DETERMINATION OF BENZOIC ACID

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INTRODUCTION

Benzoic acid is used as a preservative in the form of the sodium or potassium salt. The amount present is calculated as the acid itself. The undissociated acid is effective in retarding the growth of yeasts and moulds. It is therefore used as an anti-fungal agent in some food.

In Singapore, benzoic acid is not permitted in fish products. Its content should therefore be absent in the products.

PREPARATION OF SAMPLE

I GENERAL METHOD

Transfer 150 g minced sample into 500 ml volumetric flask. Add saturated NaCl solution. Use 10% NaOH solution to adjust the pH in flask to alkali (> pH 7).

Shake well manually and then with magnetic stirrer for about 2 hours. Filter. Retain filtrate for further test.

II SALTED DRIED FISH

Transfer 50 g minced sample into 500 ml volumetric flask with water. Make slightly alkaline with 10% NaOH solution using litmus paper. Stand for 2 hours shaking frequently or using magnetic stand and stirrer. Filter.

Pipette as large a measured portion of filtrate as possible (> 300 ml) into a second 500 ml volumetric flask. Add 30 g pulverised NaCl for each 100 ml solution. Shake until NaCl dissolves and make-up to the mark with saturated NaCl solution. Mix well and filter off precipitated protein and extraneous matter.

REAGENTS

- a) Saturated NaCl solution
- b) 10% NaOH
- c) Chloroform (GR grade)
- d) Conc. H₂SO₄
- e) Ethyl alcohol
- f) 0.05N NaOH
- g) Indicator phenolphthalein
- h) Diluted HCI (1part conc. HCL + 3 parts distilled water)

PROCEDURE

- 1. Pipette 100-200 ml filtrate from SAMPLE PREPARATION into a 500 ml separator. Neutralise with diluted HCl using litmus paper and add excess 5 ml HCl.
 - NOTE: With salted fish, protein usually precipitates on acidifying, but the precipitate does not interfere with extraction.
- 2. Extract carefully with CHCl₃, using successive portions of 70, 50, 40 and twice 30 ml.
- 3. Shake slowly using rotary motion to avoid formation of emulsion. The CHCl₃ layer will separate easily after a few minutes.
 - NOTE: If emulsion forms, break it by stirring CHCl₃ layer with glass rod or by drawing CHCl₃ layer into a second separator and giving 1-2 sharp shakes.
- 4. Carefully drain out as much **clear** CHCl₃ solution as possible after each extraction into a 400 ml beaker.
- 5. Leave the beaker of CHCl₃ to evaporate overnight in fume cupboard till dry.
- 6. Put beaker of dry residue in desiccator containing a small beaker of concentrated H₂SO₄ to remove the moisture in residue. Leave it for half day or overnight.
- 7. Dissolve residue of benzoic acid in 40 ml ethyl alcohol. Add few drops of indicator and 10 ml distilled water.
- 8. Titrate with 0.05N NaOH till end point. Carry out a blank titration without benzoic residue.

CALCULATION

1 ml 0.05N NaOH = 0.0072 g anhyd. Na benzoate

Therefore, Sample = (A-B) 0.0072 g anhyd. Na benzoate

where A = Sample - titrated volume (ml) of 0.05N NaOH

B = Blank - titrated volume (ml) of 0.05N NaOH

REFERENCES

David Pearson. The Chemical Analysis of food. 7th Edition, p 33.

Official Methods of analysis of the Association of Official Analytical Chemists (1984). p 376.