EXPERIMENT 13
ESTIMATION OF FORMALDEHYDE

Structure

13.1 INTRODUCTION

Formaldehyde is usually available as an aqeous solution containing 33 to 37 percent by weight of formaldehyde. This aqeous solution is known as formalin. Here, we will describe one of the methods which has frequently to be applied for the estimation of formaldehyde in commercial formalin solution. In this method formaldehyde is oxidised quantitatively to formic acid by excess of iodine in alkaline solution. Then liberated iodine is titrated with standard sodium sulphate thiosolution.

The unreacted hypo-iodite is then acidified and the liberated iodine is titrated against sodium thiosulphate solution using starch as indicator. Further experimental details are given in next section.

Objectives

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

- determine the amount of formaldehyde as in given formaldehyde solution
- describe the oxidation reation of formaldehyde with sodium hypoiodite solution, and
- perform iodometric titration

13.2 PRINCIPLE

Formaldehyde may be estimated in solution by oxidising it to formic acid by means of a known quantity (in excess) of iodine dissolved in an excess of NaOH solution (hypoiodite solution). The formic acid thus formed is neutralised by the alkali present. The unreacted hypoiodite is then acidified with HCl and the liberated iodine is titrated with standard sodium thiosulphate solution using starch as indicator.

$$I_2 + 2NaOH \longrightarrow NaOI + NaI + H_2O$$

$$HCHO + NaOI + NaOH \longrightarrow HCOONa + NaI + H_2O$$

$$HCHO + I_2 + 3NaOH \longrightarrow HCOONa + 2NaCl + I_2 + H_2O$$

$$NaOI + NaI + 2HCl \longrightarrow 2NaCl + I_2 + H_2O$$

$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$$

13.3 REQUIREMENTS

Apparatus		Chemicals
Burette (50 cm^3)	- 1	Formalin solution
Pipette (25 cm^3)	- 1	Iodine
Vol. flasks (250 cm^3)	- 1	Sodium thiosulphate
Conical flask (250 cm^3)	- 1	Sodium hydroxide
Weighingbottle	-1	Conc.hydrochloric acid
Funnel (small)	- 1	Starch indicator
Wash-bottle for distilled water	- 1	Copper sulphate
Test-tube	- 1	
Burette stand	- 1	

Solutions Provided

i) **Iodine solution. 0.1 *M*:** Prepare iodine solution. (0.1*M*) of iodine by dissolving 3.17 g of it in 250 cm^3 volumetric flask in distilled water. Standardise it by titrating against standard sodium thiosulphate (0.1*M*) solution.

ii) **Sodium thiosulphale solution 0.1 *M* :** It is prepared by dissolving 6.25g sodium thiosulphate pentahydrate in distilled water in a 250 cm^3 volumatric flask.

iii) **Sodium hydroxide solution. 2*M*:** It is prepared by dissolving 40 gm sodium hydroxide in 500 cm^3 volumetic task distilled water.

iv) **Conc. hydrochloric acid. 2*M*:** It is prepared by taking 45 cm^3 of Conc.HCl and making up to the mark with in 250 cm^3 volumetric flask.

v) **Starch solution:** Make a paste of 1.0g of starch with a little water and pour the suspension with constant stirring into 100 cm^3 of boiling water.

13.4 PROCEDURE

i) **Formalin solution:** Weigh out accurately about 1.0 g of formalin solution, transfer it in a 250 cm^3 volumetric flask and make up to the mark with distilled water.

ii) **Titration with Iodine solution (Blank titration):** Pipette out 50 cm^3 of iodine solution in a 250 cm^3 conical flask. Titrate this solution with standard sodium thiosulphate solution. Sodium thiosulphate solution can be standarised by titrating against dichoromate solution. The procedurals details for the strandardisation is given in Experiment 9b. Repeat the titration to get atleast two concordant readings. Record the observation in Observation Table-I.

iii) **Titration with formaline Solution:** Pipette out 25 cm^3 of unknown formalin solution in a 250 cm^3 conical flask and add 50 cm^3 of 0.1*M* iodine solution. Solution develops a dark-brown colour. Now add 2*M* NaOH solution from the burette into the conical flask until the solution becomes pale yellow in colour. Shake the contents of the flask and allow to stand for 15 minutes. Acidify with 40 cm^3 of 2*M* hydrochloric acid to liberate the remaining iodine. Titrate this solution with sodium thiosulphate solution (0.1*M*) using starch as indicator.

Repeat the titration to get atleast two Concordant readings.

13.5 OBSERVATION

Observation Table I
Iodine Solution Vs. Sodium Thiosulphate Solution

Sl. No.	Volume of iodine Solution in cm^3	Burette reading Initial	Burette reading Final	Volume of Sodium thiosulphate in cm^3 (Final–Initial)
1	50			
2	50			
3	50			

Observation Table II
Formation and Iodine Solution Vs. Sodium Thiosulphate Solution

Sl. No.	Volume of Iodine solution added in cm^3	Burette reading Initial	Burette reading Final	Volume of Sodium thiosulphate in cm^3 (Final–Initial)
1	50			
2	50			
3	50			

13.6 CALCULATIONS

(a) Volume of 0.1*M* iodine solution added = 50 cm^3

(b) Volume of 0.1*M* sodium thiosulphate solution used in titration = *V* cm^3

Since V cm^3 0.1*M* sodium thiosulphate ≡ *V* cm^3 0.1*M* iodine

Hence, the volume of 0.1*M* iodine used = (50–*V*) cm^3

According to the equation of the reaction

$$HCHO + I_2 + 3NaOH \longrightarrow HCOONa + 2NaI + H_2O$$

From the above equation, it will be seen that 1 cm^3 of the *M*/10 iodine solution used in the oxidation is equivalent to 0.00150 g of formaldehyde.

Hence (50-*V*) cm^3 of 0.1*M* iodine solution = (50 – *V*)×0.00150 g of HCHO

$2\ dm^3\ 0.1\ M I_2 \equiv 1 dm^3$ of 0.1 *M* HCHO

$1\ dm^3$ of 0.1 $MI_2 \equiv 30/2 \times 10$ g *H*CHO

$1\ cm^3$ of 0.1 $MI_2 \equiv .00150$ g *H*CHO

25 cm^3 of supplied solution contains = (50 – *V*)×0.00150 g of HCHO

Percentage of formaldehyde in the given solution = $\frac{(50 - V)\times 0.00150\times 100}{25}$ =... %

13.7 Result

Percentage of formaldehyde in the solution =..... %

In next experiment we will describe the analysis of the oils and fats. This experiment will tell us how we can use organic quantitative methods to determine different parameters such as saponification value, iodine value and acid value of given oils or fat.