DETERMINATION OF SORBIC ACID, BENZOIC ACID AND DEHYDROACETIC ACID BY STEAM DISTILLATION AND UV SPECTROPHOTOMETRIC METHOD

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INTRODUCTION

The ultraviolet spectrophotometric method can be used for both qualitative and quantitative tests. The quality and quantity of the contents can be determined by measuring the specific preservatives such as sorbic acid, benzoic acid and dehydroacetic acid at specific maximum UV absorption wavelength. This method is very useful for the effective determination of many samples. However, in using this method, sometimes off odours are emitted.

In principle, preservatives from foods are extracted by steam distillation. The fraction is then purified by solvent extraction. Each acidified preservative from the extract solution is then measured at the specific absorption wavelength using UV spectrophotometer.

APPARATUS

1. Analytical balance
2. Steam distillation apparatus (Fig. 1)
3. UV absorption spectrophotometer
4. Shaker

REAGENTS

1. Buffer solution

   Add 50 ml of 2M KCl to 10.6 ml of 2N HCl and make up to 200 ml with distilled water.

2. Standard solution of sorbic acid

   Dissolve 50 mg sorbic acid in 4.5 ml of 0.1N NaOH and make up to 100 ml with distilled water. From this, make a 100 times dilution with distilled water (take 5 ml to make up to 500 ml with distilled water). 1 ml of this sorbic acid standard solution contains 5 ug of $C_2H_8O_2$.

3. Standard solution of benzoic acid

   Dissolve 100 mg benzoic acid in 8.5 ml of 0.1N NaOH and make up to 100 ml with distilled water. 1 ml of this benzoic acid standard solution contains 10 ug of $C_7H_6O_2$.

4. Standard solution of dehydroacetic acid

   Dissolve 100 mg dehydroacetic acid in 6 ml of 0.1N NaOH and make up to 100ml with distilled water. From this, make a 50 times dilution with distilled water (take 10 ml to make up to 500 ml with distilled water). 1 ml of this dehydroacetic acid standard solution contains 20 ug $C_8H_8O_4$.

PROCEDURE

Sample Preparation
(Fig. 2)

1. Weigh 50.0g sample in beaker. If the sample is a solid, add 100 ml of distilled water and mix well.

2. Adjust to pH 7 using 10% NaOH or 10% HCl.

3. Put sample into round bottomed flask (500 ml). Add 15 ml 15% tartaric acid (pH 2 - 3), 60 g NaCl, 1 drop of silicon oil and make up to 200 ml with distilled water.

4. Steam distil at a rate of 10 ml/ min. Collect 500 ml of distillate.

5. Pipette 50 ml of distillate into separatory funnel. Add 4 ml 10% HCl, 10 g NaCl, and extract 3 times with 40 ml, 30 ml and 30 ml ether respectively.

6. Drain the ether extract and pool the 3 extracts.

7. Wash ether extract with 15 ml distilled water.

8. Drain ether layer and discard water layer.

9. Add 20 ml of 1% $NaHCO_3$ and shake thoroughly.

10. Drain the $NaHCO_3$ extract and repeat Step 9. Pool the 2 $NaHCO_3$ water based extracts. The ether extract can be used for paraoxy-benzoic acid determination.

11. Neutralize the water based $NaHCO_3$ extract with 10% HCl and make up to 50 ml with distilled water. This is sample solution I and is used for benzoic acid, sorbic acid and dehydroacetic acid tests.

Analytical Procedure

1. Qualitative analysis

   1.1 Pipette 10 ml of each sample solution I, except standard solution into a test tube.

   1.2 Add 2 ml of buffer solution and 2 ml of distilled water to each sample solution.

   1.3 Measure absorption using UV Spectrophotometer at wavelength spectrum of 220 - 320 nm with liquid layer height of 10 mm in cuvette.

   1.4 Compare readings of sample solutions with those of standard solutions.

   1.5 A control solution containing a mixture of distilled water with buffer solution should be used. However, if the concentration is too high, use buffer solution to dilute 10 times and test again.

2. Quantitative analysis

Procedure same as that for Qualitative analysis.

