EXPERIMENT X6

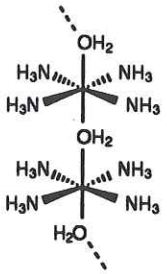
PREPARATION AND ANALYSIS OF TETRAAMMINEAQUACOPPER(II) SULFATE

Materials

Copper(II) sulfate pentahydrate
4M Ammonium hydroxide solution
Ethanol
Ether
0.1M Hydrochloric acid
0.1M Sodium thiosulfate
Potassium iodide

Introduction

Tetraammineaquacopper(II) sulfate $[Cu(NH_3)_4(H_2O)]SO_4$ is a transition metal coordination compound which contains a complex cation. Although the formula seems to show the complex to be five coordinate the crystal structure shows that the cation is more complicated. It is based upon square planar $Cu(NH_3)_4$ units which are stacked vertically and linked to one another through the oxygen atoms of water molecules placed above and below the square planes. This gives the Cu(II) ion a distorted octahedral geometry. The water hydrogens are hydrogen bonded to the sulfate ions. In this experiment you will prepare the complex and quantitatively determine ammonia and copper titrimetrically.



Preparation

Dissolve copper sulfate pentahydrate (6 g) in the **minimum** amount of water. Add 4M ammonium hydroxide until the copper hydroxide which is precipitated redissolves (the solution should be a deep blue – ***Note:*** *It is difficult to observe when all the precipitate has redissolved*). Add ethanol **slowly** to the solution until the complex precipitates. Filter the precipitate using a Buchner funnel, wash first with ethanol and then ether and dry the solid by pulling air through it for 10 min. Weigh your product and calculate the % yield.

Determination of Ammonia and Copper

Weigh accurately *ca.* 0.5 g of the complex and dissolve it in 25 ml of water in a conical flask. Add 10 drops of methyl orange and titrate with the standard 0.5M hydrochloric acid provided. **Note:** *Initial addition of the acid causes precipitation of copper(II) hydroxide with its characteristic gelatinous blue-green appearance. As the acid is added, the blue colour fades and the indicator colour begins to dominate. Just before the end point all the copper(II) hydroxide dissolves. The appearance of the end point may be affected by the blue aquacopper(II) ion which makes the change in colour appear to be green to purple rather than yellow to pink.*

Now add potassium iodide (1 g) to the solution in the conical flask and perform an iodometric titration to determine the amount of copper (see Experiment X1, for details).

Using the titre value of 0.5M HCl and the titre value of 0.1M $Na_2S_2O_3$ calculate the percentages of NH_3 and Cu in the complex. **Repeat the analysis until consistent results are obtained.**

Allow all I^- to dissolve. Titrate with $Na_2S_2O_3$ until a pale yellow colour is obtained. Add KSCN (≈1.0 g) and starch indicator and titrate until the soln is colourless