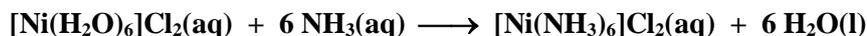


Preparation of hexaamminenickel(II) chloride

Colour of hexaamminenickel(II) chloride depends on the size of its crystals. Big crystals are of saturated blue colour, the small ones of light blue colour. It is well soluble in water and in diluted ammonia. It is insoluble in concentrated ammonia and in ethanol. It can be prepared by the complex reaction of nickel(II) chloride with ammonia solution.



Work

Prepare hexaamminenickel(II) chloride.

Chemicals

- nickel(II) chloride hexahydrate, $\text{NiCl}_2 \cdot 6 \text{H}_2\text{O}$
- ammonia, NH_3 , concentrated water solution, $w(\text{NH}_3) = 0,26$
- ammonium chloride, NH_4Cl
- ethanol, $\text{CH}_3\text{CH}_2\text{OH}$, denaturised spirit

Procedure

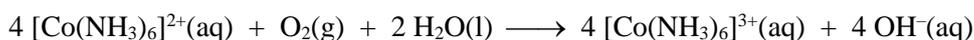
Warning! Ammonia very strongly irritates human respiration system therefore, we must work with them in a fume hood.

Dissolve 14,3 g of nickel(II) chloride hexahydrate in 6,0 cm³ of water. Add 26,1 cm³ of concentrated (26%) ammonia to the prepared solution, under continuous stirring and in small portions. In the first stage of the reaction the poorly soluble nickel(II) hydroxide is formed, but it dissolves as more ammonia is added. Nickel(II) chloride is always contaminated with iron(III) and cobalt(II) compounds, which must be separated by the following procedure:

- ❶ In excess of ammonia, hydrated iron(III) oxide precipitates, which may be separated by the filtration



- ❷ By bubbling the solution with air, we transform the formed hexaamminecobalt(II) complex to hexaamminecobalt(III) complex, which is much more soluble.



Bubbling takes around 30 minutes and is performed in the apparatus depicted in Fig. 1.

- Add one gas-washing bottle (1) with concentrated ammonia before the reactor (2) to eliminate the loss of ammonia during the bubbling. Add ammonia to 1/3 of height of the washing bottle.
- After the reactor (2) another gas-washing bottle (3) with the solution of sulphuric acid must be added to neutralize the unreacted ammonia. It has been found experimentally that, the volume of concentrated sulphuric acid should be 1/10 of volume of ammonia in the bottle (1). We may dilute the acid with water in volume ratio 1:1 or 1:2.

- ❸ When bubbling is finished, add 36 cm³ of the saturated solution of ammonium chloride to the reaction mixture to decrease the solubility of the product. Prepare the solution by dissolving 25,0 g of NH_4Cl in 100 cm³ of saturated ammonia. Use the remaining part of the solution for two decantations of the product. The third decantation with ethanol is necessary to remove last traces of ammonia.

Filter out the product on the Büchner funnel and dry it between two pieces of filter paper. Store the dry product in a well closed vessel.

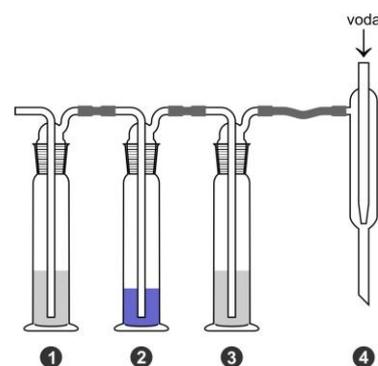


Fig. 1 The apparatus for the preparation of hexaammine nickel(II) chloride

- 1 – conc. ammonia, 2 – reactor,
3 – absorber (sulphuric acid),
4 – water pump

Safety instructions

Nickel(II) chloride hexahydrate – NiCl₂ · 6H₂O

- R45** May cause cancer.
- R23/24/25** Toxic by inhalation, in contact with skin and if swallowed.
- R36/37/38** Irritating to eyes, respiratory system and skin.
- R42/43** May cause sensitization by inhalation and skin contact.
- S22** Do not breathe dust
- S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
- S45** In case of accident or if you feel unwell seek medical advice immediately (show the label where possible)
- S36/37/39** Wear suitable protective clothing, gloves and eye/face protection.

Ammonia – NH₃

- R10** Flammable.
- R23** Toxic by inhalation.
- S16** Keep away from sources of ignition – No smoking
- S38** In case of insufficient ventilation wear suitable respiratory equipment
- S7/9** Keep container tightly closed and in a well-ventilated place.

Ammonium chloride – NH₄Cl

- R22** Harmful if swallowed.
- R36** Irritating to eyes.
- S22** Do not breathe dust