GB

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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

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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^-; 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.

3.1 Reagents

3.1.1 Glycine ($C_2H_5NO_2$).

3.1.2 Ethylenediaminetetraacetic acid disodium (EDTA) ($C_{10}H_{14}N_2Na_2O_8$).

3.1.3 Potassium iodide (KI): purity 99.5%.

3.1.4 Sodium chlorite ($NaClO_2$).

3.1.5 Acetic acid (CH_3COOH).

3.1.6 Sulfuric acid (H_2SO_4).

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 [$c(Na_2S_2O_3)$ = 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 [$c(Na_2S_2O_3)$ = 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 Standard substances

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.

5.3.1 Fruits and vegetables

Weigh 1.00 g (accurate to 0.01 g) of sample to place into a centrifuge tube of 50 mL; add 20 mL of phosphate buffer solution (pH 6.5) (3.2.8); carry out homogeneous extraction for 3 min at 8,000 r/min; carry out refrigerated centrifugation in a high-speed centrifugal machine for 10 min at 10,000 r/min; take out; use fibre filter paper to filter into a colourimetric tube with stopper of 10 mL for the determination on a spectrophotometer.

5.3.2 Livestock-Poultry Products and aquatic products

Weigh 1.00 g (accurate to 0.01 g) of sample to place into a centrifugal tube of 100 mL; add 50 mL of phosphate buffer solution (pH 6.5) (3.2.8); carry out homogeneous extraction for 3 min at 8,000 r/min; carry out refrigerated centrifugation in a high-speed centrifugal machine for 10 min at 10,000 r/min; take out; use fibre filter paper to filter into a colourimetric tube with stopper of 10 mL for the determination on a spectrophotometer.

5.4 Determination

5.4.1 Plotting of standard curve

Add a certain amount of chlorine dioxide standard using solution to a series of 10 mL stoppered colourimetric tubes to make the concentrations of all tubes equal to chlorine dioxide standard solutions of 0.00 mg/L, 0.05 mg/L, 0.10 mg/L, 0.50 mg/L, 1.00 mg/L, 2.00 mg/L and 5.00 mg/L.

Add respectively 1.0 mL of phosphate buffer solution (3.2.8), 1.0 mL of DPD solution (3.2.1) and 1.0 mL of glycine solution; make up to scale and shake up; within 60 s, use cuvettes of 1 cm to measure the absorbance at 552 nm; plot standard curve using the concentration of standard working solution as the abscissa and the response value (absorbance value) as the ordinate. See Annex A.

5.4.2 Determination of chlorine dioxide in sample

Take 5 mL of filtrate to pour into a stoppered colourimetric tube of 10 mL; add 1 mL of glycine solution (3.2.3) to the filtrate to mix up; add 1 mL of phosphate buffer solution (3.2.8) and 1.0 mL of DPD solution (3.2.1); use water to make up to 10 mL and shake up. Immediately within 60 s, use cuvettes of 1 cm to measure the absorbance at 552 nm; obtain the concentration of chlorine dioxide in accordance with the standard curve.

NOTE: The whole process shall be carried out in dark.

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