GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 77.080.01

CCS H 11

GB/T 223.54-2022

Replacing GB/T 223.54-1987

Iron, Steel and Alloy - Determination of Nickel Content - Flame Atomic Absorption Spectrometric Method

钢铁及合金 镍含量的测定

火焰原子吸收光谱法

(ISO 4940-1985, Steel and Cast Iron - Determination of Nickel Content - Flame Atomic Absorption Spectrometric Method, MOD)

Issued on: July 11, 2022 **Implemented on: February 1, 2023**

Issued by: State Administration for Market Regulation;

Standardization Administration of the People's Republic of China.

Table of Contents

Foreword

This document was drafted in accordance with the rules provided in GB/T 1.1-2020 *Directives for Standardization - Part 1: Rules for the Structure and Drafting of Standardizing Documents*.

This document is Part 54 of GB/T 223. The parts of GB/T 223 that have been issued are shown in Appendix A.

This document serves as a replacement of GB/T 223.54-1987 *Methods for Chemical Analysis of Iron, Steel and Alloy - The Flame Atomic Absorption Spectrophotometric Method for the Determination of Nickel Content*. In comparison with GB/T 223.54-1987, apart from structural adjustments and editorial modifications, the main technical changes are as follows:

---The scope of determination is modified (see Chapter 1; the Scope of Version 1987);

---Normative References is added (see Chapter 2);

---The amount of specimen is modified (see 8.1; 4.1 of Version 1987);

---The amount of acid used when the specimen is decomposed is reduced (see 8.3.1; 4.3.1 of Version 1987);

---The preparation of the calibration solution is modified (see 8.3.2; 4.4 of Version 1987);

---The stipulations on the determination of wavelength by the atomic absorption spectrometer are modified (see 8.3.3 and 8.3.4; 4.4.3 of Version 1987);

---The adjustment of the atomic absorption spectrometer is added (see 8.3.3);

---The optimization of the atomic absorption device is added (see 8.3.4);

---Spectral measurement is added (see 8.3.5);

---The drawing of calibration curve is added (see 8.4);

---The calculation formula of nickel content is modified (see 9.1; Chapter 5 of Version 1987);

---The precision requirements of the method are modified (see 9.2; Chapter 6 of Version 1987);

---Appendix B is added.

This document modifies and adopts ISO 4940-1985 *Steel and Cast Iron - Determination of Nickel Content - Flame Atomic Absorption Spectrometric Method*.

In comparison with ISO 4940-1985, this document makes the following structural adjustments:

---The Chapter "Terms and Definitions" is added (see Chapter 3);

---Chapter 4 corresponds to Chapter 3 of ISO 4940-1985;

---Chapter 5 corresponds to Chapter 4 of ISO 4940-1985, in which, 5.1 ~ 5.3, and 5.6 respectively correspond to 4.1 ~ 4.3 and 4.4 of ISO 4940-1985, 5.4 corresponds to 7.3.2 of ISO 4940-1985, and 5.5 is added;

---Chapter 6 corresponds to Chapter 5 of ISO 4940-1985;

---Chapter 7 corresponds to Chapter 6 of ISO 4940-1985;

---Chapter 8 corresponds to Chapter 7 of ISO 4940-1985;

---Chapter 9 corresponds to Chapter 8 of ISO 4940-1985;

---Chapter 10 corresponds to Chapter 9 of ISO 4940-1985;

---Appendix A and Appendix B are added, Appendix C corresponds to Appendix A of ISO 4940-1985, and Appendix D corresponds to Appendix B of ISO 4940-1985.

