EXPERIMENT 4 DETERMINATION OF SULPHUR DIOXIDE

Structure

4.1 INTRODUCTION

Sulphur dioxide and sulphites are versatile food preservatives having several beneficial functions. Sulphur oxide added to food products as preservative may exist as undissociated sulphurous acid, as free bisulphite ion, as free sulphite ion, and/or as combined SO_2 in the form of hydroxy sulphonates. However, they can cause harmful effects if consumed in higher quantities. Therefore, like for all other preservatives, maximum permissible limits of sulphites in foods have been laid down. Besides, sulphites are not permitted in all foods.

There are two methods used for the estimation of sulphites in foods. Both of them make use of the reducing property of sulphur dioxide. In one method, iodine is used to oxidize sulphur dioxide (sulphurous acid to sulphuric acid in aqueous solution) and in the other method, hydrogen peroxide is used for the oxidation reaction after liberating sulphur dioxide from the product. The latter method is more reliable and hence followed widely.

Objectives

After studying and performing this experiment, you should be able to:

- estimate the sulphur dioxide content of food products by the distillation method.

4.2 EXPERIMENT

4.2.1 Principle

This method measures total sulphur dioxide in food products. Sulphites present in the product are liberated as sulphur dioxide by boiling with hydrochloric acid. The liberated sulphur dioxide is absorbed in hydrogen peroxide solution, which oxidizes it to sulphuric acid. Sulfite content is directly related to generated suphuric acid, which is determined by titration with standard sodium hydroxide solution.

The reactions involved are:

$$SO_2 + H_2O \rightarrow H_2SO_3$$

$$H_2O_2 + SO_2 \rightarrow H_2SO_4$$

4.2.2 Requirements

Apparatus

a) All glass distillation apparatus for determination of sulphur dioxide shown in the diagram below.

Diagram: SR p 307

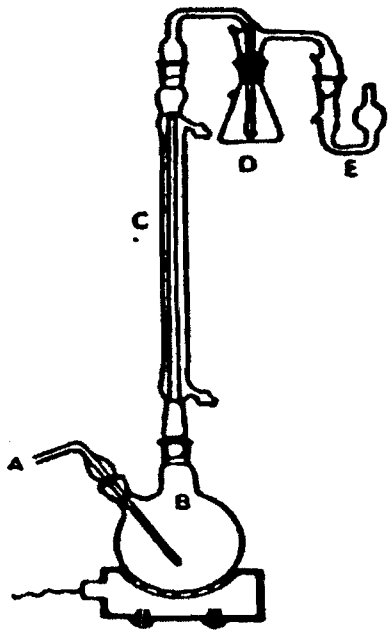

Figure 4.1: All Glass Distillation Apparatus for Determination of Sulphur Dioxide. A) Glass Inlet Tube, B) 500ml Round-Bottomed Flask, C) Condenser, D) 250 ml Conical Flask, E) Trap

b) Burette: - 10 ml

Reagents

a) **Aqueous Hydrochloric Acid:** -4M. For each analysis, prepare 90 ml solution by adding 30 ml HCl to 60 ml deionized water.

b) **Methyl Red Indicator:** -Dissolve 250 mg methyl red in 100 ml ethanol.

c) **0.05 N NaOH Solution.**

d) **3% Hydrogen Peroxide Solution:** -. For each analysis, dilute 3 ml reagent grade 30% H_2O_2 to 30 ml with distilled water. Just prior to use, add 3 drops methyl red indicator and titrate with 0.01N NaOH to yellow end point.

e) **Nitrogen Gas:** -High purity, used with regulator to maintain flow of 200 ml/min.

4.2.3 Procedure

Circulate cold water through condenser of the distillation apparatus. Add from a graduated cylinder, 20ml of 3% hydrogen peroxide solution to the conical flask (D) and 5 ml to the trap (E). Assemble the apparatus and connect condenser. Weigh 50g of blended sample into the round-bottomed flask (B) through gas inlet tube joint, using 300ml of water. Replace the inlet tube immediately, making sure all connections are well greased and tight. Remove the inlet tube, and slowly add 20ml of 4N HCl. Ensure that bubbles of nitrogen gas enter the receiving flask through the gas inlet tube. If not, check joints for leaks. Adjust nitrogen to give a flow of 15 to 20 bubbles per minute through the tube. Heat the content of the flask to boil and adjust the heater to give a slow boil. Continue

boiling for 30 mins. Stop heating and disconnect the assembly and remove the conical flask and the trap containing hydrogen peroxide. Transfer the hydrogen peroxide solution from the trap into the conical flask and rinse the trap with water and transfer the rinsing to the flask.

Determination

Add 3 drops of the indicator. Immediately titrate contents of conical flask (D) with 0.05N NaOH to yellow end point that persists for about 20 seconds. Compute sulfite content, expressed in mg SO_2/Kg food (ppm).

4.2.4 Observations

Weight of the sample = W = —— g

Normality of the NaOH solution = N

Volume of NaOH (titre) = V = —— ml

4.2.5 Calculations

1 ml of 0.05N NaOH = 1.6 mg of SO_2

$$\text{Therefore, V ml of N normal NaOH} = \frac{(V \times N \times 1.6)}{0.05} = (V \times N \times 32) \text{ — mg } SO_2$$

Since (V × N × 32) mg SO_2 is present in W g of the sample

$$\text{Therefore, } SO_2 \text{ (mg) in 1 Kg of the sample (ppm)} = \frac{(V \times N \times 32)}{W} \times 1000$$

4.2.6 Result

SO_2 in the sample = ppm = mg per kilogram.

4.3 PRECAUTIONS

The general precautions mentioned in the course 'Introduction' and those indicated in the experiments should be followed meticulously.

Handle the all glass distillation apparatus very carefully. It may easily break because the joints are rigid.