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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 223.8-2025

Replacing GB/T 223.8-2000

Iron, steel and alloy - Determination of aluminium content - Titrimetric method and spectrophotometric method

钢铁及合金 铝含量的测定 滴定法和分光光度法

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1 Scope

This document describes the methods for determining the aluminum content in iron, steel and alloy. sodium fluoride separation-EDTA titration, chrome azurol S direct spectrophotometry, and copper-iron reagent separation-chrome azurol S spectrophotometry.

Method 1, sodium fluoride separation-EDTA titration, is suitable for determining the aluminum content in iron, steel and alloy with a mass fraction of 0.50% ~ 10.00%. Method 2, chrome azurol S direct spectrophotometry, is suitable for determining the acid-soluble aluminum content in iron, steel and alloy with a mass fraction of 0.050% ~ 1.00%. Method 3, copper-iron reagent separation-chrome azurol S spectrophotometry, is suitable for determining the aluminum content in iron, steel and alloy with a mass fraction of 0.015% ~ 0.50%.

2 Normative References

GB/T 6379.1

GB/T 6379.2

GB/T 6682

GB/T 7729

GB/T 12805

GB/T 12806

GB/T 12808

GB/T 20066

3 Terms and Definitions

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

4 Method 1.Sodium Fluoride Separation-EDTA Titration

Method

4.1 Principle

Dissolve the test portion in acid. Use ammonium citrate and ammonium oxalate to complex iron, chromium, nickel and manganese, etc. In a solution with pH 4 ~ 5, use sodium fluoride to separate the aluminum precipitate from the coexisting elements. The precipitate is dissolved with a mixed acid of hydrochloric acid-boric acid. After the filter paper is damaged and fluoride is removed with nitric acid and perchloric acid, add excess EDTA solution to a slightly acidic solution. At approximately pH 6, use xylenol orange as an indicator. Use zinc standard solution to titrate the excess EDTA solution. After complexing fluoride ions with aluminum, release EDTA, which is then titrated again with zinc standard solution. When the total content of zirconium, cerium and rare earth elements in the solution does not exceed 50 mg, it does not interfere with the determination of aluminum.

4.2 Reagents and Materials

Unless otherwise specified, only reagents confirmed to be of analytical grade and Grade II water as specified in GB/T 6682 or water of equivalent purity are used in the analysis.

4.2.1 Sodium pyrosulfate (potassium). 4.2.2 Sodium fluoride.

4.2.3 Hydrochloric acid. rho is approximately 1.19 g/mL.

4.2.4 Hydrochloric acid (1 + 1). Dilute with hydrochloric acid (4.2.3).

4.2.5 Nitric acid. rho is approximately 1.42 g/mL.

4.2.6 Perchloric acid. rho is approximately 1.67 g/mL.

4.2.7 Sulfuric acid. rho is approximately 1.84 g/mL.

4.2.8 Sulfuric acid (1 + 1). Dilute with sulfuric acid (4.2.7).

4.2.9 Sulfuric acid (2 + 100). Dilute with sulfuric acid (4.2.7).

4.2.10 Hydrofluoric acid. rho is approximately 1.15 g/mL.

4.2.11 Ammonia solution. rho is approximately 0.90 g/mL.

4.2.12 Ammonia solution (1 + 1). Dilute with ammonia solution (4.2.11).

4.2.13 Sodium fluoride solution (35 g/L). Store in a plastic bottle.

4.2.14 Sodium fluoride solution (5 g/L). Store in a plastic bottle.

4.2.15 Complexing agent solution. mix ammonium citrate solution (400 g/L) and ammonium

oxalate solution (50 g/L) in equal volumes.

4.2.16 Hydrochloric acid-boric acid mixed acid. mix saturated boric acid solution, hydrochloric

acid (4.2.3), and water in a ratio of (15 + 25 + 60).

4.2.17 Acetic acid-ammonium acetate buffer solution (pH 5.0 ~ 5.5). weigh-take 200 g of ammonium acetate and dissolve it in 500 mL of water. Add 25 mL of glacial acetic acid (rho is approximately 1.05 g/mL) and use water to dilute it to 1 L. Mix well.

