EXPERIMENT

Aim

Prepare M/20 solution of Ferrous ammonium sulphate (Mohr's salt). Using this solution find out the molarity and strength of the given $KMnO_4$ solution.

Theory

The reaction between $KMnO_4$ and Mohr's salt is a redox reaction and the titration is therefore called redox titration. $KMnO_4$ acts as oxidising agent and Mohr's salt as reducing agent. $KMnO_4$ can act as oxidising agent in acidic, neutral and alkaline medium. However, the medium used for volumetric titration is acidic medium. The reaction in acidic medium is $MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2O$.

Dil. H_2SO_4 – 4 N H_2SO_4 is used to provide acidic medium. Conc. HNO_3 can't be used as it itself is an oxidising agent and would oxidise Mohr's salt or Oxalic acid used as reducing agent in the titration. (Hence, the reaction will not be quantitatively using $KMnO_4$ as required by the aim of titration). HCl can also not be used to provide acidic medium as it will get oxidised by $KMnO_4$ to evolve Cl_2.

$$2MnO_4^- + 16H^+ + 10Cl \longrightarrow 5Cl_2 + 2Mn^{2+} + 8H_2O$$

Alkaline medium, also cannot be used for the titration as MnO_2 (brown solid) formed interfere with the observation of end point.

$$MnO_4^- + 2H_2O + 3e^- \rightarrow MnO_2 + 4OH^-$$

The titration between Mohr's salt and $KMnO_4$ is fast enough at room temperature.

No separate external indicator is required for the titration as $KMnO_4$ acts as self indicator. An extra drop of $KMnO_4$ after the equivalence point imparts pink colour to the solution. The colour is due to MnO_4^- ions present.

(Mn^{2+} ions are also light pink but appear colourless in dilute solutions).

As ($KMnO_4$) reacts with Mohr's salt Mn^{2+} ions are produced. Then ions produced in the first stage itself act as catalyst for the reaction. Hence in the beginning of titration $KMnO_4$ takes time to be consumed and fade but later the colour of $KMnO_4$ fades away faster.

Material Required

Mohr's salt crystals, standard measuring flask, weighing bottle, funnel, burette, pipette, test tube, conical flask, weight boxes, beakers, and $KMnO_4$ solution.

Ionic Reactions

Molecular equation is:

$$2KMnO_4 + 8H_2SO_4 + 10FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O \rightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 68H_2O + 10(NH_4)_2SO_4$$

$$\begin{aligned} MnO_4 + 8H^+ + 5e^- &\rightarrow Mn^{2+} + 4H_2O \\ [Fe^{2+} &\rightarrow Fe^{3+} + e^-] \times 5 \\ MnO_4 + 8H^+ + 5Fe^{2+} &\rightarrow 5Fe^{3+} + 4H_2O + Mn^{2+} \end{aligned}$$

Procedure

A. Preparation of standard solution as discussed in previous experiment.

B. Titration of $KMnO_4$ solution against $\frac{M}{20}$ Mohr's salt solution.

10 mL Mohr's salt solution to be taken in conical flask, 10 mL of dil H_2SO_4 is also added to it.
$KMnO_4$ solution is taken in the burette.
Perform the titration as discussed under 'Process of titration'.
Record your observations as under.

Observations

A. Preparation of standard solution of Mohr's salt

Calculation of weight to be weighed to prepare 100 mL, $\frac{M}{20}$ solution of Mohr's salt.

Formula used $M = \frac{\text{Weight}}{\text{Molecular Weight}} \times \frac{1000}{V(\text{ in mL})} \Rightarrow \frac{1}{20} = \frac{\text{Weight}}{392} \times \frac{1000}{100} \Rightarrow \text{weight} = 1.96\text{ g}$

Weight of empty weighing bottle = y gm (say) 11.820 g

Weight of bottle + Mohr's salt = (x + y)g(11.820 + 1.96)g = 13.78 g

B. Titration between $KMnO_4$ solution and Mohr's salt solution

Solution to be taken in the pipette = Mohr's salt, Volume of the pipette = 10 mL

Dil. H_2SO_4 to be added is 10 mL

Indicator - $KMnO_4$ self indicator

Colour change - From colourless to light pink

S. No.	Initial reading of the burette	Final reading of the burette	Volume of the $KMnO_4$, solution used(in mL)
1.	1.0	11.5	10.5
2.	11.5	21.8	10.3
3.	21.8	32.1	10.3
4.	32.1	42.4	10.3

