Teacher Resource Bank

GCE Chemistry

PSA12: A2 Inorganic Chemistry

• Prepare an inorganic complex
Technical Sheet

Prepare an inorganic complex
The preparation of hexaamminecobalt(III) chloride

Whenever possible, students should work individually.
If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

This PSA is likely to require two practical sessions.

Requirements

- Cobalt (II) chloride-6-water crystals
- ammonium chloride solid
- decolourising charcoal
- concentrated (880) ammonia solution
- 20-volume hydrogen peroxide
- concentrated hydrochloric acid
- deionised or distilled water in a wash bottle
- ice

- 250 cm$^3$ beaker
- 400 cm$^3$ beaker
- Two 25 cm$^3$ measuring cylinders
- Two 10 cm$^3$ measuring cylinders
- Two 100 cm$^3$ conical flasks
- 250 cm$^3$ conical flask (with a stopper)
- Thermometer (-10 °C to 110 °C)
- Sample bottle or dry test tube
- Filter funnel and filter paper
- Bunsen burner, tripod and gauze
- Spatula

Centres are expected to carry out and be responsible for their own safety risk assessments.
Prepare an inorganic complex
The preparation of hexaamminecobalt(III) chloride

Introduction
The compound hexaamminecobalt(III) chloride can be prepared from cobalt(II) chloride by a ligand substitution reaction in which the water ligands of the hydrated cobalt(II) complex ion are replaced by ammonia. The hexaamminecobalt(II) ion is then oxidised using hydrogen peroxide as the oxidising agent.

It is the responsibility of the student to carry out and be responsible for their own safety risk assessment before carrying out this experiment. Wear safety glasses at all times. Assume that all of the reagents and liquids are toxic, corrosive and flammable.

Experiment
Part 1 - Preparation of the crude solid.
   a) Add approximately 8 g of ammonium chloride and 12 g of hydrated cobalt(II) chloride to a 100 cm$^3$ conical flask containing 20 cm$^3$ of deionised or distilled water.
   b) Add two spatula measures of decolourising charcoal and heat the contents of the conical flask gently, bringing the solution just to boiling.
   c) Cool the flask under water from the cold water tap.
   d) Working in a fume cupboard, add 25 cm$^3$ of concentrated ammonia solution to the flask and then cool it to below 10 $^\circ$C using a beaker of ice and water.
   e) Add 5 cm$^3$ of 20-volume hydrogen peroxide to the cooled flask. Shake the contents of the flask thoroughly for a few minutes. Repeat this a further four times so that a total volume of 25 cm$^3$ of hydrogen peroxide has been added.
   f) When all of the hydrogen peroxide has been added, transfer the contents of the flask to a 250 cm$^3$ conical flask.
   g) Prepare a water bath using the 400 cm$^3$ beaker provided and heat the water in the bath to approximately 60 $^\circ$C. Place the flask into the water bath and maintain this temperature for approximately 30 minutes.
   h) If the whole PSA is to be carried out in one practical session, it is necessary at this stage to cool the flask in ice and water to precipitate the crude solid product mixed with charcoal. Alternatively, the flask can be left to cool until the next practical session.
Part 2 – Purification of the product

a) Filter off the crude solid and charcoal and then place the filter paper containing the solid into a beaker containing 100 cm³ of boiling water which has been acidified with 3 cm³ of concentrated hydrochloric acid.
b) Filter the hot solution to remove the charcoal and collect the filtrate in a 100 cm³ conical flask. This filtrate should contain the dissolved cobalt salt.
c) Add 10 cm³ of concentrated hydrochloric acid to the filtrate and then cool the flask in ice and water. At this stage, crystals of the hexaamminecobalt(III) chloride should form.
d) Filter off the crystals, rinsing them with a small volume of deionised or distilled water. Dry the crystals by opening out the filter paper. Use a spatula to transfer the solid to another piece of filter paper and remove excess water by gently pressing the crystals with more filter paper. Leave the crystals to dry naturally for about five minutes.
e) Weigh a sample bottle or an empty dry test-tube. Add your dried crystals and reweigh to obtain the mass of hexaamminecobalt(III) chloride crystals formed.
f) The quality of your product may be judged by its colour, its crystallinity and its dryness.
Teacher Notes and Marking Guidance

The specific marking guidance in the specification is as follows

2 marks: All areas of the task are carried out competently.
The quantities of reagents are appropriate for the preparation.
The apparatus set-up for the preparation is safe and appropriate.
The experiment is carried out safely and produces an appropriate quantity and quality of product.

1 mark: One of the areas of the task is performed poorly.
The quantities of reagents are inappropriate for the preparation OR
The apparatus set-up for each experiment is unsafe or inappropriate OR
The experiments are carried out with insufficient care or the yield is poor.

0 marks: At least two of the areas of the task are performed poorly.
The quantities of reagents are inappropriate for the preparation.
The apparatus set-up for each experiment is unsafe or inappropriate.
The experiments are carried out with insufficient care or the yield is poor.

Guidance for Teachers

Teachers are expected to exercise professional judgement in assessing the competence of their candidates in following the instructions.

Candidates should have been given guidance in the correct use of equipment and this guidance can continue during the practical session for which this PSA forms a part.

If, however, the guidance required is fundamental or frequent, then the student should not be awarded 2 marks.

Most judgements of 2 marks, 1 mark or 0 marks will depend on whether the candidates are able to measure out the quantities of reagents with due care and attention to the hazards associated with each and whether the handling of reagents and the heating is carried out safely.

The method should produce orange or golden-brown crystals of hexaamminecobalt(III) chloride and these can be inspected visually if they occur. Success should not be measured by considering whether crystals actually form, since a number of factors may prevent that from occurring. It is possible to gain full marks for careful working even if no crystals are observed.
It is important to remember when marking these practical exercises that PSA is about student competence and that for a student to score full marks on this exercise perfection is neither expected nor required.