

Exp: 3

9/10/07

PREPARATION AND STANDARDISATION OF

0.1N CERIC AMMONIUM SULPHATE

Aim

To prepare and standardise 0.1N ceric ammonium sulphate

Chemical requirements

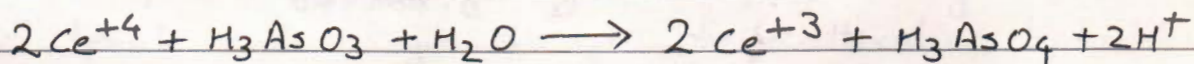
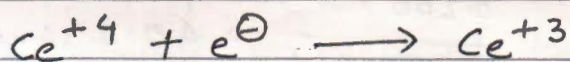
Ceric ammonium sulphate, Sulphuric acid, arsenic trioxide, sodium hydroxide, dil. sulphuric acid, osmic acid, ferrous sulphate (or) N-phenyl anthranilic acid.

Apparatus

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

Principle

Arsenic trioxide is converted into arsenious 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



Procedure

PREPARATION OF 0.1N CERIC AMMONIUM SULPHATE

66 gm of ceric ammonium sulphate was dissolved with gentle heat in a mixture of 30 ml of sulphuric acid and 500 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 \equiv 1 ml of 0.1N Ceric ammonium sulphate (os) 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 tris (1,10-phenanthroline) ferrous sulphate complex is prepared by dissolving 0.7 gm of ferrous sulphate and 1.5 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 - OsO_4 mol. wt - 254.20
A 1% (w/v) sol'n of osmic acid in water

Report

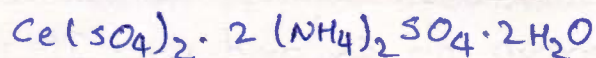
Cerriic ammonium sulphate was prepared and after standardisation the normality was found to be 0.0887 N

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chemical formula

Ceric ammonium sulphate

Mol. wt \Rightarrow 632.53

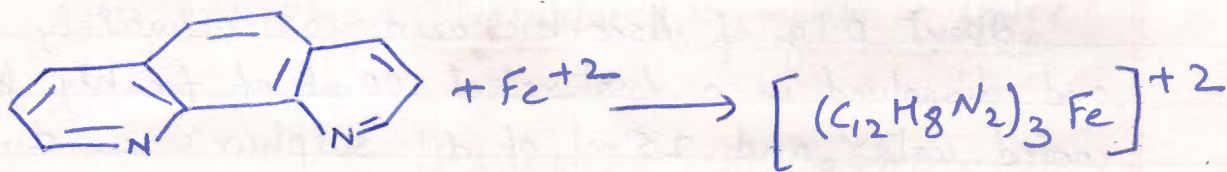


1a) what is purpose of adding Osmic acid?

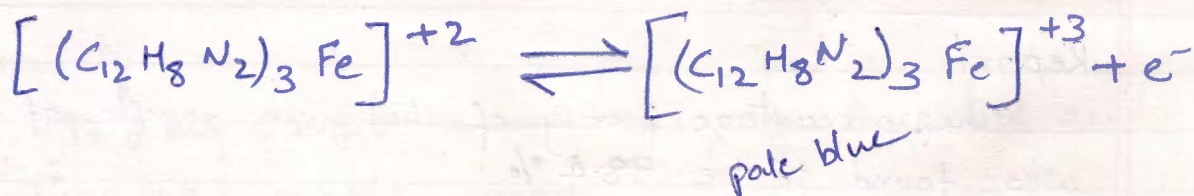
The addition of Osmic acid is to bring about a sharp end point. In the absence the end point cannot be observed sharply.

2a) 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



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



CALCULATIONS

→ weight of Arsenic trioxide

Weight of As_2O_3 & paper — 390 mg

Weight of paper after transferring — 190 mg

Amount of As_2O_3 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 As_2O_3 \cong 1ml of 0.1N Ceric ammonium sulphate

0.200 g of As_2O_3 \cong ?

$$\frac{0.200 \times 1}{0.004946} = 40.436 \text{ ml of } 0.1N \text{ Ce-NH}_4\text{S}$$

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2} = \frac{0.1 \times 40.436}{46.1}$$

$$= 0.0887 N$$