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# EXPERIMENT 7 ESTIMATION OF RESIDUAL CHLORINE IN WATER

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## 7.1 INTRODUCTION

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Water is routinely chlorinated to make it microbiologically safe. When chlorine is added to water other than distilled water, initially a small amount reacts with impurities in the water and does not show as residual chlorine. This is called the chlorine demand of the water, which has no germicidal effect. Chlorine added subsequently remains as the residual chlorine (free available and combined available chlorine), which is important for disinfection. The residual chlorine levels of water used for different purposes have been specified.

The most common method for routine estimation of residual chlorine is the iodometric method, which you will learn in this practical.

### Objectives

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

- determine the residual chlorine content in water; and
- follow the necessary sampling procedures.

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## 7.2 EXPERIMENT: RESIDUAL CHLORINE BY IODOMETRIC METHOD

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### 7.2.1 Principle

Chlorine liberates free iodine from potassium iodide solution in acidic pH quantitatively. The liberated iodine is determined by titration with standard sodium thiosulphate solution. As chlorine in aqueous solution is not stable, the determination of chlorine must be performed immediately after sampling. Care should be taken to avoid excessive exposure of the water sample to sunlight and agitation.

## 7.2.2 Requirements

### Apparatus / Glassware

#### Analytical Balance

Burette, 25 ml

Volumetric flask, 100 and 250 ml

Conical flask, 250 ml

Measuring cylinder, 500 ml

White porcelain dish, 500 ml

Beaker, 250 ml

#### Reagents

- i) Acetic acid, glacial.
- ii) Potassium iodide crystals.
- iii) Starch indicator.
- iv) N sodium thiosulphate solution: Dissolve 24.8192 g of sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) in 200 ml water and transfer to a 1 litre volumetric flask and make up to volume. Standardize the solution with potassium dichromate. Weigh 0.20 to 0.23 g  $\text{K}_2\text{Cr}_2\text{O}_7$  and transfer to a 250 ml beaker using about 150 ml water. Add 2 g potassium iodide and mix. Add 20 ml of 1 N HCl, swirl and allow to stand for 10 min. Titrate with the thiosulphate solution adding 1 ml of 1% starch solution towards the end of titration and complete titration where the solution changes from blue green to light green.

$$\text{Normality of sodium thiosulphate solution} = \frac{\text{Wt of } \text{K}_2\text{Cr}_2\text{O}_7 \text{ (g)} \times 1000}{\text{Vol. of } \text{Na}_2\text{S}_2\text{O}_3 \text{ (ml)} \times 49.037}$$

Prepare 0.01 N working standard by diluting the 0.1 N thiosulphate solution.

## 7.2.3 Procedure

Take a suitable volume of the water sample into a porcelain dish or beaker. For water containing 1 mg / litre or less chlorine take 1000 ml and for 1 to 10 mg / litre take 500 ml. The titre value of 0.01 N thiosulphate should not be more than 20 ml. Add 5 ml glacial acetic acid followed by 1 g potassium iodide, stir and titrate with 0.01 N thiosulphate solution until the yellow colour of the liberated iodine is almost disappears. Add 1 ml starch solution and titrate until the blue colour is discharged. Do not carry out the titration in direct sunlight. Blank titration can be carried out by taking equal volume of distilled water.

## 7.2.4 Observations

Volume of water taken for titration = V = — ml

Volume of thiosulphate solution required (titre) =  $V_1$  = — ml

Normality of sodium thiosulphate solution = 0.01 N

### 7.2.5 Calculations

1000 ml 1N sodium thiosulphate = 35.46 g chlorine (i.e. 1 g mole of chlorine)

or 1 ml 1N sodium thiosulphate = 35.46 mg chlorine

or  $V_1$  ml of 0.1 N sodium thiosulphate =  $V_1 \times 0.01 \times 35.46$  mg chlorine

Therefore, residual chlorine content of the water (mg per litre)

$$\begin{aligned} &= \frac{V_1 \times 0.01 \times 35.46 \times 1000}{V} \\ &= \frac{V_1 \times 354.6}{V} \end{aligned}$$

### 7.2.6 Result

Residual chlorine content of the water sample = mg per litre or ppm.

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## 7.3 PRECAUTIONS

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