GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 71.060.10

CCS G 13

GB/T 5138-2021

Replacing GB/T 5138-2006

Liquid Chlorine for Industrial Use

工业用液氯

Issued on: August 20, 2021 **Implemented on: July 1, 2022**

Issued by: State Administration for Market Regulation;

Standardization Administration of the People's Republic of China.

Table of Contents

Foreword....3
1 Scope....5
2 Normative References....5
3 Terms and Definitions....6
4 Technical Requirements....6
5 Sampling....6
6 Test Methods....6
7 Inspection Rules....15
8 Markings, Accompanying Documents, Packaging, Transportation and Storage....16

Liquid Chlorine for Industrial Use

WARNING---the personnel using this document shall have practical experience in formal laboratory work. This Standard does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring the compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

This document specifies the technical requirements, sampling, test methods, inspection rules, markings, accompanying documents, packaging, transportation and storage of liquid chlorine for industrial use.

This document is applicable to liquid chlorine produced by electrolytic method-generated chlorine through drying and liquefying.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB 190 Packing Symbol of Dangerous Goods

GB/T 602 Chemical Reagent - Preparations of Standard Solutions for Impurity

GB/T 603 Chemical Reagent - Preparations of Reagent Solutions for Use in Test Methods

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

GB 11984 Safety Regulation for Chlorine

GB 15258 General Rules for Preparation of Precautionary Label for Chemicals

HG/T 3941 Liquid Chlorine for Industrial Use - Determination of Water Content - Electrolytic Method

A---gas burette, 100 mL, with 0.05 mL division value on the upper part;

B, C---rotary cock;

D---outlet of gas burette;

E---leveling bottle, 250 mL;

F---absorption bottle, 500 mL, filled with sodium hydroxide solution.

Figure 1 -- Schematic Diagram of Chlorine Content Determination Device

6.2.4 Analytical procedures

6.2.4.1 Connect the gas burette to the leveling bottle as shown in Figure 1. Rotate Cock C of the gas burette, so that the gas burette is connected to the atmosphere. Then, rotate Cock B; connect the gas burette to the leveling bottle; adjust the position of the leveling bottle; make the liquid level of the potassium iodide solution in the leveling bottle on a level with the “0” scale at the lower end of the gas burette; close Cock B.

6.2.4.2 Connect the outlet D of the gas burette to the absorption bottle; rotate Cock B to connect the gas burette to the sampler. Slowly open the valve of the sampler to let chlorine flow into the gas burette for 2 min ~ 3 min; completely replace the air in the gas burette. Close the valve of the sampler; quickly and successively close Cock C and Cock B of the gas burette; remove the absorption bottle and the connecting pipe that passes chlorine. Leave it for a while, so that the chlorine temperature in the gas burette reaches equilibrium with the outside world. Quickly rotate Cock C of the gas burette by one turn.

6.2.4.3 Gradually raise the leveling bottle; rotate Cock B, so that the potassium iodide solution flows a little into the gas burette, and shake it; close Cock B, so that the chlorine is absorbed by the potassium iodide solution. Repeat this operation, until no chlorine is absorbed by the solution, then, let it stand and cool for 10 min ~ 15 min. Adjust the position of the leveling bottle, make the gas burette on a level with the liquid level of the leveling bottle; read the scale value of the absorption liquid level in the gas burette.

6.2.5 Result calculation

The chlorine content is calculated by the volume fraction (φ) of chlorine (Cl_2) and in accordance with Formula (1):

$$\varphi = \frac{V_1}{V} \times 100\% \qquad \cdots\cdots(1)$$

Where,

E---gas absorption tube;

H---air purification bottle (within 1 + 5 sulfuric acid solution inside);

I---electric heating device;

J---hydrochloric acid separation bottle;

K---sodium hydroxide solution absorption bottle;

L---conical flask (with color-changing silica gel inside);

M---conical flask (with calcium oxide inside).

Figure 4 -- Schematic Diagram of Hydrochloric Acid Separation Device

6.4.5 Analytical procedures

6.4.5.1 Draw a standard curve

6.4.5.1.1 Successively measure-take 0.0 mL, 0.4 mL, 0.8 mL, 1.2 mL, 1.6 mL and 2.0 mL of ammonium standard solution (6.4.3.8); place them in six 20 mL colorimetric tubes with stopper. Use water to dilute them to the scale. Respectively add 1 mL of Nessler's reagent; shake them well; let them stand for 10 min.

6.4.5.1.2 Use an appropriate cuvette. At a wavelength of 420 nm, use water to adjust the zero point of the spectrophotometer; determine the absorbance of the solution.

6.4.5.1.3 Subtract the absorbance of the blank solution from the absorbance of the colorimetric solution. Take the ammonium content (μg) as the x-coordinate and the corresponding absorbance as the y-coordinate; draw a standard curve.

6.4.5.2 Specimen

Use filter paper to wipe the sampling valve clean; carefully open the valve; let an appropriate amount of chlorine into the unmetered sodium hydroxide solution absorption bottle to clean the valve. In accordance with Figure 3, properly install the sampling device; control the gauge pressure to sample at 0.02 MPa ~ 0.04 MPa for about 10 min. The chlorine passes through a gas absorption tube (keep away from light) containing 5 mL of hydrochloric acid solution and is absorbed by the metered sodium hydroxide solution absorption bottle that is about 200 mL. In accordance with the mass (accurate to 0.1 g) of the absorption bottle before and after the sampling, calculate the sampling size.

6.4.5.3 Separation

Put the gas absorption tube into the pre-heated hydrochloric acid separation device (see Figure 4) to remove hydrochloric acid.