The technical differences between this document and ISO 4940-1985 are marked with a vertical single line (|) in the outer margin of the clauses involved. These technical differences and the reasons for these differences are as follows:

---The scope of determination is expanded (see Chapter 1), so as to enhance the applicability of this document;

---Normative References is modified (see Chapter 2), so as to adapt to the technical documents of China;

---Iron solutions of two concentrations are used (see 5.4 and 5.5), so as to adapt to the requirements of different amounts of specimen;

---The detection limit (see 6.1.3) is modified, so as to be compatible with the determination range of this document;

---The amount of specimen is modified (see 8.1), so as to simplify the operating process;

---The amount of acid used when the specimen is decomposed is reduced (see 8.3.1), so as to satisfy the demands of environmental protection;

---The preparation of the calibration solution is modified (see 8.3.2.3), so as to satisfy the demands of the expansion of the detection range;

---The description of instrument optimization in the high concentration section is added (see 8.3.4), so as to satisfy the accuracy requirements for the measurement;

Iron, Steel and Alloy - Determination of Nickel Content - Flame Atomic Absorption Spectrometric Method

WARNING---the personnel using this document shall have practical experience in formal laboratory work. This document does not point out all possible safety issues. It is the user's responsibility to take appropriate safety and health measures and ensure the compliance with the conditions stipulated in the relevant national laws and regulations.

1 Scope

This document specifies the method for the determination of nickel content in steel and cast iron by the flame atomic absorption spectrometric method.

This document is applicable to the determination of nickel content with a mass fraction of 0.002% ~ 5.0%.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

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 12805 Laboratory Glassware - Burettes (GB/T 12805-2011, ISO 385:2005, NEQ)

GB/T 12806 Laboratory Glassware - One-mark Volumetric Flasks (GB/T 12806-2011, ISO 1042:1998, NEQ)

GB/T 12808 Laboratory Glassware - One-mark Pipettes

GB/T 20066 Sample and Iron - Sampling and Preparation of Samples for the Determination of Chemical Composition (GB/T 20066-2006, ISO 14284:1996, IDT) min, so that the white perchloric acid fumes maintain a steady reflux on the wall of the beaker. Cool it, add 100 mL of water, and heat to dissolve the salts. After cooling, transfer it to a 250 mL one-mark volumetric flask, use water to dilute to the mark and mix it well.

5.5 Iron Solution, 8 mg/mL

Weigh-take 2 g ± 0.01 g of pure iron (see 5.1), put it into an 800 mL beaker, add 100 mL of hydrochloric acid - nitric acid mixed acid (see 5.2) and use a watch glass to cover it; heat it, until it is completely dissolved. Then, add 80 mL of perchloric acid (ρ about 1.67 g/mL), evaporate it, until white perchloric acid fumes are emitted. Let it continue to emit fumes for 1 min, so that the white perchloric acid fumes maintain a steady reflux on the wall of the beaker. Cool it, add 100 mL of water, and heat to dissolve the salts. After cooling, transfer it to a 250 mL one-mark volumetric flask, use water to dilute to the mark and mix it well.

5.6 Nickel Standard Solution

5.6.1 Nickel stock solution, 1,000 μg/mL

Weigh-take 0.5000 g of pure nickel (with a mass fraction not less than 99.9%), accurate to 0.0001 g, place it in a 250 mL beaker, add 25 mL of nitric acid (ρ about 1.42 g/mL, diluted to 1 + 1). After heating to dissolve it, boil it to remove nitrogen oxides. After cooling to room temperature, transfer it to a 500 mL one-mark volumetric flask, use water to dilute to the mark and mix it well.

1 mL of this stock solution contains 1,000 μg of nickel.

5.6.2 Nickel standard solution, 40 μg/mL

Weigh-take 10.0 mL of the nickel stock solution (5.6.1) and put it in a 250 mL one-mark volumetric flask. Use water to dilute to the mark and mix it well.

1 mL of this standard solution contains 40 μg of nickel.

Prepare it right before use.

6 Instruments and Equipment

6.1 Atomic Absorption Spectrometer

6.1.1 General requirements

Configure a nickel hollow cathode lamp; the supply air and acetylene shall be sufficiently pure, and free of water, oil and nickel, so as to provide a stable and clear lean-burn flame.

After the atomic absorption spectrometer used is optimized in accordance with 8.3.4, the detection limit and characteristic concentration shall be consistent with the parameters provided by the instrument manufacturer and satisfy the indicators in 6.1.2 ~ 6.1.4; the instrument shall also reach the additional performance requirements provided in 6.1.5.

