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| **Foundation Year** | **Pt 1: The Equilibrium Between [Co(H2O)6]2+ and [CoCl4]2-**  **Pt 2: Preparation of**  **Tetraamminecopper(II) sulfate – 1 – water** | **Semester 2** |

**Part 1: The Equilibrium Between [Co(H2O)6]2+ and [CoCl4]2-**

**Aim**

To illustrate the dynamic equilibrium between hexaaquacobalt(II) and tetrachlorocobaltate(II) and explain the theory behind any observations.

**Introduction**

The transition metal complexes hexaaquacobalt(II) and tetrachlorocobaltate(II) exist in a dynamic equilibrium which adheres to Le Chatelier’s principle.

**Equation**: [CoCl4]2-(aq) + 6H2O(l) [Co(H2O)6]2+(aq) + 4Cl-(aq)

**Colour:**

**Shape:**

**Coordination No.:**

**Diagram:**

The solution at the start of demonstration contains an equilibrium mixture of the two complexes. The different steps in the demonstration are i) the effect of heating, ii) the effect of cooling, iii) the effect of adding water and iv) the effect of adding conc. hydrochloric acid.

**Skills associated with this demonstration**

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| **Practical Skills** | **Scientific Skills**   * Making and recording observations * Interpreting observations |

You are required to write up your observations of this demonstration for your *Skills Portfolio*. You should therefore ensure that you record these observations as effectively as possible in your lab notebook. You will also be required to explain the theory behind the observations for your skills portfolio, so you may need to make some notes to accompany this in your lab notebook.

**Part 2: Preparation of Tetraamminecopper(II) sulfate – 1 – water**

**Aim**

To prepare crystals of a complex salt, tetraamminecopper(II) sulfate – 1 – water.

**Introduction**

Most transition metal ions in aqueous solution exist as aqua-complexes where water molecules act as coordinating ligands to the transition metal centre. When dissolved in water, copper(II) sulfate forms the hexaaquacopper(II) complex ion [Cu(H2O)6]2+, resulting in a light blue solution.

Ligand exchange (or substitution) occurs when a certain number of water ligand molecules are replaced by different ligands. The reaction you will carry out in this experiment is an example of a ligand exchange reaction.

**Skills associated with this practical**

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| **Practical Skills**   * Correctly handling concentrated ammonia * Carrying out a vacuum filtration | **Scientific Skills**   * Calculating % yield |

**Signposts**

Chemistry, Conoley & Hill, 3rd Edition, Chapter 24, pages 485-492; chapter 27, pages 566-567

**Understanding Hazard and Minimising Risk**

You must stand up throughout the practical, and safety glasses must be worn at ALL times in the lab. You must wear a labcoat whilst you are carrying out ALL practical work. Long hair must be tied back, and trousers (jeans are OK) must be worn. Open-toed shoes and clothing revealing bare skin are not permitted.

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| Substance | Amount | Hazards | Minimising Hazards | Disposal / spillage |
| Concentrated ammonia | 2 cm3 | Corrosive, causes severe burns, eye damage, very toxic to aquatic life. Harmful if inhaled, swallowed, absorbed through skin. | Handle in a fume cupboard, use gloves, lab coat, goggles. | Dilute with water (10 water: 1 ammonia) and pour down the sink with copious amounts of water. |
| Copper(II) sulfate | ~1.5 g | Harmful if swallowed. Causes severe eye and skin irritation. Very toxic to aquatic organisms. | Wear gloves, lab coat, goggles. | Pour down the sink with copious amounts of water. |
| Ethanol | <10 cm3 | Highly flammable. | Wear lab coat, gloves and goggles. | Pour down the sink with copious amounts of water. |

**Procedure**

Apparatus

PER PAIR: Glass rod Buchner funnel and flask

100 cm3 beaker Deionised water

50 cm3 beaker 10 cm3 graduated pipette and filler

Test tube Spatula

Filter papers Sample bottle

Method

1. Prepare a hot water bath by pouring recently boiled water from a kettle into a 100 cm3 beaker.
2. Measure 6 cm3 of ethanol using a graduated pipette and place it in a 50 cm3 beaker.
3. Weigh approximately 1.5 g of copper(II) sulfate then place into a test tube. Record the mass.
4. Add 4 cm3 of water to the copper(II) sulfate in the test tube using a graduated pipette and place the test tube in a hot water bath. Stir gently with a glass rod to dissolve the copper(II) sulfate.
5. Remove the test tube of copper(II) sulfate solution from the water bath once all solids has dissolved.
6. Take the test tube containing the copper solution and the beaker containing ethanol to the fumehood. Whilst wearing gloves, add, with stirring, 2 cm3 of concentrated ammonia solution to the copper(II) sulfate solution. Record your observations.
7. Pour the contents of the test tube into the beaker containing 6 cm3 of ethanol and mix using the glass rod. Note your observations and return to your bench. Cool the mixture in an ice-water bath. Note any further observations.
8. Set up a Buchner funnel, flask and filter paper. Filter off any crystals that have formed in the beaker.
9. Rinse out the test tube with a small volume of cold ethanol and add the washings to the Buchner funnel. Rinse the crystals with a small volume of cold ethanol.
10. Change the Buchner flask for a dry one and then dry the crystals at the pump (ask a demonstrator to explain if you are unsure of the procedure). Transfer the semi-dry crystals onto a large piece of filter paper and allow to air-dry (ask a demonstrator to explain if you are unsure of the procedure). Transfer the crystals to a sample bottle and record the mass of the crystals.

**Calculating % yield**

1. Write an equation for this reaction.
2. Calculate the relative formula masses of CuSO4•5H2O and Cu(NH3)4SO4•H2O.
3. Calculate the amount in moles of copper(II) sulfate (starting material) used. Use the result to calculate the theoretical yield (mass) of tetraamminecopper(II) sulfate – 1 – water (product) your reaction should have produced.
4. Use your recorded masses to calculate the mass of copper(II) sulfate and the actual yield of product.
5. Calculate the % yield you obtained:

**Supplementary tasks – you will be required to discuss these in your *Skills Portfolio*. The tasks do not need to be completed in the lab, but you should think about them before leaving.**

1. Comment on your % yield, and explain any loss/gain in mass compared with the theoretical yield.
2. Carry out some research to identify the structure of the complexes formed during this reaction a) in solution and b) in the solid-state. Draw diagrams to show their shape and give the coordination number.

**Deadlines, Assessment and Feedback on Performance**

You are required to complete the *Skills Portfolio* document associated with this practical. This should be completed electronically with all photos inserted in the appropriate places and accompanying text typed in. The submission deadline for *Skills Portfolio*s will normally be midnight on the Sunday following the practical, although you will be given specific guidance during the practical session. Submission is via the e-submission system Turnitin which you will be able to access in the appropriate folder in the Laboratories and Coursework Blackboard course.