6.4.5.4 Determination

6.4.5.4.1 Use water to rinse the inner wall of the gas inlet of the sampling gas absorption tube after sample separation and the outer wall of the part immersed in the hydrochloric acid absorption solution. Add 1 drop of potassium sodium tartrate solution; use water to dilute to the scale. Then, add 1 mL of Nessler's reagent; shake it well; let it stand for 10 min.

6.4.5.4.2 At a wavelength of 420 nm, use the same cuvette as the cuvette used to draw the standard curve; use water to adjust the zero point of the spectrophotometer; determine the absorbance of the solution.

6.4.5.5 Blank test

DO NOT add the specimen. Add 5 mL of hydrochloric acid solution; use the same reagents and dosage as the specimen. In accordance with 6.4.5.3 and 6.4.5.4, conduct the blank test.

6.4.6 Result calculation

Nitrogen trichloride is calculated by the mass fraction (w_1) of (NCl_3) and in accordance with Formula (2):

$$w_1 = \frac{(m_1 - m_2) \times 10^{-6} \times 6.67 \times 1.025}{m_3} \times 100\% \qquad \cdots\cdots\cdots\cdots\cdots\cdots (2)$$

Where,

w_1---the mass fraction of nitrogen trichloride, %;

m_1---the mass of ammonium in the specimen obtained from the standard curve, expressed in (μg);

m_2---the mass of ammonium in the blank test obtained from the standard curve, expressed in (μg);

m_3---the mass of the specimen (the mass difference of the sodium hydroxide absorption bottle E before and after sampling), expressed in (g);

6.67---the conversion factor of ammonium and nitrogen trichloride;

1.025---the absorption coefficient.

6.5 Determination of Evaporation Residue

6.5.1 Principle

Under laboratory temperature conditions, after a certain volume of specimen is gasified and evaporated, weigh the mass of the evaporation residue.

6.5.2 Reagents or materials

6.5.2.1 Sodium hydroxide (industrial product) solution: 200 g/L.

6.5.2.2 Dry air or nitrogen.

6.5.3 Instruments

Sampling evaporation device (see Figure 5), analytical balance (division value: 0.0001 g), balance (load: 5,000 g; division value: 0.1 g), thermometer (-80 °C), and general laboratory instruments.

6.5.4 Analytical procedures

6.5.4.1 Dry the conical flask at 105 °C ~ 110 °C for 1 h; cool it down. Place it in a desiccator for 30 min; weigh it (accurate to 0.0001 g).

6.5.4.2 In accordance with Figure 5, properly install the sampling evaporation device. Add dry ice and absolute ethanol to 800 mL beaker; the liquid level is about 2 cm higher than the outlet of the feeding tube of the conical flask. After the temperature of the cold source is lower than -50 °C for 5 min, when the outlet of the conical flask is not connected to the inlet of the buffer bottle, slowly open the outlet valve of the stainless steel sampler, so that the liquid chlorine slowly flows into the conical flask for about 150 mL. Close the outlet valve of the stainless steel sampler; clamp the inlet rubber hose of the conical flask; lower the height of the lifting platform to separate the conical flask away from the cold source. When the chlorine starts to evaporate, quickly use a hose to connect the outlet of the conical flask and the inlet of the buffer bottle, so as to gasify the liquid chlorine at laboratory temperature, then, let it pass the buffer bottle into the sodium hydroxide solution absorption bottle for absorption.

6.5.4.3 After the gasification is completed, let an appropriate amount of dry air or nitrogen into the absorption bottle through the conical flask and buffer bottle. Use a clean and soft cloth to carefully wipe the outer wall of the conical flask; place it in a desiccator for 10 min; weigh it (accurate to 0.0001 g). Use a balance to weigh the sodium hydroxide absorption bottle (accurate to 0.1 g) that absorbs chlorine.

6.5.5 Result calculation

Evaporation residue is calculated by the mass fraction (w_2) of the residue and in accordance with Formula (3):

$$w_2 = \frac{m_4}{m_5} \times 100\% \qquad \cdots\cdots (3)$$

Where,

w_2---the mass fraction of the residue, %;

---When there are relatively significant changes in the production process (such as: materials and process conditions, etc.);

---After there are major fluctuations and adjustments in the production equipment.

Under normal production, type inspection shall be carried out at least once a month (except for evaporation residue); evaporation residue shall receive type inspection at least once every three months.

7.1.3 Exit-factory inspection

The exit-factory inspection item is chlorine content.

7.2 Determination Rules

The product quality indicators shall be determined in accordance with the “full numerical comparison method” specified in GB/T 8170.

7.3 Re-inspection Rules

If there is an indicator in the inspection result that does not comply with the requirements of this documents, double the sampling in the packaging unit, storage tank or tank wagon for re-inspection. If there is an indicator in the re-inspection result that does not comply with the requirements of this document, then, this batch of products shall be determined as disqualified.

8 Markings, Accompanying Documents, Packaging, Transportation and Storage

8.1 Markings

The tank wagon or gas cylinder for the exiting-factory liquid chlorine for industrial use shall have obvious and firm markings, which include: manufacturer name, address, product name, trademark, serial No. of implemented standard, container mass and packing unit, tank wagon No. or cylinder No., production license No. and “hazardous” sign that complies with the stipulations of GB 190. The label shall comply with the stipulations of GB 15258.

8.2 Accompanying Documents

Each batch of the exiting-factory liquid chlorine products shall be inspected by the quality supervision and inspection department of the manufacturer in accordance with the requirements of this document, and accompanied by a quality certificate, which includes: manufacturer name, product name, “hazardous chemicals” sign, quality index, grade, batch No. or production date and serial No. of implemented standard.

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------