

Aim: Preparation and Standardization of Ceric Ammonium Sulfate

References:

1. Vogel, A.I. (1989). "Vogel's Textbook of Quantitative Chemical Analysis." 5th Edition, Longman Scientific & Technical.
2. Harris, D.C. (2010). "Quantitative Chemical Analysis." 8th Edition, W.H. Freeman.

Objective:

To prepare a standard solution of ceric ammonium sulfate ($\text{Ce}(\text{NH}_4)_4(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$) and standardize it using a primary standard solution of sodium oxalate.

Theory:

Ceric ammonium sulfate is a strong oxidizing agent used in redox titrations, particularly in acidic media. It is not a primary standard due to its instability in solution. Hence, it needs to be standardized before use. Sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) is a reliable primary standard for this purpose due to its high purity and stability.

Materials Required:

1. Ceric ammonium sulfate ($\text{Ce}(\text{NH}_4)_4(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$)
2. Sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) – primary standard
3. Concentrated sulfuric acid (H_2SO_4)
4. Distilled water
5. Beakers, burette, pipette, conical flask, measuring cylinder, funnel

Preparation of Ceric Ammonium Sulfate Solution:

1. Weighing the Ceric Ammonium Sulfate:

- Accurately weigh about 30.4 g of ceric ammonium sulfate ($\text{Ce}(\text{NH}_4)_4(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$).

2. Dissolution:

- Dissolve the weighed ceric ammonium sulfate in about 500 mL of distilled water in a beaker.

3. Acidification:

- Add 10 mL of concentrated sulfuric acid (H₂SO₄) to the solution to maintain an acidic medium, which prevents hydrolysis of ceric ammonium sulfate.

4. Transfer and Dilution:

- Transfer the solution to a 1000 mL volumetric flask and make up the volume to 1000 mL with distilled water.

5. Storage:

- Store the prepared ceric ammonium sulfate solution in a tightly sealed container to prevent oxidation by atmospheric oxygen.

Standardization of Ceric Ammonium Sulfate Solution:

1. Preparation of Sodium Oxalate Solution:

- Accurately weigh 0.0675 g of sodium oxalate (Na₂C₂O₄) and dissolve it in distilled water in a 250 mL volumetric flask. Make up the volume to 250 mL to get a 0.01 N solution.

2. Reaction Setup:

- Pipette 25.0 mL of the sodium oxalate solution into a 250 mL conical flask.

- Add about 25 mL of distilled water and 10 mL of concentrated H₂SO₄ to acidify the solution.

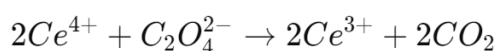
3. Titration:

- Heat the solution to about 60-70°C.

- Titrate with the ceric ammonium sulfate solution until a faint yellow color persists for 30 seconds, indicating the endpoint.

4. Calculation:

- The reaction between ceric ammonium sulfate and sodium oxalate in an acidic medium can be represented as:



- Calculate the normality (N) of the ceric ammonium sulfate solution using the formula:

$$N_1V_1 = N_2V_2$$

Where:

- N_1 = Normality of the ceric ammonium sulfate solution
- V_1 = Volume of the ceric ammonium sulfate solution used
- N_2 = Normality of the sodium oxalate solution (0.01 N)
- V_2 = Volume of the sodium oxalate solution used (25 mL)

Example Calculation:

- Suppose the volume of ceric ammonium sulfate used (V_1) is 15.0 mL:

$$N_1 = \frac{N_2 \times V_2}{V_1} = \frac{0.01 \times 25}{15.0} = 0.0167N$$

Precautions:

1. Ensure the solution is heated to 60-70°C for accurate results.
2. Use freshly prepared sodium oxalate solution.
3. Perform the titration quickly to prevent decomposition of oxalic acid.

Result: The normality of the prepared ceric ammonium sulfate solution was found to be 0.0167 N.