buffer solution at pH 4.6, with PAN...

As an indicator, excess EDTA is titrated with copper standard titration solution, and the amount of nickel in the sample is calculated based on the amount of copper standard titration solution consumed.

The total amount of copper and cobalt was used to subtract the amount of copper and cobalt using a mathematical correction method to calculate the mass fraction of nickel in the sample.

5 Reagents

Unless otherwise specified, only reagents of analytical purity or higher shall be used in the analysis, and the test water shall be of the standard grade specified in GB/T 6682.

Distilled water of grade 1 or 3 or higher, or water of equivalent purity.

5.1 Nitric acid, $\rho \sim 1.42 \text{ g/mL}$.

5.2 Hydrochloric acid, $\rho \sim 1.19 \text{ g/mL}$.

5.3 Sulfuric acid, $\rho \sim 1.84 \text{ g/mL}$.

5.4 Perchloric acid, $\rho \sim 1.67 \text{ g/mL}$.

5.5 Hydrofluoric acid, $\rho \sim 1.15 \text{ g/mL}$.

5.6 Ammonia water, $\rho \sim 0.90 \text{ g/mL}$.

5.7 Glacial acetic acid, $\rho \sim 1.05 \text{ g/mL}$. 5.8 Hydrochloric acid, 1 L.

5.9 Ammonium fluoride, 250 g/L. Weigh 125 g of ammonium fluoride (NH_4F) into a 600 mL plastic beaker, add 300 mL of water, and add 3 drops of...

Add 5 drops of hydrochloric acid (5.2), stir until completely dissolved, then dilute with water to 500 mL and mix well. Prepare and use immediately.

5.10 Hydroxylamine hydrochloride, 100 g/L. Weigh 10 g of hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) into a 200 mL beaker, add 50 mL of water, and stir.

After stirring until completely dissolved, dilute with water to 100 mL and mix well. Prepare and use immediately.

5.11 Acetic acid-sodium acetate buffer solution, $\text{pH}=4.6$. Weigh 144 g of anhydrous sodium acetate (CH_3COONa) and dissolve it in 500 mL of water.

Add 115 mL of glacial acetic acid (5.7) to the solution, dilute with water to 1 L, and mix well.

5.12 Sodium hexametaphosphate solution, 250 g/L. Weigh 25 g of sodium hexametaphosphate [$(\text{NaPO}_3)_6$] into a 200 mL beaker, add 50 mL of...

Add water, stir until completely dissolved, then dilute with water to 100 mL and mix well.

5.13 Nickel standard solution, 1.000 mg/mL. Weigh 1.0000 g of pure nickel ($\geq 99.99\%$) into a 400 mL beaker, add 30 mL of...

Nitric acid (5.1) was completely dissolved by heating at low temperature, then boiled to remove nitrogen oxides and evaporated to a small volume. It was then removed and allowed to cool slightly. The salts were dissolved in water and cooled to room temperature.

Transfer the solution to a 1000 mL volumetric flask, dilute to the mark with water, and mix well. 1 mL of this solution contains 1.000 mg of nickel. Alternatively, commercially available certified solutions may be used. Standard solution.

5.14 PAN indicator, PAN[1-(2-pyridinylazo)-2-naphthol] ethanol solution, 2.0 g/L.

5.15 Copper sulfate standard titration solution, 0.010 mol/L.