



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

GB 5009.244-2016

**National food safety standard - Determination of chlorine
dioxide in food**

食品安全国家标准 食品中二氧化氯的测定

(English Translation)

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Foreword

This Standard replaces SN/T 1954-2007, Determination of chlorine dioxide residues in iced meat for import and export - Spectrophotometric method.

Compared with SN/T 1954-2007, the major technical changes of this Standard are as follows.

- it changes test methods and their applicable scope;
- it adds classification of samples;
- it changes sample processing conditions;
- it changes detection limit and adds quantitative limit.

National food safety standard -

Determination of chlorine dioxide in food

1 Scope

This Standard specifies the spectrophotometric method for the determination of chlorine dioxide in vegetables, fruits, livestock and poultry meat and aquatic products.

This Standard applies to the determination of chlorine dioxide in vegetables, fruits, livestock and poultry meat and aquatic products.

2 Principle

Use phosphate buffer to extract chlorine dioxide in sample; after refrigerated centrifugation, use fibre filter paper for filtration; use glycine as the masking agent to eliminate the false positive interferences of the substances such as Cl_2 and ClO_2^- ; add N, N-diethyl-p-phenylenediamine (DPD) colour developing agent for developing with chlorine dioxide; use the spectrophotometric method to measure the maximum absorbance at 552 nm to determine the content of chlorine dioxide in food.

3 Reagents and materials

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

3.2.1 DPD colour developing agent (1 g/L). weigh 1 g of DPD (3.1.10) to dissolve in

chloride-free grade two water (3.2.2) containing 8 mL of sulfuric acid solution (1 + 3) and 200 mg of EDTA; use chloride-free grade two water to dilute to 1,000 mL; store in a brown glass bottle with stopper; store in a dark place.

3.2.2 Oxidizing chloride-free grade two water. add sodium sulfite (3.1.4) in grade two

water; reduce oxidizing chloride to chloride ion (no colour develops when checked using DPD); carry out distillation. The water obtained is oxidizing chloride-free grade two water.

3.2.3 Glycine solution (10%). take 10 g of glycine (3.1.1); use water to dissolve; make

up to 100 mL.

3.2.4 Sodium hydroxide solution (2 mol/L). weigh 80 g of sodium hydroxide; use water

to dissolve; make up to 1 L.

3.2.5 Sodium hydroxide solution (0.1 mol/L). weigh 4 g of sodium hydroxide; use water

to dissolve; make up to 1 L.

3.2.6 Sulfuric acid solution (1 mol/L). measure 54 mL of 98.4% sulfuric acid (3.1.6);

slowly add into about 300 mL of distilled water; after cooling, transfer to a volumetric flask; use about 20 mL of distilled water to wash the container for 3 times; also transfer the washings to the volumetric flask; use water to make up to 1 L.

3.2.8 Phosphate buffer solution (pH = 6.5). take 500 mL of 0.1 mol/L KH₂PO₄ and 81

mL of 0.1 mol/L NaOH; use water to dilute to 1 L.

3.2.9 Sodium chlorite saturated solution. take an appropriate amount of sodium chlorite

(3.1.4) to place in a beaker; add a small amount of water; stir to make a saturated solution (the solubility of sodium chlorite is quite high, so it shall be prepared in accordance with the amount required).

3.2.10 Preparation of sodium thiosulfate standard solution [c(Na₂S₂O₃) = 0.1000 mol/L].

weigh 16 g of anhydrous sodium sulfate (3.1.7); dissolve in 1 L of water; heat to boil for 10 min; allow to cool; filter to use after two weeks' storage in dark.

3.2.11 Calibration of sodium thiosulfate standard solution [c(Na₂S₂O₃) = 0.1000 mol/L].

weigh 0.15 g of benchmark potassium dichromate (3.1.13) dried to constant weight at 120°C, accurate to 0.000 1 g; place into an iodine flask; add 25 mL of water; after dissolving, add 2 g of potassium iodide and 20 mL of 20% sulfuric acid solution; shake up; store in dark for 10 min. Add 150 mL of water; use prepared sodium thiosulfate standard solution (3.2.10) for titration; add 3 mL of starch indicating liquid (5 g/L) (3.1.12) near the end; continue titration until the solution changes from blue into bright

green. Meanwhile, carry out blank test.

3.2.12 Starch indicator (5 g/L). weigh 5.0 g of starch to place into a beaker of 50 mL;

measure 1 L of distilled water; use several drops to make starch into a paste; then take about 900 mL of water to heat to slightly boiling when pour the pasty starch in; use the residual distilled water to wash the beaker of 50 mL for 3 times; pour the washings into the beaker; then add 1 drop of 10% hydrochloric acid; slightly boil for 3 min.

3.2.13 Starch solution (1%). weigh 1 g of soluble starch; use a small amount of water

to make a paste; use 100 mL of boiling water to dissolve it; heat to boil until clarified.

3.3.1 Preparation of chlorine dioxide standard stock solution

Add 300 mL of pure water into bottle A; connect the glass at one end of bottle A to air compressor and another glass tube to bottle B. Bottle B is a high-strength borosilicate glass bottle with three glass tubes at the opening; the first one is inserted to 5 mm to the bottom to introduce air; the second is connected with a graduated cylindrical separating funnel with its lower end stretching to below the liquid level; the lower end of the third is apart from the liquid level and the upper end is connected with bottle C. Dissolve 10 g of sodium chlorite in 750 mL of pure water and pour into bottle B; load

20 mL of sulfuric acid solution in the separating funnel. Bottle C is loaded with the

scrubber tower of sodium chlorite saturated solution. Bottle D is a borosilicate glass receiving flask of 2 L, which contains 1,500 mL of pure water used to absorb the chlorine dioxide generated; the residual gas is discharged through exhaust pipes. The whole apparatus is placed in a fuming cupboard. For the generation and absorption apparatus of chlorine dioxide see Figure 1.

Figure 1 - Generation-absorption apparatus of chlorine dioxide

Start air compressor to make the air pass through the whole apparatus evenly. Add 5 mL of sulfuric acid solution through separating funnel every 5 min. After adding sulfuric