Core practical 12: Prepare a transition metal complex

Objective

- To prepare a transition metal complex

Safety

- Wear a lab coat and use eye protection.
- Tie long hair back.
- Concentrated ammonia solution is corrosive and dangerous to the environment. It must be kept in the fume cupboard and handled wearing gloves.
- Copper salts are harmful and dangerous to the environment.
- Ethanol is flammable and must be kept away from flames.

Specification links

- Practical techniques 1, 4, 11
- CPAC 1a, 2a, 2b, 3a, 4a, 4b

Procedure

1. Weigh between 1.4 g and 1.6 g of copper(II) sulfate. To do this you should weigh a test tube and record its mass. Then add the copper(II) sulfate to the test tube, reweigh and record the mass. The mass of the copper(II) sulfate is the difference between the two masses.
2. Add 4 cm^3 of water to the test tube using a graduated pipette.
3. Prepare a water bath using hot water from a kettle in a 100 cm^3 beaker. Stand the test tube in the water bath. Stir gently to dissolve the copper(II) sulfate.
4. Remove the test tube containing copper(II) sulfate solution from the water bath.
5. *Perform this step in the fume cupboard wearing gloves.* Add, while stirring, 2 cm^3 of concentrated ammonia solution to the copper(II) sulfate solution.
6. Pour the contents of the test tube into 6 cm^3 of ethanol that has been pipetted into a beaker, – mix well and then cool the mixture in an ice bath.
7. Using a Büchner funnel and flask, filter the crystals. Wash your test tube with cold ethanol and add the washings to the Büchner funnel. Finally, rinse the crystals with cold ethanol.
8. Carefully scrape the crystals off the filter paper onto a fresh piece of filter paper. Cover the crystals with a second piece of filter paper, Carefully pat the paper to dry the crystals. Note that to get the crystals completely dry, you may need to repeatedly move the crystals to dry parts of the filter paper.
9. Once dry, measure and record the mass of crystals.

Answers to questions

1. $CuSO_4 \cdot 5H_2O + 4NH_3 \rightarrow Cu(NH_3)_4SO_4 \cdot H_2O + 4H_2O$
2. $CuSO_4 \cdot 5H_2O = 249.5$
   $Cu(NH_3)_4SO_4 \cdot H_2O = 245.5$
3. 0.00601 moles (based on 1.5 g of $CuSO_4 \cdot 5H_2O$)
4. Theoretical yield of $Cu(NH_3)_4SO_4.H_2O$ = 1.48 g
5. Percentage yield = $\frac{1.2}{1.48} \times 100 = 81\%$
6. Losses could be due to reaction not going to completion and some product staying in solution and not crystallising out; gains could be due to crystals not being dry or not being pure.

Sample data

Yield of $Cu(NH_3)_4SO_4 \cdot H_2O$ = 1.2 g

Core practical 12: Prepare a transition metal complex

Objective

- To prepare a transition metal complex

Safety

- Wear a lab coat and use eye protection.
- Tie long hair back.
- Concentrated ammonia solution is corrosive and dangerous to the environment. It must be kept in the fume cupboard and handled wearing gloves.
- Copper salts are harmful and dangerous to the environment.
- Ethanol is flammable and must be kept away from flames.

All the maths you need

- Use ratios fractions and percentages.

Equipment

- copper(II) sulfate
- 10 cm^3 ethanol
- 2 cm^3 concentrated ammonia solution
- crushed ice
- Büchner flask and funnel and vacuum filtration apparatus
- filter paper
- 1 pair of gloves
- 1 test tube
- one 50 cm^3 beaker
- one 100 cm^3 beaker
- one 10 cm^3 graduated pipette
- 1 pipette filler
- 1 spatula
- 1 stirring rod
- mass balance (2 d.p.) and weighing boat
- access to a kettle

Procedure

1. Weigh between 1.4 g and 1.6 g of copper(II) sulfate. To do this you should weigh a test tube and record its mass. Then add the copper(II) sulfate to the test tube, reweigh and record the mass. The mass of the copper(II) sulfate is the difference between the two masses.
2. Add 4 cm^3 of water to the test tube using a graduated pipette.
3. Prepare a water bath using hot water from a kettle in a 100 cm^3 beaker. Stand the test tube in the water bath. Stir gently to dissolve the copper(II) sulfate.
4. Remove the test tube containing copper(II) sulfate solution from the water bath.
5. *Perform this step in the fume cupboard wearing gloves.* Add, while stirring, 2 cm^3 of concentrated ammonia solution to the copper(II) sulfate solution.
6. Pour the contents of the test tube into 6 cm^3 of ethanol that has been pipetted into a beaker – mix well and then cool the mixture in an ice bath.
7. Using a Büchner funnel and flask, filter the crystals. Wash your test tube with cold ethanol and add the washings to the Büchner funnel. Finally, rinse the crystals with cold ethanol.
8. Carefully scrape the crystals off the filter paper onto a fresh piece of filter paper. Cover the crystals with a second piece of filter paper, Carefully pat the paper to dry the crystals. Note that to get the crystals completely dry, you may need to repeatedly move the crystals to dry parts of the filter paper.
9. Once dry, measure and record the mass of the crystals.

Analysis of results

1. Record the mass of copper(II) sulfate used in the reaction.
2. Record the yield of dry tetraamminecopper(II) sulfate-1-water obtained.

Questions

1. Write the equation for this reaction.
2. Calculate the relative formula masses of $CuSO_4 \cdot 5H_2O$ and $Cu(NH_3)_4SO_4 \cdot H_2O$.
3. Calculate the number of moles of copper(II) sulfate used in the reaction.
4. Use your answer to calculate the theoretical yield of tetraamminecopper(II) sulfate-1-water your reaction should have produced.
5. Calculate the percentage yield obtained in this reaction.
6. Comment on your percentage yield. Explain any loss or gain in mass compared with the theoretical yield.

Core practical 12: Prepare a transition metal complex

Objective
● To prepare a transition metal complex

Safety
● Wear a lab coat and use eye protection. ● Consult CLEAPSS Hazcards® 6, 27C, and 40A. Perform a risk assessment using up-to-date information before this practical is carried out.

Equipment per student/group	Notes on equipment
copper(II) sulfate	Harmful and dangerous to the environment.
10 cm^3 ethanol	Flammable – keep away from flames.
2 cm^3 concentrated ammonia solution	Corrosive and dangerous to the environment. Keep in the fume cupboard and handle wearing gloves.
crushed ice	
Büchner flask and funnel and vacuum filtration apparatus	
filter paper	
1 pair of gloves	
1 test tube	
one 50 cm^3 beaker	
one 100 cm^3 beaker	
one 10 cm^3 graduated pipette	
1 pipette filler	
1 spatula	
1 stirring rod	
mass balance (2 d.p.) and weighing boat	
access to a kettle	

Notes