4.2.18 2,4-dinitrophenol solution (2 g/L). weigh-take 0.2 g of 2,4-dinitrophenol ammonium acetate, dissolve it in approximately 50 mL of ethanol (1 + 1) (1 portion of ethanol + 1 portion of water), add ethanol (1 + 1) to a final volume of 100 mL, and mix well.

4.2.19 Xylenol orange solution (2 g/L). prepare fresh in summer; in winter, do not exceed one week after preparation.

4.2.20 Ethylenediaminetetraacetic acid disodium salt (EDTA) solution (0.02 mol/L). weigh-

take 7 4448 g of EDTA, place it in a 400 mL beaker, dissolve it in water, transfer it to a 1,000 mL volumetric flask, dilute to the scale, and mix well.

4.2.21 Zinc standard solution (cZnO = 0 0200 mol/L). weigh-take 1 6276 g of zinc oxide (primary standard) that has been dried to constant mass at 110 ?C, place it in a 400 mL beaker;

add 20 mL of hydrochloric acid (4.2.4) and heat to dissolve; add about 200 mL of water, and dropwise add ammonia water (4.2.12), until Congo red test paper changes from blue to purple.

Cool, transfer to a 1,000 mL volumetric flask, use water to dilute to the scale, and mix well.

4.3 Instruments and Equipment

All glassware shall conform to Class A as specified in GB/T 12805 and GB/T 12806.

4.4 Sampling and Sample Preparation

Conduct sampling and sample preparation in accordance with GB/T 20066 and appropriate national standards.

4.5.3.3 Separation of precipitate

4.5.3.3.1 Acidity adjustment Add 100 mL of complexing agent solution (4.2.15) to the test solution obtained in 4.5.3.2.1 or 4.5.3.2.2 Use ammonia solution (4.2.12) and sulfuric acid (4.2.8) to adjust the pH of the test solution to 4 ~ 5 [if tungsten-containing test portions precipitate, first add ammonia solution (4.2.11) to make it alkaline, heat to dissolve the tungstic acid precipitate, cool, and use sulfuric acid (4.2.8) and ammonia solution (4.2.12) to adjust the pH of the test solution to 4 ~ 5]. If the test solution is turbid at this point, heat it to boiling, until it becomes clear. Cool, and then adjust the pH of the test solution to 4 ~ 5 again.

4.5.3.3.2 Precipitation and its dissolution Transfer the test solution (see 4.5.3.3.1) to a 400 mL plastic beaker, add a small amount of paper pulp, and while stirring, add 100 mL of sodium fluoride solution (4.2.13). Thoroughly stir for 5 min, let stand for 40 min, use slow-speed filter paper containing a small amount of paper pulp to filter it, and use sodium fluoride solution (4.2.14) to wash it 7 ~ 8 times. Discard the filtrate.

Transfer the precipitate and filter paper to the original glass beaker, immediately add 20 mL of hydrochloric acid-boric acid mixed acid (4.2.16) to dissolve the precipitate (this shall be immediately added, otherwise the precipitate will not completely dissolve). Add 20 mL ~ 25 mL of nitric acid (4.2.5), boil to break the filter paper (without covering the watch glass).

Slightly cool, add 7 mL of perchloric acid (4.2.6), heat to evaporate until fuming and nearly dry.

Slightly cool and add 50 mL of water to dissolve the salts.

4.5.3.4 Titration

Add an appropriate amount of EDTA solution (4.2.20) to the test solution (see 4.5.3.3.2) in accordance with the aluminum content (for 1 mg aluminum, add 2 mL of EDTA solution), with an excess of 5 mL. Heat it to boiling for 2 ~ 3 minutes, add 2 drops of 2,4-dinitrophenol solution (4.2.18), and dropwise add ammonia solution (4.2.12), until the test solution turns slightly yellow. Continue boiling for 1 ~ 2 minutes, and cool [if the yellow color of the test solution fades at this time, dropwise add ammonia solution (4.2.12), until the test solution is slightly yellow]. Add 20 mL of acetic acid-ammonium acetate buffer solution (4.2.17) and 4 drops of xylenol orange solution (4.2.19). Use zinc standard solution (4.2.21) to titrate the test solution, until it turns purple red (not counting the milliliters). Add 1 g ~ 2 g of sodium