# Method of Test for Sulfur Dioxide in Foods

## 1. Scope

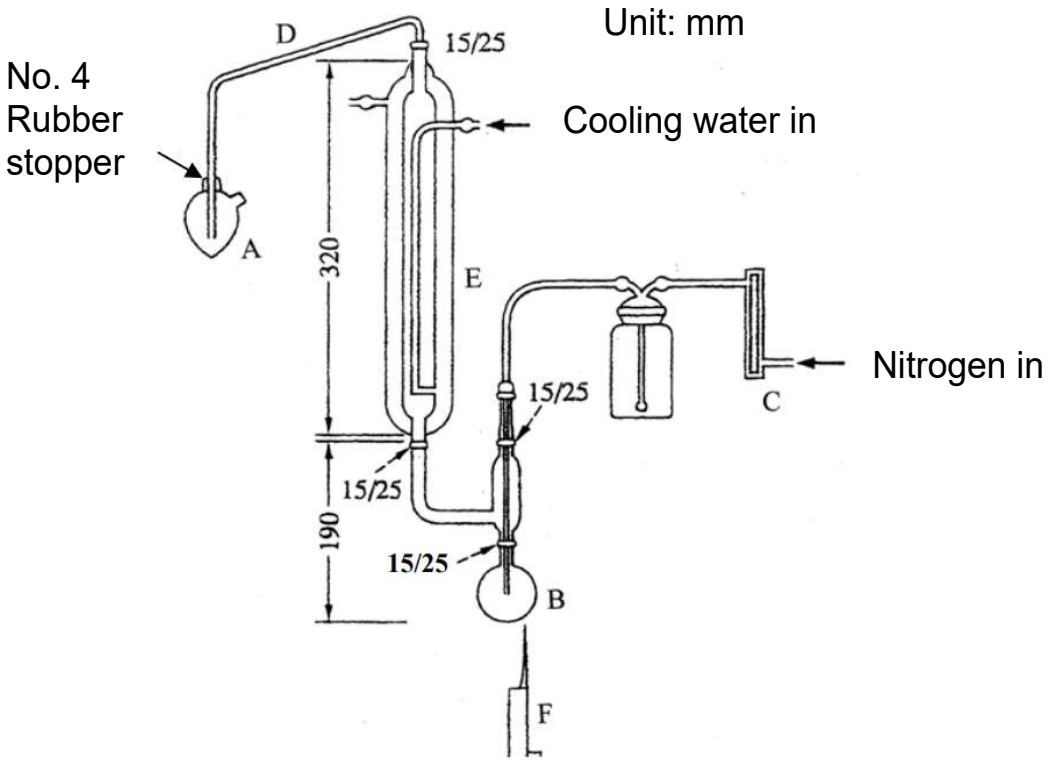
This method is applicable to the determination of sulfur dioxide in foods.

## 2. Method

After the samples are subjected to aeration distillation, sulfur dioxide is determined by alkalimetric titration.

**2.1.** Equipment

**2.1.1.** Aeration distillation apparatus.




A. Pear-shaped flask: 50 mL, two neck.

B. Round-bottom flask: 100 mL, ground joint 15/25.

C. Nitrogen gas with flowmeter.

D. Glass tubing: Ground joint, 10/19.

E. Condenser.

F. Bunsen burner.

**2.2.** Chemicals

Methyl red, Reagent grade;

Methylene blue, Reagent grade;

Hydrogen peroxide, 30%, Reagent grade;

Sodium hydroxide, 0.01 N, Reagent grade;

Phosphoric acid, 85%, Reagent grade;

Alcohol, 95%, Reagent grade;

Silicone oil, GR grade;

Boiling chip, GR grade;

Deionized water, resistivity ≥ 18 MΩ · cm (at 25℃).

**2.3.** Apparatus

**2.3.1.** Burette: 25 mL, accuracy 0.05 mL.

**2.4.** Reagents

**2.4.1.** Mixed indicator

Dissolve 0.2 g of methyl red and 0.1 g of methylene blue with alcohol to 100 mL.

**2.4.2.** 0.3% hydrogen peroxide

Dilute 1 mL of 30% hydrogen peroxide to 100 mL with deionized water prior to use.

**2.4.3.** 25% phosphoric acid

Dilute 29.4 mL of phosphoric acid with deionized water to 100 mL.

**2.5.** Sample solution preparation

Transfer 10 mL of 0.3% hydrogen peroxide into a 50-mL pear-shaped flask, then add 3 drops of mixed indicator, the solution is purple in color. Add 1-2 drops of 0.01 N sodium hydroxide to make solution to olive-green color, then connect the pear-shaped flask to aeration distillation apparatus. For solid sample, cut into small pieces (2 mm or less), and transfer about 1-5 g of sample accurately weighed into a round-bottom flask with 20 mL of deionized water(note). For liquid sample, transfer 20 g of well-mixed sample accurately weighed into a round-bottom flask. Two mL of alcohol, 10 mL of 25% phosphoric acid, 2 drops of silicone oil, and boiling chips are added into the round-bottom flask, and then connect to aeration distillation apparatus immediately. The nitrogen flow rate is set to 0.5-0.6 L/min. Heat the round-bottom flask by the Bunsen burner with about 4-5 cm flame height for 10 min. After heating, disconnect the pear-shaped flask, and rinse the tip of glass tubing with deionized water into the pear-shaped flask as sample solution. For a blank sample solution, take another round-bottom flask, add 20 mL of deionized water(note), 2 mL of alcohol, 10 mL of 25% phosphoric acid, and boiling chips, and the following steps are the same as the sample solution.

Note: degas deionized water prior to use.

**2.6.** Quantification

Titrate the sample solution and the blank solution with 0.01 N sodium hydroxide until the color appear olive green, respectively. Calculate the amount of sulfur dioxide in the sample by the following formula:

$$\text{The amount of sulfur dioxide in the sample (g/kg)} = \frac{(C-B)\times f\times 0.32}{W}$$

C: the titration volume of 0.01 N sodium hydroxide for the sample solution (mL)

B: the titration volume of 0.01 N sodium hydroxide for the blank solution (mL)

f: the titer of 0.01 N sodium hydroxide

0.32: the titration of 0.01 N sodium hydroxide, 1 mL = 0.32 mg $SO_2$

W: the weight of the sample (g)

**Remark**

1. Limit of quantification (LOQ) for sulfur dioxide is 0.01 g/kg.
2. If used other heating methods (such as electric furnace, heating bag or water bath, etc.) for aerated distillation, method validation or verification should be performed to ensure the correctness of the test results.
3. When the content of sulfur dioxide in the sample is close to or slightly higher than the regulatory limit, to ensure the sulfite contained in the sample is completely converted to sulfur dioxide and absorbed by the receiver solution, the sampling volume could be moderately adjusted and compared the correlation between the output value and the sampling volume to ensure the correctness of the results.
4. Further validation should be done when interference compounds appear in samples.

**Reference**

1. The Pharmaceutical Society of Japan. 2015. Methods of Analysis in Health Science, p. 351-358. Kanehara & Co., Ltd, Tokyo, Japan.
2. Ministry of Health, Labor and Welfare of Japan. 2021. Sulfur dioxide and sulfites. Analysis of food additives in food. Reiwa 3 June 24 notification.