NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 223.63-2022

Iron, steel and alloy - Determination of manganese content - Sodium (potassium) periodate spectrophotometric method

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

Issued by: SAMR; SAC

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1 Scope

This document specifies the method for the determination of manganese content in iron, steel and alloy by the sodium (potassium) periodate spectrophotometric method.

This document is applicable to the determination of manganese content in iron, steel, high-temperature alloy and precision alloy. The range of determination is: 0.001% ~ 4.00%.

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 6379.2 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 7729 Chemical Analysis of Metallurgical Products - General Rule for Spectrophotometric Methods

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 20066 Sample and Iron - Sampling and Preparation of Samples for the Determination of Chemical Composition

3 Terms and Definitions

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

4 Principle

After the specimen is dissolved in acid, in sulfuric acid and phosphoric acid medium, use sodium (potassium) periodate to oxidize manganese to heptavalent; at a wavelength of 530 nm, use a spectrophotometer to measure the absorbance.

5 Reagents or Materials

Unless it is otherwise specified, only reagents confirmed to be analytically pure, as well as distilled water or deionized water (or water of equivalent purity) are used in the analysis.

5.1 Hydrofluoric Acid

rho is about 1.15 g/mL.

5.2 Hydrochloric Acid

rho is about 1.19 g/mL.

5.3 Nitric Acid

rho is about 1.42 g/mL.

5.4 Nitric Acid, 1 + 4

Dilute with nitric acid (5.3).

5.5 Sulfuric Acid, 1 + 1

Dilute with sulfuric acid with rho about 1.84 g/mL.

5.6 Perchloric Acid, 1 + 499

Dilute with perchloric acid with rho about 1.67 g/mL.

5.7 Phosphoric Acid - Perchloric Acid Mixed Acid

Mix three portions of phosphoric acid (rho about 1.69 g/mL) and one portion of perchloric acid (rho about 1.67 g/mL).

5.8 Sodium (potassium) Periodate Solution, 50 g/L

Weigh-take 5 g of sodium periodate or potassium periodate and place in a 250 mL beaker; add

60 mL of water and 20 mL of nitric acid (see 5.3), warm to dissolve it, then, cool it. Use water to dilute to 100 mL and mix it well.

5.9 Sodium Nitrite Solution, 10 g/L

Weigh-take 1 g of sodium nitrite, place in a 250 mL beaker, add 60 mL of water to dissolve it, then, use water to dilute to 100 mL and mix it well.

5.10.1 Manganese standard solution, 500 ?g/mL

Weigh-take 1.4383 g of potassium permanganate (mass fraction not less than 99.9%) and place in a 600 mL beaker; add 300 mL of water to dissolve it, add 10 mL of sulfuric acid (see 5.5)

and dropwise add hydrogen peroxide (rho about 1.10 g/mL), until the red color just disappears;

heat to boil for 5 min ~ 10 min, then, cool it. Transfer it to a 1,000 mL volumetric flask; use water to dilute to the mark and mix it well.

Alternatively, weigh-take 0.5000 g of electrolytic manganese (mass fraction not less than 99.9%)

[electrolytic manganese needs to be cleaned in sulfuric acid (5 + 95) in advance, and after manganese oxide on the surface is cleaned, take it out, immediately use distilled water to repeatedly wash it, then, wash it in absolute ethanol for 4 ~ 5 times, take it out and dry it in a desiccator before use] and place in a 250 mL beaker; add 20 mL of nitric acid (1 + 3), heat to dissolve it and boil to drive out nitrogen oxides; remove and cool it to room temperature. Then, transfer to a 1,000 mL volumetric flask, use water to dilute to the mark

and mix it well.

1 mL of this standard solution contains 500 ?g of manganese.

5.10.2 Manganese standard solution, 100 ?g/mL

Weigh-take 20.0 mL of manganese standard solution (see 5.10.1) and place in a 100 mL volumetric flask; use water to dilute to the mark and mix it well.

1 mL of this standard solution contains 100 ?g of manganese.

5.10.3 Manganese standard solution, 20 ?g/mL

Weigh-take 20.0 mL of manganese standard solution (see 5.10.2) and place in a 100 mL volumetric flask; use water to dilute to the mark and mix it well.

1 mL of this standard solution contains 20 ?g of manganese.

5.11 Water without Reducing Substances

Heat distilled water or deionized water to boil, use 10 mL of sulfuric acid (1 + 3) to acidify each liter; add a few granules of sodium (potassium) periodate and continue to boil for 1 min ~ 2 min, cool it before use.

reaction stops. Add 1 mL ~ 2 mL of hydrochloric acid (see 5.2), and continue heating, until it is completely decomposed.

---For high-nickel-chromium specimens, add 10 mL of (3 + 1), (6 + 1) or (10 + 1) mixed acid of hydrochloric acid (see 5.2) and nitric acid (see 5.3), heat at low temperature to dissolve it.

NOTE: when the amount of specimen is 2 g, depending on the situation, the amount of acid can be increased, so that the specimen can be completely dissolved.

Add 10 mL of phosphoric acid - perchloric acid mixed acid (see 5.7) [when the amount of specimen is 2.0 g, add 15 mL of phosphoric acid - perchloric acid mixed acid (see 5.7); when using 15 mL of phosphoric acid - perchloric acid mixed acid (see 5.7) to dissolve high-tungsten specimens, it is not necessary to add], heat and evaporate it, until perchloric acid fumes are emitted for 2 min ~ 5 min (for specimens with high chromium content, the chromium needs to be oxidized); slightly cool and add water to dissolve salts. If necessary, remove graphitic carbon by filtration and use hot perchloric acid (see 5.6) to wash it.

---When the manganese content is not greater than 2.00%, add 10 mL of sulfuric acid (see 5.5) and use water to dilute to about 40 mL.

---When the manganese content is greater than 2.00%, add 10 mL of sulfuric acid (see 5.5)

and use water to dilute to about 40 mL; cool to room temperature, then, transfer to a 100