CALCULATION

$$\text{Sorbic acid (g/kg)} = 5 \times \frac{A\,(265\ nm)}{As\,(265\ nm)} \times \frac{1}{2} \times \frac{1}{\text{sample (g)}}$$

$$\text{Benzoic acid (g/kg)} = 10 \times \frac{A\,(230\ nm)}{As\,(230\ nm)} \times \frac{1}{2} \times \frac{1}{\text{sample (g)}}$$

$$\text{Dehydroaceticacid (g/kg)} = 20 \times \frac{A\,(308\ nm)}{As\,(308\ nm)} \times \frac{1}{2} \times \frac{1}{\text{sample (g)}}$$

where,

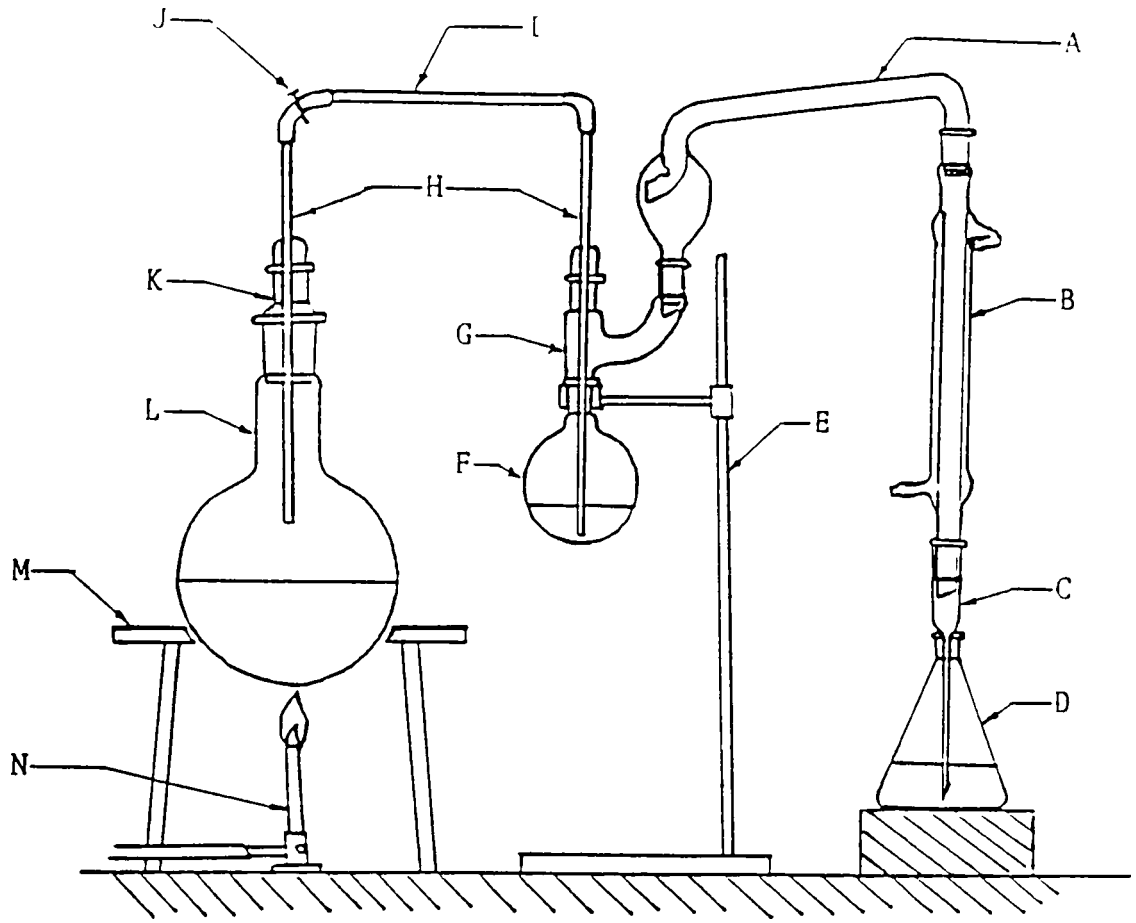
A = UV absorption of sample solution at wavelengths 230, 265 and 308 nm (Fig. 3).

