

# DETERMINATION OF SULPHUR DIOXIDE (residual SO<sub>2</sub>) BY RANKINE METHOD A : ALKALI TITRATION METHOD

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## INTRODUCTION

Sulphur dioxide in foods is extracted by heating with phosphoric acid (H<sub>3</sub>PO<sub>4</sub>). The sulphur dioxide is changed to sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) in the presence of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). The sulphuric acid is then titrated against sodium hydroxide (NaOH).

This method is applicable to all foods. However, the colorimetric method is better than this alkali method for foods containing a large amount of sulphide and salt.

## APPARATUS

1. Round bottomed flasks, 100 ml
2. Oval shaped flasks, 50 ml
3. Burette, 50 ml
4. Apparatus for distillation as shown in Fig. 1.

## REAGENTS

1. Carbon dioxide (CO<sub>2</sub>) free distilled water
2. Indicator solution

Dissolve 0.2 g methyl red and 0.1 g methylene blue in ethanol (95%, v/v) and make up to 100 ml.

3. 0.3% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) solution

Pipette 1 ml of 30% H<sub>2</sub>O<sub>2</sub> (analytical grade) into distilled water in a 100 ml volumetric flask, and make up to 100 ml with distilled water.

4. 25% phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) solution

Add 240 ml of distilled water to 100 ml of 85% H<sub>3</sub>PO<sub>4</sub>. Mix thoroughly.

5. 0.01N (N/100) sodium hydroxide (NaOH) solution
6. Silicon oil as antifoaming agent (food additive).

## PROCEDURE

### Sample Preparation

The weight required for various samples are as follows :

20 g	—————	Wine, natural fruit juice, soybean sauce.
10 g	—————	Green mustard paste, molasses, millet jelly, candied cherry, dried fruit.
1 g	—————	Paste made from the arum root powder (Konniaku), boiled bean, cut and dried radish, miso (soybean paste), chocolate, sugared beans, garlic, frozen shrimp, gelatin.
0.1 - 0.2 g	—————	Dried gourd shavings (Kanpyo).

### Analytical Procedure (Figs. 1 and 2)

1. Into Flask A pipette 10 ml of 0.3%  $\text{H}_2\text{O}_2$  followed by 3 drops of indicator solution, 1 - 2 drops of 0.01N  $\text{NaOH}^{*1}$ . The colour should change to olive green.
2. Fix Flask A to double cooling tube C.
3. Weigh sample into Flask B<sup>\*2</sup> and add 40 ml of distilled water, followed by 1 - 2 drops of silicon oil<sup>\*3</sup> and 10 ml of 25%  $\text{H}_3\text{PO}_4$ .
4. Quickly fix Flask B to glass capillary E.
5. Flow in air or nitrogen gas at a speed of 0.6 l/min.
6. Set micro-burner flame height at 4 - 5 cm and heat Flask B for 10 min.
7. Pull out Flask A, and wash the tip of the glass delivery tube with a small amount of distilled water into Flask A.
8. Titrate the solution in Flask A against 0.01N  $\text{NaOH}$  till solution in Flask A turns olive green.

## CALCULATION

$$\text{SO}_2 \text{ (g/kg)} = 0.3205 \times (a - b) \times f \times 1/W$$

Where a = titration volume of sample solution (ml)

b = titration volume of blank test solution (ml)

f = factor of 0.01N NaOH

W = sample weight (g)

0.3205 = 1 ml of 0.01N NaOH contains 0.3205 mg of SO<sub>2</sub>

Detection limit : 5 ppm (0.005 g/kg)

## REMARKS

- \*1 = For the purpose of neutralising solution in SO<sub>2</sub> receiver i.e. Flask A.
- \*2 = Sulphur dioxide is unstable. Thus, after weighing, the sample must be analysed immediately.
- \*3 = For use as antifoaming agent.

## REFERENCE

Shokuhin Eisei Kensa Shishin, Guidelines for Food Hygiene Inspection. (Analytical Methods for Food Additives in Food), Japan Food Hygiene Association (1989).

