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

GB/T 11213.2-2007

**Sodium hydroxide for chemical fiber use - Determination of
sodium chloride content - Spectrometric method**

(English Translation)

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Foreword

GB/T 11213 Sodium Hydroxide for Chemical Fiber Use can be divided into the following parts:

- Part 1: Sodium Hydroxide for Chemical Fiber Use - Determination of Sodium Hydroxide Content;
- Part 2: Sodium Hydroxide for Chemical Fiber Use - Determination of Sodium Chloride Content - Spectrometric Method;
- Part 3: Sodium Hydroxide for Chemical Fiber Use - Determination of the Calcium Mass Fraction - EDTA Complexometric Method;
- Part 4: Sodium Hydroxide for Chemical Fiber Use - Determination of Silicon Content -Reduced Molybdosilicate Spectrophotometric Method;
- Part 5: Sodium Hydroxide for Chemical Fiber Use - Determination of Sulphate Content;
- Part 7: Sodium Hydroxide for Chemical Fiber Use - Determination of Copper Content -Spectrometric Method.

This Part is Part 2 of GB/T 11213, which corresponds to the Japanese Standard JIS K 1200-3-

1.2000 Sodium Hydroxide for Industrial Use - Part 3: Determination of Chlorides Content -Section 1: Mercury (II) Thiocyanate Photometry (Japanese Version). The consistency

between this Part and JIS K 1200-3-1:2000 is modified to use.

The major differences between this Part and Japanese Standard JIS K 1200-3-1:2000 are as follows:

- The applicable sodium chloride content range is different;
- Add absolute ethanol to increase dissolution and stability when preparing mercury thiocyanate solution, and improve color rendering effect;
- Adjust the preparation operation method according to the range of sodium chloride content in the specimen;
- The representation method of sodium chloride standard solution is different;
- The result calculation formula is changed;
- The content of waste liquid treatment is given in the form of appendix.

This Part replaced GB/T 11213.2-1989 Sodium Hydroxide for Chemical Fiber Use -Determination of Sodium Chloride Content - Spectrometric Method.

Compared with GB/T 11213.2-1989, the major changes of this Part are as follows:

Sodium Hydroxide for Chemical Fiber Use - Determination of Sodium Chloride Content - Spectrometric Method

1 Scope

This Part of GB/T 11213 specifies the method for the determination of sodium chloride content in sodium hydroxide for chemical fiber use.

This Part applies to products with a sodium chloride content of 0.0002% ~ 0.02% in sodium hydroxide.

2 Normative References

The provisions in following documents become the provisions of this Part through reference in this Part. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Part, however, parties who reach an agreement based on this Part are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 603 Chemical Reagent - Preparations of Reagent Solution for Use in Test Methods (GB/T 603-2002, ISO 6353-1:1982, NEQ) GB/T 6682 Water for Laboratory Use - Specifications (GB/T 6682-1992, neq ISO 3696:1987)

3 Principle

Chloride ions (Cl^-) in the specimen completely replace thiocyanate (SCN^-) in mercury thiocyanate; and the substituted thiocyanate (SCN^-) reacts with ferric nitrate to form ferric thiocyanate, which appears red; at the wavelength of 450 nm, the colored solution is photometrically measured. The reaction formula is as follows:

4 Reagents and Materials

The reagents and water used in this method are all analytical reagents and third-grade water specified in GB/T 6682 or water of corresponding purity. The standard solutions, preparations and products required in the test shall be prepared according to the provisions of GB/T 603, except those specified in this Part. The preparation, storage, sampling and determination of reagents shall be carried out in an environment free of chlorine and hydrogen chloride.

4.1

Nitric acid.

4.2

Iron nitrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$].

4.3

Hydrogen peroxide.

4.4

Ferric nitrate solution

8g/L (calculated as Fe).

Choose one of the two preparation methods of ferric nitrate solution.

Method 1: In a 500mL conical flask, add about 4.0g of pure iron (purity >99.5%), accurate to 0.01g. Add 80mL of water; then carefully add 80mL of nitric acid (4.1); and slowly heat the solution in the hood until it boils. After the reaction is complete and all the nitrous acid gas has been driven off; add a few drops of hydrogen peroxide (4.3) to decolorize the solution. Continue to boil for 2 min; stop heating; after cooling off, transfer all the solution into a 500mL volumetric flask; dilute with water to the mark, and shake well.

Method 2: In a 500mL conical flask, add about 29.0g of ferric nitrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$], accurate to 0.01g. Add 60mL of water; then carefully add 60mL of nitric acid (4.1); slowly heat the solution in the hood until it boils. After the reaction is complete and all the nitrous acid gas has been driven off; add a few drops of hydrogen peroxide (4.3) to decolorize the solution.

Continue to boil for 2 min; stop heating; after cooling off, transfer all the solution into a

500mL volumetric flask; dilute with water to the mark, and shake well.

4.5

Mercury thiocyanate solution

0.5g/L.

Weigh 0.1g of mercury thiocyanate $[\text{Hg}(\text{SCN})_2]$, accurate to 0.001g. Put it in a 250mL beaker;

add 30mL of absolute ethanol; and add 150mL of warm water under constant stirring to dissolve it. Then filter the solution into a 200mL volumetric flask; dilute to the mark with water; and shake well.

4.6

Sodium chloride standard solution

0.1mg/mL.

Weigh 0.1g of sodium chloride standard reagent that has been pre-burned at 500°C ~ 600°C to constant weight, accurate to 0.0001g. Put it in a beaker; add a small amount of water to dissolve it; and then transfer the entire solution into a 1000mL volumetric flask. Dilute to the mark with water and shake well.

4.7

Sodium chloride standard solution

0.01mg/mL.

Pipette 20.0mL of sodium chloride standard solution (4.6); place it in a 200mL volumetric flask;

dilute with water to the mark; and shake well.

4.8

Phenolphthalein indicator solution

10g/L.

5 Apparatus

General laboratory instruments and spectrophotometers.

6.1.1 Preparation of standard colorimetric solution

Pipette 0.0mL, 2.0mL, 4.0mL, 6.0mL, 8.0mL, 10.0mL, 12.0mL, 15.0mL of sodium chloride standard solution (4.7) in sequence; respectively placed in 50mL volumetric flasks; then, in each volumetric flask, add 5mL of nitric acid (4.1), 5mL of ferric nitrate solution (4.4) and 20mL of mercuric thiocyanate solution (4.5); dilute to the mark with water; shake well; let stand for 30min to develop color.