PREPARATION AND STANDARISATION OF 0.1N CERIC AMMONIUM SULPHATE

Aim

to prepare and standardise 0.1N ceric ammonium sulphate

Chemical requirements

Ceric ammonium sulphate, sulphuric acid, arsenic trioxide, sodium hydroxide, dil. sulphuric acid, osmic acid, ferric sulphate (ox). N-phenyl anthranilic acid.

Apparatus

Burette, conical flask, measuring jar, volumetric flask, glass jar etc.

Principle

Arsenic trioxide is converted into arsensious acid. This is oxidised by ceric ions into Arsenic acid. In the process ceric ions are reduced to cerrous ions. The oxidation reaction taking place is given by

\[ \text{Ce}^{4+} + e^- \rightarrow \text{Ce}^{3+} \]

\[ 2\text{Ce}^{4+} + \text{H}_3\text{AsO}_3 + \text{H}_2\text{O} \rightarrow 2\text{Ce}^{3+} + \text{H}_3\text{AsO}_4 + 2\text{H}^+ \]

Procedure

PREPARATION OF 0.1N CERIC AMMONIUM SULPHATE

66 gms of ceric ammonium sulphate was dissolved with gentle heat in a mixture of 30 ml of sulphuric acid and 200 ml of water. It was then cooled and filtered.
The obtained solution was diluted to 1000 ml with water.

**STANDARDISATION OF 0.1N CERIC AMMONIUM SULPHATE**

About 0.2 gm of Arsenic trioxide which was previously dried for one hour was accurately weighed and was transferred to a 500 ml conical flask. The inner walls of flask were washed with 100 ml of water and mixed. Then 300 ml of dilute sulphuric acid, 0.15 ml of osmic acid, 0.1 ml of ferroin sulphate indicator were added. It was then titrated slowly with ceric ammonium sulphate until pink colour changed to pale blue or yellowish green or purple colour.

4.946 gm of Arsenic trioxide = 1 ml of 0.1N Cerium ammonium sulphate (08) 0.6326 gm of Ceric ammonium sulphate

**CAUTION:** Osmic acid solution is corrosive to eyes, skin and mucous membranes.

**PREPARATION OF FERROIN SULPHATE INDICATOR**

Ferroin sulphate or tvs (1,10-phenanthroline) ferrous sulphate complex is prepared by dissolving 0.7 gm of ferrous sulphate and 15 gm of 1,10-phenanthroline hydrochloride in 70 ml of water and adding water to produce 100 ml.

**PREPARATION OF OSMIC ACID SOLUTION**

Osmium tetroxide - OsO₄ mol. wt - 254.20

A 1% (w/v) sol'n of osmic acid in water
Report

Ceric ammonium sulphate was prepared and after standardisation the normality was found to be 0.0887 N.

7/3/08
Chemical formula

Ceric ammonium sulphate  \[ \text{Mol. wt} = 632.53 \]
\[ \text{Ce}_2(\text{SO}_4)_3 \cdot 2(\text{NH}_4)_2\text{SO}_4 \cdot 2\text{H}_2\text{O} \]

10) What is the purpose of adding Osmic acid?
The addition of Osmic acid is to bring about a sharp end point. In the absence, the end point cannot be observed sharply.

20) What is the reaction between ferroin sulphate & ceric ions?
Ferroin sulphate (Tris (1,10-phenanthroline) - Iron II sulphate. Ferroin sulphate is bright red complex formed by combination of the base, 1,10-phenanthroline with iron(II) sulphate.

\[
\text{[ } \text{Fe}^{2+} + \text{Fe}^{3+} \text{]} \rightarrow [\text{(C}_{12}\text{H}_8\text{N}_2)_3\text{Fe}]^{+2} \]

This complex is readily oxidised reversibly to the corresponding ortho-phenanthroline (III) complex which is pale blue in cobalt.

\[
[\text{(C}_{12}\text{H}_8\text{N}_2)_3\text{Fe}]^{+2} \rightleftharpoons [\text{(C}_{12}\text{H}_8\text{N}_2)_3\text{Fe}]^{+3} + e^- \quad \text{pale blue} \]
CALCULATIONS

→ weight of Arsenic Trioxide

weight of As₂O₃ & paper = 390 mg
weight of paper after transferring = 190 mg
Amount of As₂O₃ transferred = 200 mg.

→ Burette reading

s.No | Initial | Final | Vol. of Ceric ammonium sulphate consumed
--- | --- | --- | ---
1 | 0 | 46.1 | 46.1 ml

→ wt. of A

0.004946 g of As₂O₃ = 1 ml of 0.1 N Ceric ammonium sulphate

0.200 g of As₂O₃ = ?

\[
\frac{0.200 \times 1}{0.004946} = 40.436 \text{ ml of 0.1 N Ceric sulphate}
\]

\[N_1 V_1 = N_2 V_2\]

\[N_2 = \frac{N_1 V_1}{V_2} = \frac{0.1 \times 40.436}{46.1} = 0.0887 N\]