

## EXPERIMENT 5

### REDOX TITRATION: TITRATION USING SODIUM THIOSULPHATE

#### Objectives

1. To prepare a standard solution of potassium iodate for use to determine the concentration of sodium thiosulphate solution accurately.
2. To acquire the proper techniques of carrying out a titration.

#### Introduction

Redox titrations using sodium thiosulphate as a reducing agent is known as iodometric titration since it is used specifically to titrate iodine. The reaction involved is:



In this equation  $I_2$  has been reduced to  $I^-$  :-



The iodine/thiosulphate titration is a general method for determining the concentration of an oxidising agent solution. A known volume of an oxidising agent is added into an excess solution of acidified potassium iodide. The reaction will release iodine:-

Example:

(a) With  $KMnO_4$



(b) With  $KIO_3$



The iodine that is released is titrated against a standard thiosulphate solution. From the stoichiometry of the reaction, the amount of iodine can be determined and from this, the concentration of the oxidising agent which released the iodine, can be calculated.

In an iodometric titration, a starch solution is used as an indicator as it can absorb the iodine that is released. This absorption will cause the solution to change to a dark blue colour. When this dark blue solution is titrated with the standardised thiosulphate solution, iodine will react with the thiosulphate solution. When all the iodine has reacted with the thiosulphate solution, the dark blue colour will disappear. So, the end point of the titration is when the dark blue colour disappears.

Iodine is usually dissolved in water by adding an excess of KI so that  $KI_3$  which has similar properties to iodine is formed.



Oxidising agents used other than thiosulphate are iron (II) salts, arsenic III oxide, sulphur dioxide and stibium (III) oxide. The following are reactions of sulphur dioxide and stibium (III) oxide with iodine:



Experimental aspects to be considered:

1. The indicator (starch) in the iodometric titration is not added in the early stage of the experiment as in acid-base titrations. Starch is only added after titration has begun, i.e. when the colour of the reaction mixture has changed from brown to a light yellow colour.

Starch is a colloid that can absorb iodine and form a complex. When this happens, it would be difficult to release the iodine when titrating with the thiosulphate. This will influence the determination of the end point. Hence, the addition of the starch should only be done when the solution is light yellow in colour. At this point, there is only a small amount of released iodine left and the complex formed would also be small in quantity and

can easily react with the thiosulphate.

2. As soon as the solution is mixed with KI, titrate immediately with the thiosulphate. This will prevent the iodine from evaporating.

### **Apparatus**

### **Chemical Reagents**

weighing bottle/50 mL beaker

Potassium iodate crystals

glass rod

Potassium iodide solids

analytical balance

H<sub>2</sub>SO<sub>4</sub> 1.0M

pipette filler

Starch solution

filter funnel

Sodium thiosulphate solution 0.1 M

250 mL volumetric flask

Distilled water

burette

25 mL pipette

250 mL conical flask

5 mL and 25 ml measuring cylinder

100 mL beaker

### **Method**

#### **A. Preparation of Potassium Iodate Solution (To be shared by two students)**

1. Weigh approximately 0.75g of potassium iodate crystals in a 50 mL beaker.
2. Add 25 mL of distilled water into the beaker and stir with a glass rod to dissolve all the potassium iodate.
3. Pour the potassium iodate solution through a filter funnel into a 250 mL volumetric flask. Rinse the beaker with distilled water and pour this water into the volumetric flask.
4. Add in more distilled water to the volumetric flask up to the mark on the neck of the flask. Put the stopper in place and shake the flask until you get a homogenous solution.

The standard solution is now ready for use in part B.

#### **B. Standardisation of 0.1 M Sodium Thiosulphate Solution (To be done**

### **individually)**

1. Rinse and fill a clean burette with 0.1 M sodium thiosulphate solution that is to be standardised. Make sure that there are no air bubbles in your burette.
2. Record the initial reading of your burette. (Note: You should read your burette at eye level. Accuracy of reading the burette reading should be within the range of  $\pm 0.05$  mL).
3. Pipette 25.0 mL of the standard potassium iodate solution that has been prepared in part A into a 250 mL conical flask.
4. Weigh approximately 1 g of potassium iodide crystals and add it into the solution in the conical flask.
5. Then add in 10.0 mL of 1.0 M sulphuric acid solution and swirl the conical flask until all the KI has dissolved.
6.
  - (i) Titrate immediately the released iodine with the sodium thiosulphate solution while swirling the conical flask until a light yellow solution is obtained.
  - (ii) Dilute this solution with distilled water until the total volume is about 100 mL.
  - (iii) Add in 1 mL of the starch solution and continue titrating until the blue colour disappears and the solution becomes colourless. This is the end point of the titration process.
7. Record the final reading of your burette.
8. Repeat the titration three times.

### **Calculation**

1. Write the equations for reactions between
  - (i) iodate ion and iodide
  - (ii) iodine and thiosulphate ion
2. Calculate the molarity of  $\text{KIO}_3$  solution.
3. From the reaction equations in 1 (i) and (ii) determine the mole ratio between the iodate ion and thiosulphate ion.

4. Calculate the molarity of the sodium thiosulphate solution using the formula:

$$\frac{M_1 V_1}{a} = \frac{M_2 V_2}{b}$$

#### EXPERIMENT 4

Appendix A

#### ***REDOX TITRATION: TITRATION USING SODIUM THIOSULPHATE***

STUDENT'S NAME: \_\_\_\_\_

MATRIC NO: \_\_\_\_\_

GROUP: \_\_\_\_\_

DATE: \_\_\_\_\_

LECTURER'S NAME: \_\_\_\_\_

AIM OF EXPERIMENT: \_\_\_\_\_

\_\_\_\_\_  
THEORY: (Provide the chemical equations used in calculating the values and a summary of the experiment.)

## Results

A: Preparation of standard potassium iodate ( $\text{KIO}_3$ ) solution

$$\begin{array}{rcl} \text{Weight of beaker} + \text{KIO}_3 & & = \\ \text{Weight of beaker} & & = \\ \text{Weight of KIO}_3 & & = \end{array}$$

B: Standardization of sodium thiosulphate solution

	Gross	1	2	3
Final burette reading				
Initial burette reading				
Volume of $\text{Na}_2\text{S}_2\text{O}_3$ used				

$$\text{Average volume of } \text{Na}_2\text{S}_2\text{O}_3 \text{ used} =$$

### Calculation:

A: Calculate the concentration of  $\text{KIO}_3$  solution

Relative atomic mass: Potassium = 39.1 Iodine = 126.9 Oxygen = 16.0

B: (i) Write the chemical equations for these reactions :

(a) Iodate ion and iodide

(b) Iodine and thiosulphate

(ii) Determine the mole ratio of the iodate ion and thiosulphate:

\_\_\_\_\_ : \_\_\_\_\_  
(iodate ion) (thiosulphate)

(iii) By using the formula,  $M_1V_1 = M_2V_2$ , calculate the molarity of sodium thiosulphate.

a b

**Results:**

A: Molarity of  $KIO_3$  is: \_\_\_\_\_ M

B: Molarity of  $Na_2S_2O_3$  is: \_\_\_\_\_ M

**Solution To The Exercise:**