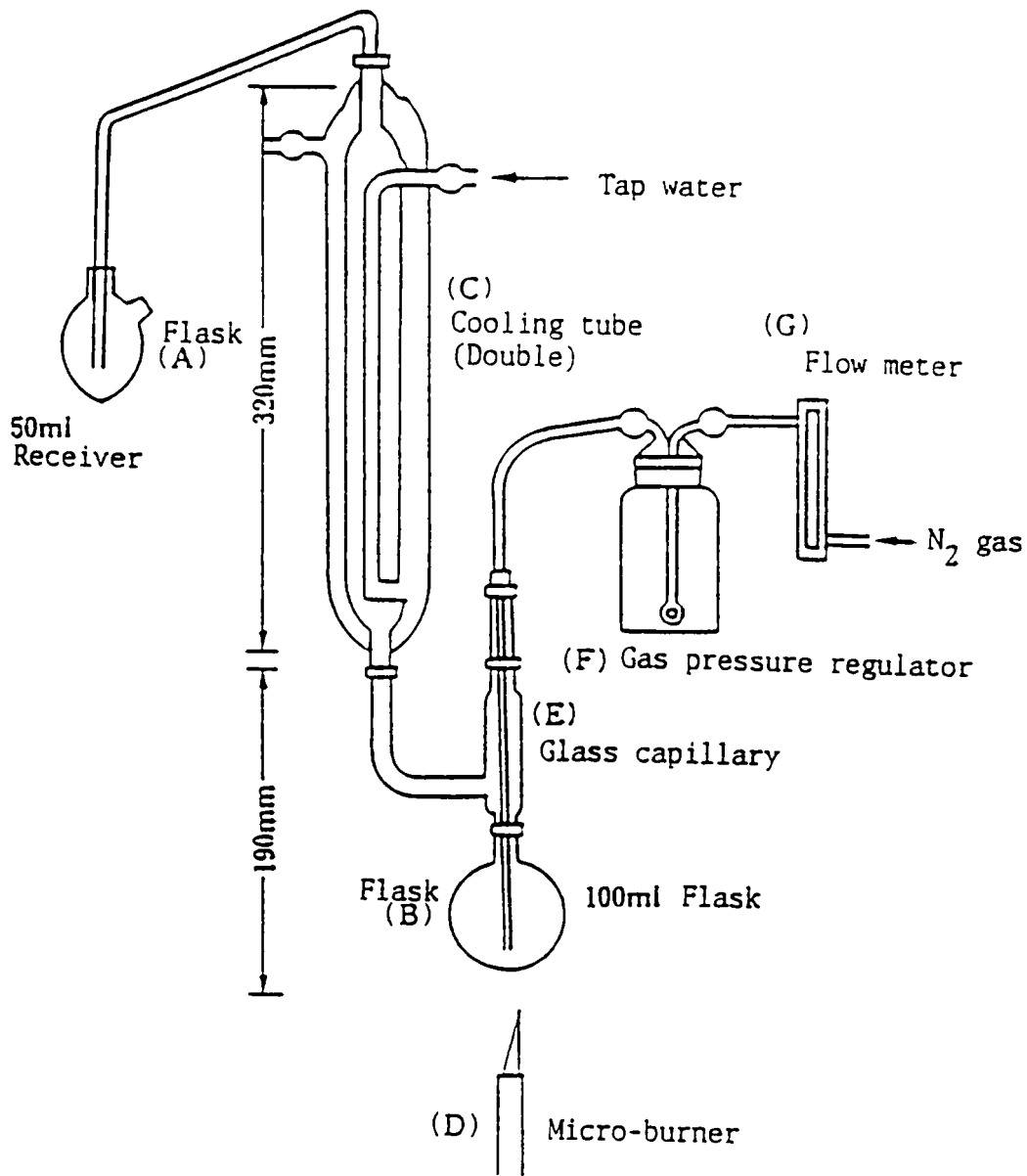


Fig. 1. APPARATUS FOR DISTILLATION (RANKINE METHOD)