To establish a standard method for flame atomic absorption spectrometry, the working group shall determine the value of the instrument indicators in accordance with the inter-laboratory measurement results.

6.1.2 Lowest precision (in accordance with B.1 of Appendix B)

Use the calibration solution of the highest concentration, measure the absorbance for 10 times and calculate its standard deviation. The standard deviation shall not exceed 1.0% of the mean absorbance.

Use the calibration solution of the lowest concentration (not the zero-concentration calibration solution), measure the absorbance for 10 times and calculate its standard deviation. This standard deviation shall not exceed 0.5% of the mean absorbance of the calibration solution of the highest concentration.

6.1.3 Detection limit (in accordance with B.2)

The detection limit is defined as the concentration corresponding to 4.65 times the standard deviation of the 10 absorbance measurements of the analytical element in solution at a concentration level slightly above the zero-calibration solution.

In a solution similar to the final specimen solution matrix, the detection limit of nickel shall be less than 0.06 μg/mL.

6.1.4 Linearity of calibration curve (in accordance with B.3)

When using the same method for the determination, the ratio of the slope value of the upper 20% concentration range of the calibration curve (expressed as a change in absorbance) to the slope value of the lower 20% concentration range (expressed as a change in absorbance) shall not be less than 0.7.

For instruments that are automatically calibrated with 2 or more standard samples, before the analysis, the obtained absorbance readings shall be used to establish a calibration curve whose linearity satisfies the above-mentioned requirements.

6.1.5 Characteristic concentration (in accordance with B.4)

In a solution consistent with the matrix of the final specimen solution, for a wavelength of 232.0 nm, the characteristic concentration of nickel shall be less than 0.10 μg/mL; for a wavelength of 352.5 nm, the characteristic concentration of nickel shall be less than 0.60 μg/mL.

6.2 Auxiliary Device

It is recommended to use a suitable recorder or digital reading device to evaluate the indicators in 6.1.2 ~ 6.1.4 and to conduct the subsequent measurements.

In accordance with the expected nickel content, in the order of increasing concentration, successively spray the corresponding series of calibration solutions (see 8.3.2.1, 8.3.2.2 or 8.3.2.3) into the flame; measure the absorbance of each calibration solution for 2 ~ 3 times to obtain the average absorbance of each calibration solution. It shall be noted that a constant sample injection rate shall be maintained during the determination of the calibration solution. After each measurement, water shall be sprayed to clean the burner.

Under the same conditions, successively measure the absorbance of the blank test solution and the test solution at least twice to obtain the average absorbance of the blank test solution and the test solution.

During the determination of batches of specimens, at regular intervals, the calibration solution of intermediate concentration shall be sprayed into for inspection. If the results show loss of absorbance or precision due to clogging, the burner shall be cleaned.

Make sure to thoroughly rinse the atomizing system and drain system after using perchloric acid.

8.4 Drawing of Calibration Curve

For each measurement series and range of nickel content to be measured, a new calibration curve shall be drawn.

Before drawing the curve, the concentration of the zero-calibration solution of the calibration curve shall be determined. If the zero-calibration solution has significant absorption, then, in accordance with Formula (1), calculate the nickel concentration ($\rho_{Ni,z}$) in the zero-calibration solution:

$$\rho_{Ni,z} = \rho_{Ni,C1} \times \frac{A_Z}{A_{Ni,C1} - A_Z} \qquad \cdots\cdots(1)$$

Where,

$\rho_{Ni,z}$---the nickel concentration after adding the first calibration solution, expressed in (μg/mL);

A_Z---the absorbance of the zero-calibration solution;

$A_{Ni,C1}$---the absorbance of the first calibration solution.

Add the calibrated nickel concentration ($\rho_{Ni,z}$) in the zero-calibration solution to the concentration of each calibration solution, and make the calibration curve pass through the origin of coordinates.

Take the average absorbance value of each calibration solution to draw against the nickel content (μg/mL) to establish a calibration curve.

Alternatively, the instrument data processing software that comes with the instrument may also be used to subtract the absorbance of the zero-calibration solution from the absorbance of each

------ The End ------