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# EXPERIMENT 5 ESTIMATION OF BENZOIC ACID

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## Structure

- 5.1 Introduction
  - Objectives
- 5.2 Experiment
  - 5.2.1 Principle
  - 5.2.2 Requirements
  - 5.2.3 Procedure
  - 5.2.4 Observations
  - 5.2.5 Calculations
  - 5.2.6 Result
- 5.3 Precautions

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## 5.1 INTRODUCTION

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Benzoic acid is the second most common preservative added to fruit and vegetable products. Being insoluble in water, it is added as the soluble sodium benzoate. The method commonly followed for the estimation of benzoic acid involves conversion of the salt into benzoic acid by acidification, extraction into an organic solvent and titrating against alkali.

### Objectives

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

- determine the benzoic acid content of foods by the extraction and alkali titration method.

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## 5.2 EXPERIMENT

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### 5.2.1 Principle

In sodium chloride solution of the sample, the benzoic acid present is converted into water-soluble sodium benzoate by the addition of NaOH. When the sodium benzoate solution is acidified with excess HCl, it is converted into benzoic acid. The water insoluble benzoic acid is extracted with chloroform. The chloroform is removed by evaporation and the residue containing benzoic acid, which appears as leafy crystals, is dissolved in neutral alcohol and titrated against standard NaOH to phenolphthalein end point.

### 5.2.2 Requirements

#### Equipment and Apparatus

Separating funnel, 500 ml	-1
Beaker, 250 ml	-2
Volumetric flask, 250 ml & 500 ml	-1 each
Burette, 50 ml	-1
Pipette, 25 ml	-1
Measuring cylinders, 50 ml and 10 ml	-1 each
Water-bath	-1
Chemical balance	-1
Desiccator	-1

Whatman No. 1 or No. 4 filter paper circles

#### Chemicals and Reagents

Diethyl ether,

Hydrochloric acid - (1+3)

Anhydrous sodium sulphate

Ethyl alcohol

Phenolphthalein indicator - 0.1% in alcohol

Sodium hydroxide solution - 0.05 N

Ferric chloride solution - 0.5%

Sodium chloride - saturated solution

Sodium hydroxide - 10 % solution

Chloroform

Sodium chloride - powder

### 5.2.3 Procedure

#### a) Detection

Transfer homogenized sample into a separating funnel. Acidify with 1+3 HCl. Extract with solvent ether. Collect ether in a dry conical flask. Evaporate ether to near dryness and completely evaporate under a current of dry air. To the white leafy crystals of benzoic acid add few drops of 0.5% neutral  $\text{FeCl}_3$  solution. A salmon coloured precipitate confirms presence of benzoic acid.

#### b) Determination

Take 50-75 g of the homogenized sample in a 500ml beaker. Add 200ml saturated solution of NaCl and 50g powder NaCl and stir well. Make the solution alkaline to litmus paper with 10% NaOH. Quantitatively transfer the solution to 250ml volumetric flask and make up to volume. Shake well. Let stand 2-3 hr. with frequent shaking. Filter through whatman No. 4 filter paper. Pipette 50ml filtrate into a separating funnel. Neutralize to litmus paper with HCl (1+3). Add 5ml in excess of the acid. Extract the solution thrice with 70, 50, 30 ml portions of chloroform. Shake funnel using rotary motion with each extraction. If emulsion forms, break it by (a) Stirring  $\text{CHCl}_3$  layer with a glass rod, or (b) draw  $\text{CHCl}_3$  layer into a second separating funnel and give one or two sharp shake. Pool clear  $\text{CHCl}_3$  extracts and transfer to separating funnel. Wash the chloroform extract with 5ml water. Draw the  $\text{CHCl}_3$  layer into a 250ml conical flask. (the  $\text{CHCl}_3$  extract may be passed through a bed of cotton and anhydrous  $\text{Na}_2\text{SO}_4$  to remove traces of moisture). Evaporate  $\text{CHCl}_3$  to near dryness on water bath. Dry completely under a current of dry air. Keep the flask overnight in a  $\text{H}_2\text{SO}_4$  desiccator. Remove and add 25ml neutral alcohol. Add 6-8 ml of water and one drop of phenolphthalein indicator and titrate against 0.05N NaOH.

### 5.2.4 Observations

Weight of the sample	= W = _____ g
Volume made up	= V = _____ ml
Volume taken for extracting with chloroform	= $V_1$ = _____ ml
Titre value	= $V_2$ = _____ ml
Normality of the NaOH	= N

### 5.2.5 Calculations

1000 ml 1N NaOH = 1 g equivalent of benzoic acid = 122 g benzoic acid

or 1 ml 1 N NaOH = 122 mg benzoic acid

Therefore ppm (mg per Kg) of benzoic acid in the product =

$$\frac{\text{Vol. of alkali} \times \text{Normality of NaOH} \times \text{Vol. made up}}{\text{Vol. Taken for CHCl}_3 \text{ extraction} \times \text{Weight of sample}} \times 122 \times 1000$$

$$= \frac{V_2 \times N \times V \times 122 \times 1000}{V_1 \times W} = \text{ppm}$$

### 5.2.6 Result

The result is expressed as benzoic acid in ppm.

## 5.3 PRECAUTIONS

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

Never handle ether near a flame. The solvents should be handled only in a well-ventilated room or inside a hood with exhaust. Avoid inhaling the solvents directly.