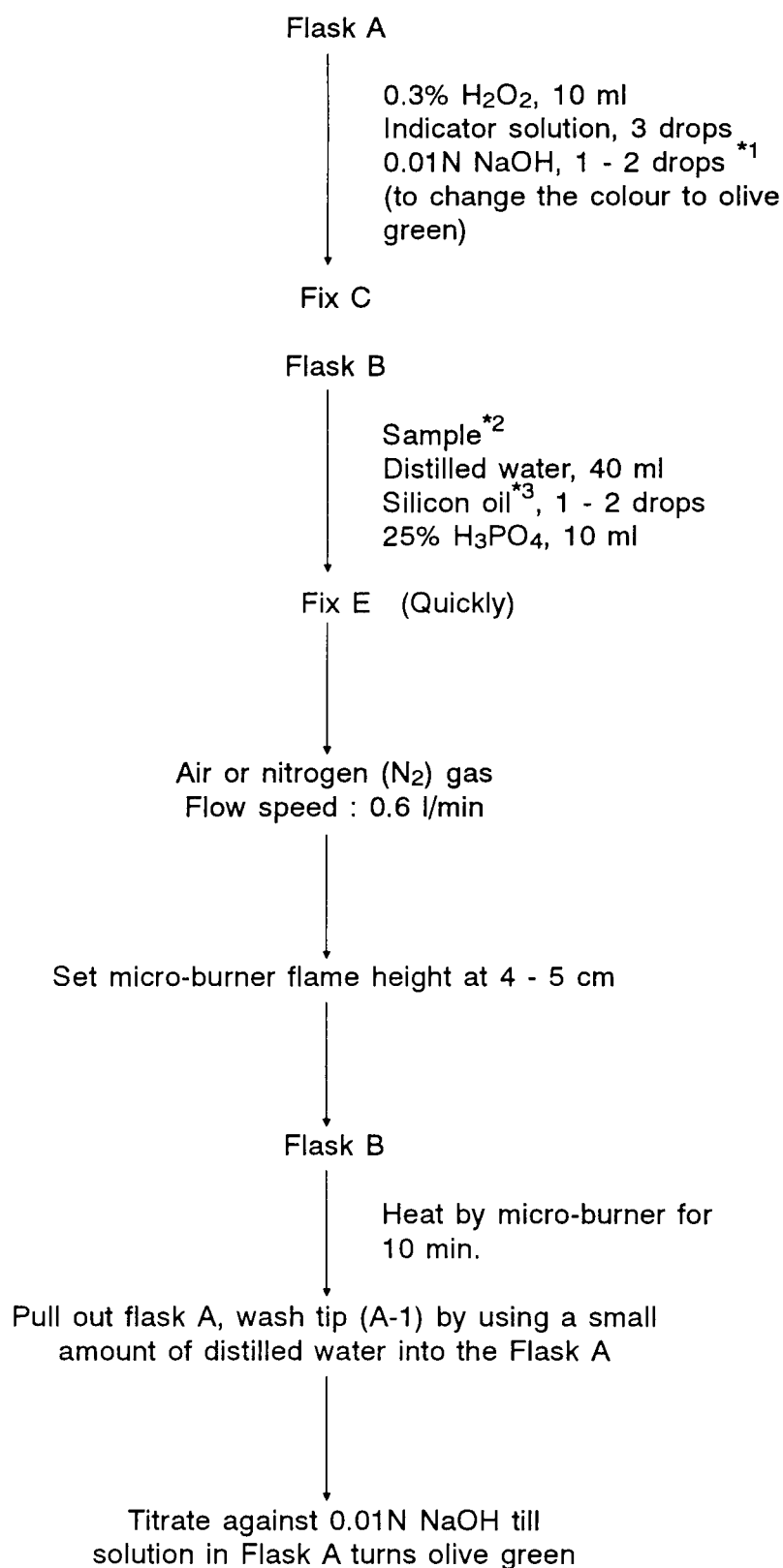


Fig. 2. Analytical procedure for the analysis of SO<sub>2</sub> by Rankine method.