EXPT. 4 POTENTIOMETRIC TITRATION OF Fe^{2+} WITH $Cr^2O^{2-}_7$

Structure

4.1 INTRODUCTION

A detail of potentiometry is already disused the unit 2 of MCH -004 course. In this experiment you are going to perform a redox titration. EMF change during the titration is measured by potentiometer. The equivalence point of the titration is detected by plotting a titration curve similar to the previous experiment.

Objectives

After performing this experiment you will be able to:

- Discuss how the potential changes with relative concentration of oxidised/reduced from,
- perform a redox titration of ammonium iron (II) sulphate using potassium dichromate as oxidizing agent,
- determine the equivalence point of the redox titration by plotting titration curve using potential change values and amount of oxidizing agent added during titration,
- estimate the strength of iron (II) ions in the given solution,
- state the advantages of potentiometry in redox titrations, and
- practice the precautions while performing a potentometric titration experiment.

4.2 PRINCIPLE

This is an example of redox titration and is based on the oxidation-reduction reaction between the titrand and the tirant. Here the end pint is detected using a potentiometer.

From the Nernst equation, you know that the potential of a given reaction will depend on the relative concentration of oxidised/reduced from.

During their titration, the solution potential changes due to the change in the concentration of oxidised/reduced form. At one stage, where either of the form is absent i.e. at the end point, there is a sharp change in potential.

Potassium dichromate is an oxidising agent and in acid medium; it follows the half reaction to give Cr (III) as the reduction product.

$$Cr_2O^{2-}_7 + 14H + 6e \rightleftharpoons 2\,Cr^{3+} + 7\,H_2O \quad \ldots (1)$$

While Fe^{2+} which is used to titrate $K_2Cr_2O_7$ gets oxidised to Fe^{3+} as per the reaction

$$Fe^{2+} \rightleftharpoons Fe^{3+} + e \quad \ldots (2)$$

The overall ionic equation of this titration can be obtained by adding the above two:

$$Cr_2O_7^{2-} + 6\ Fe^{2+} + 14H^+ \rightleftharpoons 6\ Fe^{3+} + 2Cr^{3+} + 7\ H_2O \quad \text{... (3)}$$

4.3 REQUIREMENTS

Apparatus:

Burette, pipette, volumetric flasks, beakers, magnetic stirrer, potentiometer, SCE, platinum indicator electrode, connecting wires etc.

Chemicals

Potassium dichromate, ammonium iron (II) sulphate and sulphuric acid

4.4 PROCEDURE

1. Using 100 cm^3 volumetric flasks prepare of 0.02 M potassium dichromate solution and 0.10 M ammonium iron (II) sulphate solution. You may have to add sufficient amount of dilute acid to prepare ammonium iron (II) sulphate solution.

2. Take 25 cm^3 of given Fe^{2+} solution and add 25 cm^3 dilute H_2SO_4 acid and 50 cm^3 of distilled water in a 250 cm^3 beaker.

3. SCE is used as the Reference electrode. Platinum metal foil, dipped in Fe^{2+} solution is used as the indicator electrode.

4. Carry out necessary connections as shown in Fig. 3.1.

5. Standardise the potentiometer using a standard cell before replacing it with the working cell.

6. Add 2 cm^3 of 0.02 M $K_2Cr_2O_7$ solution from burette, operate the magnetic stirrer for 2 minutes, stop it, wait it for 1 minute and measure the E.M.F.

7. Repeat the above step, each time adding two more cm^3 of $K_2Cr_2O_7$ at a time and go on noting the E.M.F. after each addition.

8. When the volume reached near about 1 cm^3 of the expected equivalence point (approximate), add the solution from burette in 0.5 cm^3 instalments and note the potential each time.

9. Continue adding these instalments even after the equivalence point (This can be easily observed from the change in measured potential). The change becomes very small. Continue for another 5-6 additions. Note the potential readings.

10. Record the observations in the Observation Table 1.Follow the same procedure for plotting the graphs and locate the equivalence point as given in Experiment No. 3.

Observations

Observation Table 1

Sr.No.	Volume (*V*) of $K_2Cr_2O_7$ added (cm^3)	EMF. of (*E*) the cell (millivolts)	ΔV	ΔE	$\Delta E/\Delta V$
1.	0.0		…	…	…
2.	2.0		…	…	…
3.	4.0				
4.	6.0		…	…	…
5.	8.0		…	…	…
6.	10.0		…	…	…
7.	12.0		…	…	…
8.	14.0		…	…	…
9.	16.0		…	…	…
10.	18.0		…	…	…
11.	20.0		…	…	…
12.	22.0		…	…	…
13.	24.0		…	…	…
14.	24.5		…	…	…
15.	25.5		…	…	…
16.	26.0		…	…	…
17.	26.5		…	…	…
18.	27.0		…	…	…
19.	28.0		…	…	…
20.	20.0		…	…	…

4.5 CALCULATIONS

From the plotted curve, the volume of $K_2Cr_2O_7$ used corresponding to the equivalence point = V cm^3 ……..

Apply the molarity equation to calculate molarity of potassium dichromate solution.

$$M_1V_1 = 6M_2V_2$$

Ferrous ammonium Sulphate $K_2Cr_2O_7$

$$M_2 = \frac{M_1V_1}{6V_2} \text{ mol } dm^{-3}$$

..... mol dm^{-3}

4.6 RESULTS

Strength of the potassium dichromate solution is …… mol dm^{-3}