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# Diploma in Pharmacy 1<sup>st</sup> Year

## Pharmaceutical Chemistry

### Experiment

To perform the assay of the given sample of ferrous sulphate by redox titration.

#### Aim:

To perform the assay of the given sample of ferrous sulphate by redox titration.

#### Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 33 - 36

#### Apparatus and Material Required :

Burette, burette stand, conical flask, volumetric pipette, beaker, volumetric flask, funnel, glass rod, and wash bottle, digital/analytical balance, ultrasonicator, ferrous sulphate ( $\text{FeSO}_4$ ), sulphuric acid ( $\text{H}_2\text{SO}_4$ ) and potassium permanganate ( $\text{KMnO}_4$ ).

#### Theory:

- The assay of ferrous sulphate is based on redox titration (cerimetry). Ferrous sulphate is a potent oxidant that is employed as a reducing agent. The known concentration solution of ceric ammonium sulphate is titrated with ferrous sulphate using ferroin solution as an indicator.
- In the presence of dilute  $\text{H}_2\text{SO}_4$ , ferrous sulphate is oxidised to ferric sulphate. A drop of  $\text{KMnO}_4$  is applied when the oxidation of ferrous sulphate is completed. The appearance of the permanent pink colour determines the titration's endpoint, and potassium permanganate ( $\text{KMnO}_4$ ) serves as a self-indicator.

## Chemical Equations

**Reduction Half Reaction:**  $2 \text{KMnO}_4 + 3 \text{H}_2\text{SO}_4 \rightarrow \text{K}_2\text{SO}_4 + 2 \text{MnSO}_4 + 3\text{H}_2\text{O} + 5 [\text{O}]$

**Oxidation Half Reaction:**  $2 \text{FeSO}_4 (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O} + \text{H}_2\text{SO}_4 + [\text{O}] \rightarrow \text{Fe}_2(\text{SO}_4)_3 + 2 (\text{NH}_4)_2\text{SO}_4 + 13 \text{H}_2\text{O} \times 5$

$= 2 \text{KMnO}_4 + 8 \text{H}_2\text{SO}_4 + 10 \text{FeSO}_4 (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O} \rightarrow \text{K}_2\text{SO}_4 + 2 \text{MnSO}_4 + 5\text{Fe}_2(\text{SO}_4)_3 + 10 (\text{NH}_4)_2\text{SO}_4 + 68 \text{H}_2\text{O}$

## Ionic Equation

**Reduction Half Reaction:**  $\text{MnO}_4^- + 5\text{e}^- + 8\text{H}^+ \rightarrow \text{Mn}^{2+} + 4\text{H}_2\text{O}$

**Oxidation Half Reaction:**  $\text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + \text{e}^- \times 5$

$= \text{MnO}_4^- + 5\text{Fe}^{2+} + 8\text{H}^+ \rightarrow \text{Mn}^{2+} + 5\text{Fe}^{3+} + 4\text{H}_2\text{O}$

## Procedure:

### Preparation of 0.05 M, Standard Solution of Ferrous Ammonium

**Sulphate** (Molar mass of  $\text{FeSO}_4 (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O} = 392 \text{ g mol}^{-1}$ ).

- 1) 49000 g of ferrous ammonium sulphate should be weighed and transferred into 250 ml measuring flask through a funnel.
- 2) The solid sticking to the funnel should be transferred to the funnel with the help of distilled water into the flask add dilute  $\text{H}_2\text{SO}_4$ , drop by drop to obtain a clear solution.
- 3) The flask should be shaken till the substance dissolves and make the solution up to the mark

### Titration of Ferrous Ammonium Sulphate

- 1) The burette should be washed and filled with potassium permanganate solution. Air bubbles should be removed from the burette tip by releasing some solution via it.
- 2) 10 mL of 0.05 M ferrous ammonium sulphate solution should be taken in a conical flask and half test tube (= 5 mL) full of (1.0M)  $\text{H}_2\text{SO}_4$  should be added to it.

- 3) The above solution should be titrated with potassium permanganate solution until the permanent pink colour appears. The content of the flask should be agitated during the titration.
- 4) The titration should be repeated until three concordant readings are obtained.
- 5) The readings should be recorded as shown in observation Table 2 and the strength of potassium permanganate solution should be calculated in mols/litre

## Observation Table

S. No.	Volume of Ferrous Ammonium	Burette Reading		Volume of KMnO <sub>4</sub> Rundown
		Initial	Final	
1	20ml	0	10	10
2	20ml	10	15	15
3	20	15	18	18

## Calculation

Percentage purity can be determined by the following formula.

$$\text{Percentage} = \frac{\text{Titre value} \times \text{Equivalent wt factor} \times \text{Normality of titrant (actual)}}{\text{Weigh of sample} \times \text{Normality of titrant (expected)}} \times 100$$

OR

$$\text{Percentage (\%) Purity} = \frac{\text{Calculated mass of given sample}}{\text{Given mass of sample}} \times 100$$

OR

$$\% \text{ purity} = \frac{\text{Mass}_{\text{calculated}}}{\text{Mass}_{\text{Given}}} \times 100$$

$$\text{Average} = \frac{10+15+18}{3} = 14.3$$

$$M_1 = \frac{0.05 \times 10}{14.3} = \frac{0.5}{14.3}$$

$$M_2 = 0.034$$

Standardisation of 0.05M Ferrous Ammonium Sulphate

$$M_1 V_1 = M_2 V_2$$

$$M_2 = M_1 V_1 / V_2$$

Where,

$M_1$  = Molarity of Ferrous Ammonium Sulphate

$V_1$  = Volume of Ferrous Ammonium Sulphate

$M_2$  = Molarity of  $KMnO_4$  Solution

$V_2$  = Volume of  $KMnO_4$  Solution

$$M_1 = 0.05$$

$$V_1 = 10$$

$$M_2 = ?$$

$$V_2 = 14.3$$

$$\text{Purity of solution} = \frac{0.02063 \times V_2 \times M_2}{M_1 \times w} \times 100$$

0.02063 Equivalent factor

$$0.05 = M_1$$

$$14.3 = V_2$$

$$0.49 = w$$

$$= \frac{(0.02063 \times 14.3 \times 0.034)}{(0.05 * 0.49)}$$

$$\% \text{ Purity} = 1.003/0.02 = 33.4\%$$

**Result:** The percentage purity of the given sample of  $FeSO_4$  is 33.4%.

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