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Diploma in Pharmacy 1st 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 ($FeSO_4$), sulphuric acid (H_2SO_4) and potassium permanganate ($KMnO_4$).

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 H_2SO_4, ferrous sulphate is oxidised to ferric sulphate. A drop of KMnO, 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 ($KMnO_4$) serves as a self-indicator.

Chemical Equations

Reduction Half Reaction: $2\ KMnO_4 + 3\ H2SO_4 \rightarrow K_2SO_4 + 2\ MnSO_4 + 3H_2O + 5\ [O]$

Oxidation Half Reaction: $2\ FeSO_4\ (NH_4)_2SO_4.\ 6H_2O + H_2SO_4 + [O] \rightarrow Fe_2\ (SO_4)_3 + 2\ (NH_4)_2SO_4 + 13\ H_2O \times 5$

$= 2\ KMnO_4 + 8\ H2SO_4 + 10\ FeSO_4\ (NH_4)_2SO_4.\ 6H_2O \rightarrow K_2SO_4 + 2\ MnSO_4 + 5Fe_2\ (SO_4)_3 + 10\ (NH_4)_2SO_4 + 68\ H_2O$

Ionic Equation

Reduction Half Reaction: $MnO_4^- + 5e^- + 8H^+ \rightarrow Mn^{2+} + 4H_2O$

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

$= MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$

Procedure:

Preparation of 0.05 M, Standard Solution of Ferrous Ammonium Sulphate (Molar mass of FeSO4 (NH4): SO4. 6H2O = 392 g mol-l).

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 H,SO, 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) H2SO4 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 $KMnO_4$ 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.

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

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

$M_1 V_1 = M_2 V_2$

$M_2 = M_1V_1 / V_2$

Where,

M_1 = Molarity of Ferrous Ammonium Sulphate

V_1 = Volume of Ferrous Ammonium Sulphate

M_2= Molarity of KMnO, Solution

V_2 = VVolume of $KMnO_4$ Solution

M_1 = 0.05

V_1= 10

M_1=?

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

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

% Purity = 1.003/0.02 = 33.4%

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