Estimation of Iron in Mohr's Salt Solution

1. Aim

To estimate the amount of iron present in the given whole of the Mohr's salt solution by titrating it against a standard potassium permanganate ($KMnO_4$) solution.

2. Principle & Theory

The estimation is based on a redox (oxidation-reduction) titration in an acidic medium. Ferrous ammonium sulfate, commonly known as **Mohr's salt** [$FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$], acts as a reducing agent that supplies ferrous ions (Fe^{2+}). Potassium permanganate ($KMnO_4$) serves as a strong oxidizing agent.

In the presence of dilute sulfuric acid (H_2SO_4), $KMnO_4$ oxidizes the ferrous ions (Fe^{2+}) to ferric ions (Fe^{3+}), while the permanganate ions (MnO_4^-) are reduced to manganese ions (Mn^{2+}).

Chemical Equations:

Ionic Reduction Half-Reaction:

> $$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$$

Ionic Oxidation Half-Reaction:

> $$5Fe^{2+} \rightarrow 5Fe^{3+} + 5e^-$$

Overall Molecular Equation:

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

> ***Self-Indicator Property:*** *$KMnO_4$ acts as its own indicator. The addition of a single excess drop of $KMnO_4$ after all Fe^{2+} ions are oxidized imparts a persistent light pink color to the solution, signaling the completion of the reaction.*

3. Apparatus & Reagents Required

- Burette (50 mL) and Burette Stand
- Pipette (10 mL or 20 mL)
- Conical Flask (250 mL)
- Standard Potassium Permanganate ($KMnO_4$) solution
- Given Mohr's Salt solution
- Dilute Sulfuric Acid (approx. 2M H_2SO_4)
- Volumetric Flask, Funnel, and Glass Rod

4. Procedure

1. **Burette Preparation:** Rinse the burette thoroughly with distilled water, then with a small amount of the standard $KMnO_4$ solution. Fill the burette with $KMnO_4$, remove any air bubbles from the nozzle, and adjust the level to the zero mark. Read the upper meniscus due to the dark color of the solution.
2. **Sampling:** Pipette out exactly 10 mL (or 20 mL) of the given Mohr's salt solution into a clean conical flask.
3. **Acidification:** Add approximately one full test tube (about 20 mL) of dilute sulfuric acid (H_2SO_4) to the conical flask. This prevents the precipitation or hydrolysis of iron ions and provides the necessary acidic medium.
4. **Titration:** Run the $KMnO_4$ solution from the burette dropwise into the conical flask while continuously swirling the flask. The purple color of $KMnO_4$ discharges instantly upon mixing.
5. **Endpoint:** Continue adding $KMnO_4$ dropwise until a single drop produces a permanent faint pink color that persists for at least 30 seconds.
6. **Replicates:** Note the final burette reading. Repeat the titration until at least two consecutive concordant readings are obtained.

5. Observation Table

Sl. No.	Volume of Mohr's Salt Solution (mL)	Burette Readings (mL)		Volume of $KMnO_4$ Used (V_1 mL)
		Initial	Final	
1	20.0	0.0		
2	20.0			
3	20.0			

Concordant Volume of $KMnO_4$ (V_1) = _____ mL

6. Calculations

Using the Volumetric Law of Equivalence:

$$N_1 \times V_1\ (KMnO_4) = N_2 \times V_2\ (\text{Mohr's Salt})$$

Where:

- **N_1:** Normality of standard $KMnO_4$ solution
- **V_1:** Concordant volume of $KMnO_4$ consumed (from table)
- **N_2:** Normality of the given Mohr's Salt solution
- **V_2:** Volume of Mohr's salt solution pipetted out (e.g., 20 mL)

Calculate Normality of Mohr's Salt solution (N_2):

$$N_2 = (N_1 \times V_1) / V_2$$

Estimation of Iron Content:

Since the equivalent weight of Iron (Fe) in this redox reaction is **55.85 g/eq**:

$$\text{Strength of Iron (g/L)} = N_2 \times 55.85$$

> **Total Amount of Iron in Whole Solution = [Strength (g/L) × Total Volume of Solution (mL)] / 1000**

7. Precautions

- Always read the **upper meniscus** for dark-colored solutions like $KMnO_4$.
- Never use hydrochloric acid (HCl) or nitric acid (HNO_3) for acidification. HCl is oxidized by $KMnO_4$ to chlorine, and HNO_3 is itself a strong oxidizing agent.
- Add the titrant dropwise near the endpoint to avoid over-titration.
- The glassware must be thoroughly rinsed with distilled water, and the burette/pipette must be pre-rinsed with their respective solutions.