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

GB/T 7698-2014

**Sodium hydroxide for industrial use -- Determination of
carbonates content -- Titrimetric method
(English Translation)**

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1 Scope

This standard specifies the method for the determination of carbonate content in sodium hydroxide for industrial use.

This standard applies to products with a mass fraction of carbonate (expressed as Na_2CO_3) greater than or equal to 0.02%.

Through the pre-test, the samples are divided into three categories:

a) Samples free of sulfides and chlorates.

b) Samples containing sulfides. This standard applies to products with a mass fraction of sulfide (expressed as Na_2S) less than 0.1%.

c) Samples containing chlorates. This standard applies to products with a mass fraction of chlorate (expressed as NaClO_3) less than 0.2%.

Due to the presence of interfering components in samples of types b) and c), the analysis steps are modified appropriately (see Chapter 10).

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document;

for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 601 Chemical reagent - Preparations of reference titration solutions GB/T 603 Chemical reagent - Preparations of reagent solutions for use in test methods (GB/T 603-2002, ISO 6353-1:1982, NEQ) GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD) GB/T 29643 Sodium hydroxide for industrial use - Laboratory sample - Preparation of the main solution for carrying out certain determinations [GB/T 29643-2013, ISO 3195:1975(2002), NEQ]

3 Principles

The sample is acidified and heated to release carbon dioxide, and introduced into excess barium hydroxide solution for absorption; with thymolphthalein as indicator solution, the remaining barium hydroxide is titrated with hydrochloric acid standard titration solution until the solution changes from blue to colorless as the end point.

4.1 General rules: Unless otherwise specified, the reagents and water used in this

method refer to analytical reagents or above, and the third-grade water (free of carbon dioxide) specified in GB/T 6682 or water of equivalent purity. The standard titration solutions, preparations, and products required in the test shall be prepared according to the provisions of GB/T 601 and GB/T 603 unless otherwise specified.

4.2 Hydrogen peroxide.

4.3 Hydrochloric acid solution: 6 mol/L.

4.4 Ferrous sulfate solution: 280 g/L.

Weigh 28 g of ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) (accurate to 0.01 g), dissolve it in an appropriate amount of water, add 4 drops of hydrochloric acid solution (see 4.3), dilute the solution to 100 mL, and shake well. When hydrolysis occurs, the solution shall be reconstituted.

4.5 Sodium hydroxide solution: 200 g/L.

4.6 Saturated solution of barium hydroxide.

4.7 Barium hydroxide solution: 0.05 mol/L.

Weigh 15.0 g of barium hydroxide [Ba(OH)₂ · 8H₂O] (accurate to 0.01 g), dissolve it in an appropriate amount of water, transfer the solution to a 1000 mL volumetric flask, dilute the solution with water to the mark, and shake well. Remove the barium carbonate precipitate before use (see Appendix B).

4.8 Hydrochloric acid standard titration solution: c(HCl)=0.1 mol/L.

4.9 Methyl orange indicator solution: 0.5 g/L.

4.10 Thymolphthalein indicator solution: 5 g/L.

4.11 Lead acetate test paper (see Appendix C).

4.12 Nitrogen or air, free of carbon dioxide.

5 Instruments

5.1

Ordinary laboratory equipment.

5.2

Flat-bottom (or round-bottom) two-necked flask

500 mL.

5.3

Separating funnel

100 mL.

5.4

Gas-washing bottle

It contains sodium hydroxide solution (4.5).

5.5

Gas-washing bottle

It contains saturated barium hydroxide solution (4.6).

5.6

Spiral condenser.

5.7

Absorption vessel.

5.8

Absorption coil

At least 700 mm in length.

5.9

Burette

50 mL, graduated in 0.1 mL, grade A.

6.1 Principle

After an acidified sample containing methyl orange indicator solution is boiled, the lead acetate test paper strip is put into the steam; the test paper turns black to indicate the presence of sulfide, and the decolorization of methyl orange indicates the presence of chlorate.

6.2 Pre-test steps

Weigh about 20 g of solid or liquid sample, place it in a 300 mL conical flask, and add 150 mL of water and 3 drops of methyl orange indicator solution; neutralize the solution with a the hydrochloric acid solution, and add an excess of about 5 mL. Put one end of the lead acetate test paper strip on the inside of the conical flask, and the other end on the outside of the neck of the bottle. Boil the solution for 5 min, and determine the analysis steps according to the appearance listed in Table 1.

Description:

A -- flat-bottomed (or round-bottomed) two-necked flask;

B -- separating funnel;

C1, C2, C3 -- gas-washing bottle;

D -- spiral condenser;

E -- absorption vessel;

F -- absorption coil;

G -- burette;

H -- V-shape hole stopcock;

I, J -- inlet and outlet for nitrogen or air.

Figure 1 -- Schematic diagram of the carbonate determination device

7.3.2 Introduce carbon dioxide-free nitrogen or air into the device at a rate of about 5

bubbles per second for 10 minutes.