

ICS 77-010
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**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SN/T 5253-2020

**Determination of aluminum content in ferrosilicon EDTA
titration**

硅铁中铝含量的测定 EDTA滴定法

(English Translation)

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Table of Contents

Foreword
1 Scope
2 Normative references
3 Method summary
4 Reagents
4.14 Copper sulfate standard solution
5 Equipment
6 Sample preparation
7 Analysis steps
7.1 Weighing the sample
7.3 Determination
8 Result calculation
9 Precision

Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organizations of this standard. Hohhot Customs of the People's Republic of China, Nanjing Customs of the People's Republic of China.

1 Scope

This standard specifies the analytical method for the determination of aluminum content in ferrosilicon by EDTA titration.

This standard applies to the determination of aluminum content in ferrosilicon. Measuring range. 0.1%-3.0%.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article Pieces. For undated reference documents, the latest version (including all amendments) is applicable to this

document.

GB/T4010 Collection and preparation of samples for chemical analysis of ferroalloys.

GB/T 6379.2 Accuracy of measurement methods and results (accuracy and precision) Part 2 Determine the repeatability and accuracy of standard measurement methods the basic method of reproducibility.

GB/T 6682 Analyze laboratory water specifications and test methods.

3 Method summary

The sample is dissolved with nitric acid and hydrofluoric acid, perchloric acid is added to evaporate to nearly dryness, and other metal ions are formed by adding a strong alkali solution.

Formed a precipitate and removed by filtration. Add excess EDTA to complex with aluminum ion, and back titrate the excess EDTA with copper sulfate. Add sodium fluoride Replace the EDTA complexed with aluminum ion in the same amount, then titrate and record with copper sulfate standard solution, and get the result from the volume of copper sulfate consumed Aluminum content.

4 Reagents

Unless otherwise specified, the reagents used are all analytically pure, and the water is the first grade water specified in GB/T 6682.

4.1

Perchloric acid ($\rho=1.67$ g/mL).

4.2

Nitric acid ($\rho=1.42$ g/mL).

4.3

Hydrofluoric acid ($\rho=1.15$ g/mL).

4.4

Sulfuric acid (1 1).

4.5

Hydrochloric acid (1 1).

4.6

Saturated sodium fluoride solution. add 300 mL of water to a 500 mL beaker with a graduated cylinder, then add sodium fluoride, and stir well with a plastic rod.

Until the solution is saturated, store in a plastic bottle and let stand overnight.

4.7

Sodium hydroxide solution (250 g/L). Weigh 250 g of sodium hydroxide, dissolve it with water and dilute to 1 L, and store it in a plastic bottle.

4.8

Acetic acid-sodium acetate buffer solution. Weigh 100 g of sodium acetate, first dissolve it with a small amount of water, and then add 4.6 mL of glacial acetic acid to mix.

Dilute with water to 500 mL and mix well.

4.9

Sodium hydroxide lotion (10 g/L). Weigh 10 g of sodium hydroxide to dissolve in water and dilute to 1 L, and store it in a plastic bottle.

4.10

EDTA solution (about 0.02 mol/L). Weigh 7.5 g of disodium edetate, dissolve in water and dilute to 1000 mL, and mix well.

4.11

Aluminum standard solution (0.2000 mg/mL). accurately weigh 0.2000 g aluminum powder (high-purity reagent, content $\geq 99.99\%$) into 250 mL in the beaker, add 20 mL of hydrochloric acid (4.5), heat at low temperature to completely dissolve, transfer to a 1000 mL volumetric flask, dilute to the mark with water, and shake well.

4.12

Phenolphthalein indicator (10 g/L ethanol solution).

4.13

PAN indicator (2 g/L ethanol solution).

4.14 Copper sulfate standard solution

a) Standard solution of copper sulfate (0.007 mol/L)

1) Preparation. Weigh 1.7478 g (accurate to 0.0001 g) of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), dissolve it in a small amount of water, and transfer it to 1000 mL volumetric flask, add 10 mL of sulfuric acid (4.4) to the mL volumetric flask, dilute the volume and shake well.

2) Calibration. Use a single-scale pipette to accurately pipette 20 mL of aluminum standard solution (4.11) into a 200 mL Erlenmeyer flask, and add it with a graduated cylinder 20 mL EDTA (4.10), 20 mL acetic acid-sodium acetate buffer (4.8), heat and boil for 5 min, remove and add 8 drops of PAN finger Indicator (4.13), immediately titrate with copper sulfate standard solution (4.14.1) until a stable purple-red color appears, and then add 20 mL of

sodium fluoride saturated Liquid (4.6), the solution appears fruity green, heat and boil for 5 minutes, remove it and add 4 drops of PAN (4.13) indicator while it is hot, and then use copper sulfate the standard solution (4.14.1) is titrated until a stable purple-red color appears, and the volume of the copper sulfate standard solution (4.14.1) consumed for the second time is recorded.

b) Standard solution of copper sulfate (0.002 mol/L)

1) Preparation. Measure 200 mL of copper sulfate standard solution (4.14.1) in a 1000 mL volumetric flask with a graduated cylinder, and mix with 500 mL of water.

2) Calibration. Use a single-scale pipette to accurately pipette 10 mL of aluminum standard solution (4.11) into a 200 mL conical flask, and add 20 mL EDTA (4.10), 20 mL of acetic acid-sodium acetate buffer solution (4.8), heat and boil for 5 min, remove it and add 8 drops of PAN while hot.

Reagent (4.13), immediately titrate with copper sulfate standard solution (4.14.2) until a stable purple-red color appears, and then add 20 mL of saturated sodium fluoride solution (4.6), the solution appears fruity green, heat and boil for 5 min, remove it and add 4 drops of PAN (4.13) indicator while it is hot, then use copper sulfate standard the solution (4.14.2) was titrated until a stable purple-red color appeared, and the volume of the copper sulfate standard solution (4.14.2) consumed for the second time was recorded.

c) Calculate the concentration of copper sulfate standard solution according to formula (1).

5 Equipment

5.1

Analytical balance, with a sensitivity of 0.0001 g.

5.2

Analytical balance, with a sense of 0.01 g.

5.3

Adjustable electric heating plate. the highest temperature can reach 500°C.

5.4

Single-scale pipette, 10 mL.

5.5

Single-scale pipette, 20 mL.