As = UV absorption of standard solution at wavelengths 230, 265 and 308 nm.

Detection limit : 5 ppm (0.005 g/kg)

REFERENCE

Standard Methods of Analysis for Hygiene Chemist - With Commentary - authorized by the Pharmaceutical Society of Japan, Kanehara Shippan K.K. (1990).



A	:	Splash head	H	:	Steam inlet tube
B	:	Liebig condenser	I	:	Glass tubing
C	:	Straight delivery adapters	J	:	Clip
D	:	Conical flask	K	:	Reduction adaptors
E	:	Retort stand	L	:	Round bottomed flask, 2L
F	:	Round bottomed flask, 250 ml	M	:	Tripod stand
G	:	Two neck multiple adaptor	N	:	Bunsen burner

Fig. 1. Steam distillation apparatus.

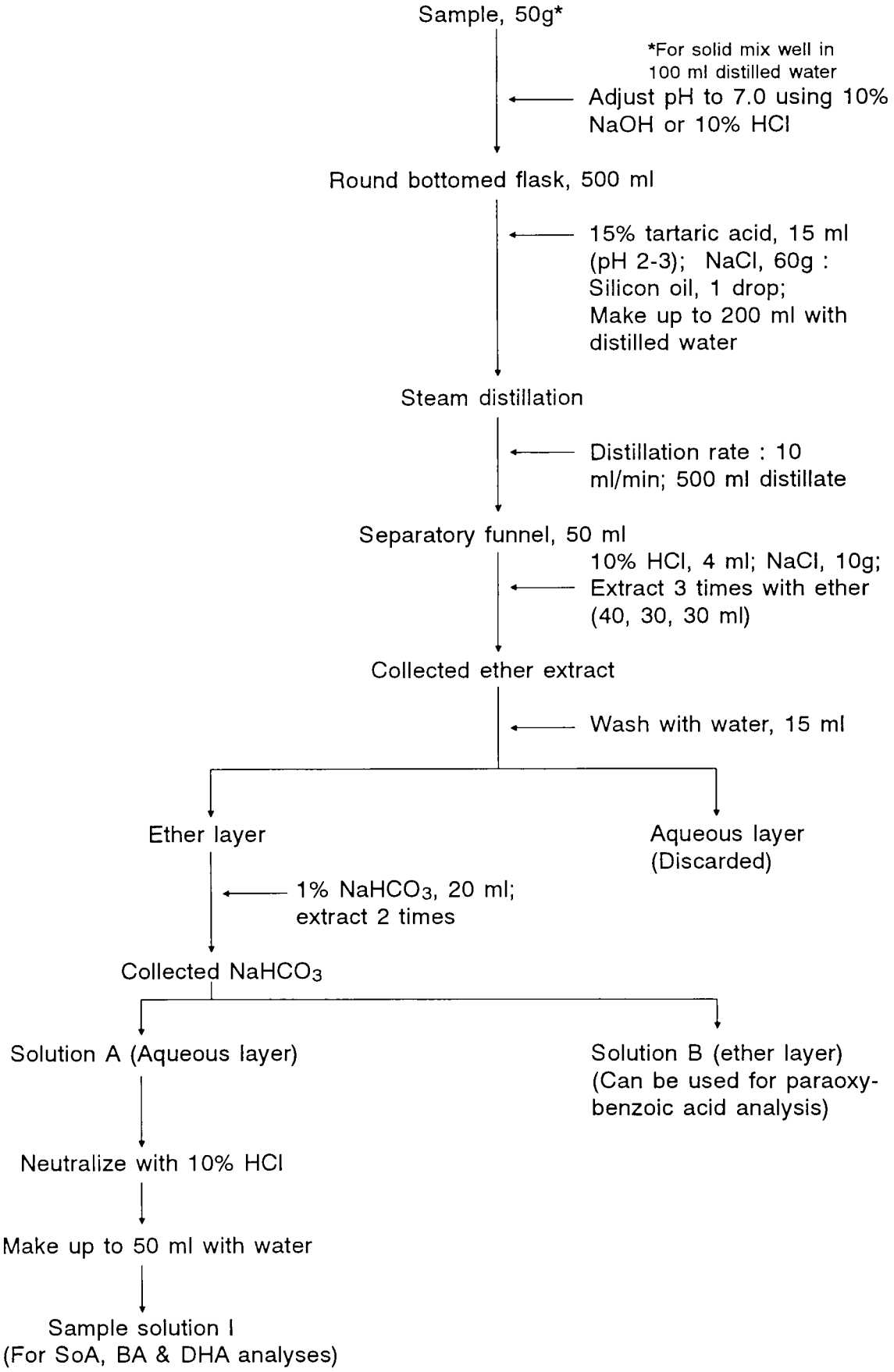


Fig. 2. Sample preparation for sorbic acid, benzoic acid and dehydroacetic acid analyses.

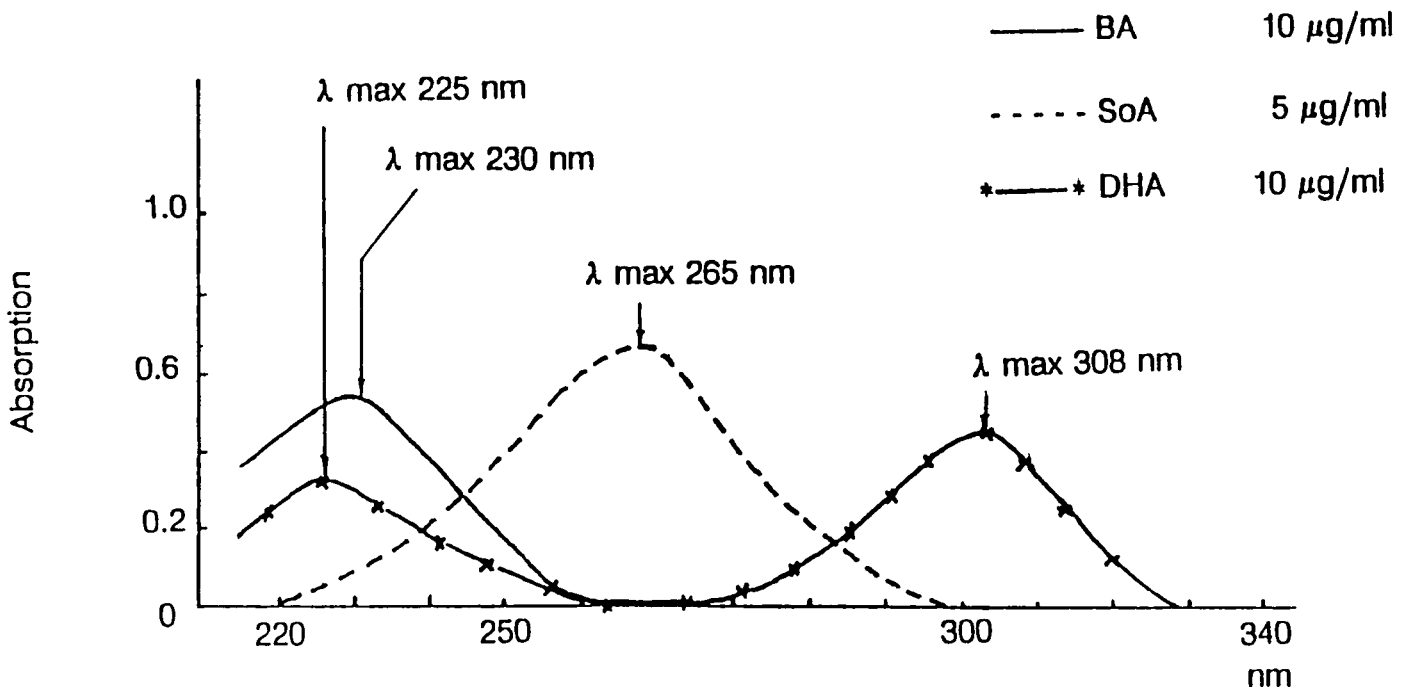


Fig. 3. UV absorption spectrum for standard solutions of each preservative at pH 2.