

ICS 77.040.01
CCS H 10

SN

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

SN/T 0254-2020

Replacing SN/T 0254-2011

**Calcined magnesium Determination of acid-soluble chloride
Potentiometric titration**

轻烧镁 酸溶氯化物的测定 电位滴定法

(English Translation)

Issued on: August 27, 2020

Implemented on: March 1, 2021

Issued by: General Administration of Customs

Table of Contents

Foreword
1 Scope
2 Normative references
3 Method summary
4 Reagents and materials
4.9 Silver nitrate standard solution (0.01 mol/L)
5 Instruments and utensils
6 Sample preparation
7 Analysis steps
7.2 Blank test
7.3 Determination
8 Result calculation
9 Precision

Foreword

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

This standard replaces SN/T 0254-2011 "Potentiometric Titration Method for the Determination of Acid-soluble Chloride in Calcined Magnesium".

Compared with SN/T 0254-2011, this standard has the following main changes except for editorial changes.

- Amend the title, amend "Potentiometric Titration Method for the Determination of Acid-Soluble Chloride in Calcined Magnesium" to "Determination of Acid-soluble Chloride in Calcined Magnesium" Constant Potentiometric Titration";
- Edited revisions to the text of the 1 scope and 2 normative references;
- Prefix 7.3.3 in the original standard as 4.9.2, and delete the step of adding potassium nitrate in 4.9.2 silver nitrate solution calibration;
- In 5 instruments and utensils, add 5 mL brown burette;
- In the decomposition of 7.3.1 samples, modify some operation steps;
- In 8 Results and calculations, editorial changes were made to the text part of the calculation method;

-Edited and revised Appendix A.

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

1 Scope

This standard specifies the potentiometric titration method to determine the content of acid-soluble chloride (as Cl) in light burnt magnesia.

This standard is applicable to the determination of acid-soluble chloride (calculated as Cl) in light burned magnesia, fused magnesia, heavy burned magnesia, brucite, and magnesite.

Measuring range (mass fraction). 0.0060% to 0.02%.

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/T 1467 General rules and general provisions of the standard for chemical analysis methods of metallurgical products GB/T 6379.2 Accuracy of measurement methods and results (accuracy and precision) Part 2: Determine the repeatability of standard measurement methods Basic methods with reproducibility GB/T 6682 Analytical laboratory water specifications and test methods GB/T 12805 Laboratory glass burette GB/T 12806 Laboratory glassware single mark volumetric flask GB/T 12808 Single-marked pipette for laboratory glassware

3 Method summary

The sample is decomposed by nitric acid, and under the condition of pH 2~4, the chloride ion selective electrode or silver electrode is used as the measuring electrode, and the double salt bridge calomel electrode it is a reference electrode, titrated with silver nitrate standard solution, according to the potential jump, the second-level derivative method is used to calculate the titration end point.

4 Reagents and materials

Unless otherwise specified in the analysis, only use reagents confirmed to be analytically pure and secondary water that meets the requirements of GB/T 6682 or whose purity is comparable.

When the water.

4.1

Sodium chloride reference reagent.

4.2

Silver nitrate reagent, excellent grade pure.

4.3

Thymol blue indicator.

4.4

Sodium hydroxide reagent, excellent grade pure.

4.5

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

4.6

Nitric acid solution (1:3).

4.7

Sodium hydroxide solution (200 g/L). Weigh 50 g of sodium hydroxide (4.4), dissolve it in 250 mL of water, and transfer it to a plastic bottle for later use.

4.8

Sodium chloride reference solution (0.010 00 mol/L). Weigh the sodium chloride reference reagent (4.1) that has been previously burned at 500 °C to 600 °C to a constant weight. Put 0.5845 g in a 150 mL beaker, add water to dissolve, transfer to a 1000 mL volumetric flask, dilute to the mark with water, and shake well.

4.9 Silver nitrate standard solution (0.01 mol/L)

a) Preparation. Weigh 1.699 g of premium grade pure silver nitrate (4.2) that has been dried at 105 °C for 2 hours in a small 150 mL beaker, and add. After the water is dissolved, transfer it to a 1000 mL brown volumetric flask, dilute to the mark with water, and shake well. This solution should be stored away from light.

b) Calibration. Pipette 5.00 mL sodium chloride reference solution (4.8) into a 100 mL small beaker, add 50 mL water, and put it in the electromagnetic stirrer. Stir, place the beaker on a magnetic stirrer, stir slightly, add 2 to 3 drops of thymol blue indicator (4.10), and add nitric acid solution dropwise (4.6). When the solution is red, adjust it to yellow with sodium hydroxide solution (4.7). At this time, the pH value is about 3-4. Insert measuring electrode and reference Electrode, record the initial potential value, and then titrate with silver nitrate standard solution (4.9). Add 4 mL silver nitrate standard solution (4.9) in advance, Then add

0.20 mL silver nitrate standard solution (4.9) one by one, and record the cumulative volume after each addition of silver nitrate standard solution (4.9) Corresponding potential value, after the potential jump occurs, continue to drip 0.20 mL silver nitrate standard solution (4.9) for 2 to 3 times, and continue to record the accumulation Volume and corresponding potential value. The volume of the silver nitrate standard solution (4.9) added each time during the titration should be the same. Calculate according to the appendix Point volume and calculate the concentration of silver nitrate standard solution according to formula (1).

4.10 Thymol blue indicator (1 g/L). Weigh 0.1 g of thymol blue (4.3) and dissolve it in 100 mL of ethanol (200 ml/L) solution.

4.11 Ethanol solution (200 ml/L).

5 Instruments and utensils

Unless otherwise specified, the volumetric flasks, burettes and pipettes used must comply with GB/T 12805, GB/T 12806, GB/T 12808 Regulations.

5.1

Brown burette, with a minimum graduation of 0.02 mL or 0.05 mL, and a capacity of 5 mL or 10 mL.

5.2

Single-marked volumetric flasks, with capacities of 500 mL and 1000 mL.

5.3

Single-marked pipette with a capacity of 5mL.

5.4

Analytical balance, with a sensitivity of 0.1 mg.

5.5

Ion meter (or automatic potentiometric titrator), with a sensitivity of 0.1 mV.

5.6

Measuring electrode, chloride ion selective electrode (or composite silver ring electrode).

5.7

Reference electrode, double salt bridge calomel electrode.

5.8

Magnetic stirrer and stir bar.