Concordant volume =10.3 ml(say)

Calculation

$$\frac{n_1M_1V_1}{KMnO_4} = \frac{n_2M_2V_2}{\text{Mohr's salt}}$$

$n = 5 \quad n = 1$

$$M_{KMnO_4} = \frac{n_{\text{Mohr's}} \times M_{\text{Mohr's salt}} \times V_{\text{Mohr's salt}}}{n_{KMnO_4} \times V_{KMnO_4}} \frac{1 \times \frac{1}{20} \times 10}{5 \times 10.3} = 0.0097M$$

Strength = (Molarity × Molecular weight)g/L.

Calculated strength of $KMnO_4 = 0.0097 \times 158 = 1.53$ g/L.

Result

The molarity and strength of $KMnO_4$ is found to be 0.0097M and 1.53 g/L.

Note: The titration between oxalic acid and $KMnO_4$ is to be done in a similar way with the following change in the reaction and procedure involved. Also, note that the ' n ' factor of oxalic acid is 2.

Reaction would be

$$[MnO_4 + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2O] \times 2$$
$$[C_2O_4^{2-} \longrightarrow 2CO_2 + 2e^-] \times 5$$
$$2MnO_4^- + 16H^+ + 5C_2O_4^{2-} \longrightarrow 2Mn^{2+} + 8H_2O + 10CO_2$$

Molecular reaction is $2KMnO_4 + 5C_2H_2O_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O$

Change in the procedure

Since, the reaction is very slow at room temperature; the titration is done at $60 - 70^{\circ}C$.

For this the contents of conical flask, i.e. (oxalic acid + dil. H_2SO_4) to be heated over the Bunsen flame by placing conical flask on the wire gauze kept on a tripod stand.

To check the temperature is $60 - 70^{\circ}C$ touch the conical flask on the back of your hand. The moment the touch becomes unbearable, start the titration.

Do not overheat the conical flask as oxalic acid on overheating will get decompose to CO_2 before undergoing reaction with $KMnO_4$ and hence, accurate volumetric analysis would not be possible.

$$\begin{matrix} COOH \\ | \\ COOH \end{matrix} \xrightarrow[(\text{high temp.})]{\Delta} CO_2 + CO + H_2O$$

VIVA VOCE

Q 1. What is the molecular formula of ferrous ammonium sulfate (Mohr's salt)?

Ans. The molecular formula of ferrous ammonium sulfate is $(NH_4)2Fe(SO_4)2{\cdot}6H_2O$.

Q 2. How would you determine the molarity of the $KMnO_4$ solution using Mohr's salt?

Ans. Titrate the M/20 solution of Mohr's salt against the KMnO4 solution using a burette until a color change occurs (from pink to colorless). The volume and molarity of the $KMnO_4$ solution can be calculated using the volume and molarity of Mohr's salt solution and the stoichiometry of the reaction.

Q 3. What is the stoichiometry of the reaction between Mohr's salt and $KMnO_4$?

Ans. The stoichiometry of the reaction is 1:5 between ferrous ammonium sulfate and potassium spermanganate, meaning that one mole of Mohr's salt reacts with five moles of KM

Q 4. How can you ensure the accuracy of the titration experiment?

Ans. Ensure that the burette readings are taken accurately, and the titration is performed slowly near the endpoint to avoid overshooting. Perform multiple titrations to ensure reproducibility of results.

Q 5. Why is it important to standardize the Mohr's salt solution before titration?

Ans. Standardization ensures that the concentration of the Mohr's salt solution is accurately known, which is crucial for determining the concentration of the $KMnO_4$ solution.

Q 6. Can you explain the principle of endpoint detection in the titration experiment?

Ans. The endpoint of the titration is reached when the color of the solution changes due to the complete reaction between the titrant and the analyte. In this case, the pink color of KMnO4 disappears when all the Fe^{2+} ions in Mohr's salt are oxidized to Fe^{3+} ions.

Q 7. What precautions should be taken during the titration experiment?

Ans. Precautions include ensuring that all glassware is clean and dry, using a white tile to aid in detecting the color change, and performing the titration under controlled lighting conditions to accurately observe the endpoint. Additionally, avoid contamination of reagents and solutions.