Preparation of copper(I) chloride

Copper(I) chloride is a white powder with sphalerite-like structure. It is moderately soluble in water. It is oxidized on wet air to form yellow and then green copper(II) hydroxide-chloride.

$$4 \operatorname{CuCl}(s) + 2 \operatorname{H}_2O(g) + O_2(g) \longrightarrow 4 \operatorname{CuCl}(OH)(s)$$

Light decomposes copper(I) chloride disproportionally changing its color to blue-black.

 $2 \operatorname{CuCl}(s) \xrightarrow{h\nu} \operatorname{CuCl}_2(s) + \operatorname{Cu}(s)$

Copper(I) chloride may be prepared by the redox (synproportionation) reaction of copper(II) salt solution with powdered copper in the presence of of alkali chloride. Alkali chloride is used to temporarily change insoluble copper(I) chloride to well-soluble tetrachloridocuprate(I) anions.

 $Cu(s) + CuSO_4(aq) + 8 MCl(aq) \xrightarrow{\Delta T} 2 M_3[CuCl_4](aq) + M_2SO_4(aq)$

Changing the equilibrium composition of reaction mixture by diluting, copper(I) chloride is precipitated.

 $M_3[CuCl_4](aq) \xrightarrow{dilution} CuCl(s) + 3 MCl(aq) M \equiv Na, K, NH_4$

Work

Prepare copper(I) chloride.

Chemicals

- copper, freshly prepared by cementation,
- copper(II) sulphate pentahydrate, light blue crystalline substance,
- sodium chloride, white crystalline substance,
- sulphuric acid, concentrated 96 wt% solution, oily colourless liquid,
- ethanol, colourless liquid.

Procedure

Put freshly prepared copper into a 100 cm³ ground-glass flask. Keep in mind that only 90 % of copper will react in the following reaction. Add stoichiometric amount of 10 wt% copper(II) sulphate solution acidified with two drops of concentrated sulphuric acid. The pH of the solution should be ≈ 2 to avoid hydrolysis of aquated copper(II) cations. Finally, add calculated amount of solid sodium chloride. Fix the reflux condenser to the flask and heat the reaction mixture to the boiling (Fig. 1). The reaction is finished when the reaction mixture is colourless.

Filter the hot reaction mixture through a fluted filter paper to at least 6-fold excess of 3 wt% sulphuric acid solution A stem of the filter funnel must be immersed to the solution to prevent the product from contact with air oxygen. Moreover, the mixture should be protected form light, therefore it must be placed in the dark sack (Fig. 2).

When the filtrated touches the acidified solution, white copper(I) chloride precipitates and sediments slowly to the bottom of a beaker. When already settled purify the prepared copper(I) chloride by decantation with 3 wt% sulphuric acid solution. Filter out the wet product quickly on the Büchner funnel, wash it with ethanol and then diethyl ether. Dry it with air flow on the filter for a short time. Immediately put the dried product to the black well-closed test tube.



Fig. 1 Apparatus for preparation of CuCl.





Safety instructions

<u>Copper(II) sulfate pentahydrate – CuSO₄ · 5H₂O</u>

- **R22** Harmful if swallowed.
- **R36/38** Irritating to eyes and skin.
- S22 Do not breathe dust.

Sulfuric acid – H₂SO₄

- **R23** Toxic by inhalation.
- **R34** Causes burns.
- **R49** May cause cancer by inhalation.
- **S23** Do not breathe gas/fumes/vapour/spray (appropriate wording to be specified by the manufacturer).
- 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.

<u>Copper – Cu</u>

R36/37/38	Irritating to eyes, respiratory system and skin.
S26	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S37/39	Wear suitable gloves and eye/face protection.

<u>Sodium chloride – NaCl</u>

R36/37/38	Irritating to eyes, respiratory system and skin.
S26	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S36	Wear suitable protective clothing.

<u>Copper(I) chloride – CuCl</u>

- **R22** Harmful if swallowed.
- S22 Do not breathe dust.

<u>Ethyl alcohol – C₂H₅OH</u>

R11	Highly flammable.

- S7 Keep container tightly closed.
- **S16** Keep away from sources of ignition No smoking.

<u>Acetone – (CH₃)₂CO</u>

- **R11** Highly flammable.
- **S9** Keep container in a well-ventilated place.
- **S16** Keep away from sources of ignition No smoking.
- S23 Do not breathe gas/fumes/vapour/spray (appropriate wording to be specified by the manufacturer).
- **S33** Take precautionary measures against static discharges.

<u>Diethyl ether – (C₂H₅)₂O</u>

- **R12** Extremely flammable.
- **R19** May form explosive peroxides.
- **S9** Keep container in a well-ventilated place.
- **S16** Keep away from sources of ignition No smoking.
- S29 Do not empty into drains.
- S33 Take precautionary measures against static discharges.