# Tittle: Preparation of copper(I) chloride

Work instructions

Task: Prepare copper chloride from 1.50 g of copper prepared by cementation. The copper recovery is 90.0%.
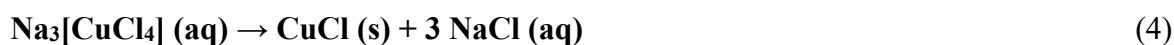
Theory

Copper(I) chloride is a white powder with the texture of sphalerite. It is sparingly soluble in water and in dilute sulfuric acid. In moist air it oxidises to yellow and later to green copper chloride hydroxide.

$$4\ CuCl\ (s) + 2\ H_2O\ (g) + O_2\ (g) \rightarrow 4\ CuCl(OH)\ (s) \tag{1}$$
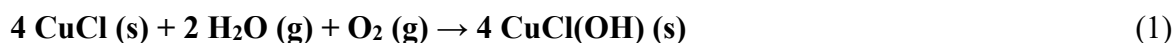
In the light it decomposes by a disproportionation reaction and takes on a blue-black colour.

$$2\ CuCl\ (s) \rightarrow CuCl_2\ (s) + Cu\ (s) \tag{2}$$

Copper chloride is prepared by the redox (synproportionation) reaction of a copper salt with copper, in the presence of water-soluble alkali metal chloride or ammonium chloride. The copper chloride formed is insoluble in water and would deposit on the surface of the copper metal, thus preventing further reaction. We therefore add to the reaction medium sufficient amount of soluble chloride to form soluble cupric chloride complex. By changing the equilibrium composition of the cupric chloride anions by diluting the solution, copper chloride is formed.

$$Cu\ (s) + CuSO_4\ (aq) + 8\ NaCl\ (aq) \rightarrow Na_3[CuCl_4]\ (aq) + Na_2SO_4\ (aq) \tag{3}$$

$$Na_3[CuCl_4]\ (aq) \rightarrow CuCl\ (s) + 3\ NaCl\ (aq) \tag{4}$$

Equipment: Erlenmeyer flask (250 ml) with stopper, watch glass, graduated cylinder (25 ml), beakers (2 × 150 ml, 400 ml), funnel, Büchner funnel, ball condenser, spoon, filter paper, ring stand, scales

Chemicals: copper (pre-prepared by cementation), copper sulfate pentahydrate, sodium chloride, sulfuric acid (96%), ethanol

Procedures:

1. Read the risk statements and safety warnings for working with chemicals. Wear safety goggles and gloves!

2. Read the full procedure first. Visualise the procedure: sketch each apparatus and note down the quantities of substances, write down the steps separated by arrows, for example.

Preparation of copper chloride

Note: Perform all copper chloride isolation operations very quickly to minimize contact with oxygen, moisture in the air, and light.

1. Weigh 1.50 g of fresh powdered copper prepared in advance by cementation into an Erlenmeyer flask with a stopper. The recovery of copper in the reaction is about 90%, so the actual amount of copper reacted will be 1.35 g.
2. Prepare a 10% solution of copper sulfate by weighing 5.30 g of its pentahydrate dissolved in 28.7 ml of water and adding a drop of concentrated sulfuric acid.
3. To the copper in the Erlenmeyer flask, add the acidified copper sulfate solution using a funnel.
4. Finally, add 9.93 g of sodium chloride.
5. Place a ball condenser on the Erlenmeyer flask, mounted on a ring stand, and bring the contents in the flask to the boil.
6. Stop boiling if the solution in the flask is colourless and clear.
7. Prepare a 3% sulfuric acid solution (17.3 ml of concentrated sulfuric acid + 990 ml of water).
8. Filter the still hot solution into a 3% sulfuric acid solution of at least six times the volume of the filtered solution.
9. The resulting solution of copper tetrachloride must not come into contact with air, therefore the funnel stem must be immersed in an acid solution in a beaker.
10. The resulting copper chloride must be protected from light, so wrap the beaker with filter paper, foil, or a black bag.
11. Purify the resulting copper chloride by decanting with the remaining 3% sulfuric acid solution.
12. Quickly filter off the wet copper chloride on a Büchner funnel, wash with anhydrous ethanol and dry by brief air percolation.
13. Weigh the product and calculate the yield.

Management of chemical substances

Chemicals	Form	H-statements	P-statements
Cu	Solid, powdery	H228, H410	P210, P273, P370 + P378
$CuSO_4 \cdot 5H_2O$	Solid	H302, H315, H319, H410	P273, P302 + P352, P305 + P351 + P338

Chemicals	Form	H-statements	P-statements
NaCl	Solid	---	---
H_2SO_4	Liquid, 96%	H290, H315, H319	P280, P302 + P352, P337 + P313, P305 + P351 + P338
C_2H_5OH	Liquid, 96%	H225, H319	P210, P233, P305 + P351 + P338

Sources of risk and assessment of risk severity

If all the principles for working with chemicals are followed and personal protective equipment (gloves, goggles, lab coat) is used, there is no risk.

Waste management method

We dispose of chemicals in designated collection containers.

Risk reduction measures

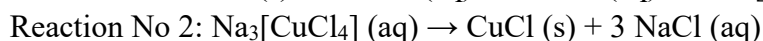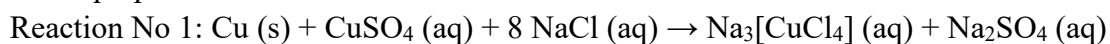Use of personal protective equipment (goggles, gloves, lab coat).

References

1. Ondrejkovičová, I. et al. *Praktikum z anorganickej chémie*. 2. vyd. Bratislava: Nakladateľstvo STU v Bratislave, 2016. 237 s. ISBN 978-80-227-4653-3.

Worksheet

Calculations

1. Calculate the theoretical yield of copper chloride if 1.5 g of powdered copper is used for its preparation.

   Reaction No 1: Cu (s) + $CuSO_4$ (aq) + 8 $NaCl$ (aq) → $Na_3[CuCl_4]$ (aq) + Na_2SO_4 (aq)

   Reaction No 2: $Na_3[CuCl_4]$ (aq) → $CuCl$ (s) + 3 $NaCl$ (aq)

   $M(Cu)$ = ... $g.mol^{-1}$, $M(CuCl)$ = ... $g.mol^{-1}$

2. Calculate the theoretical yield of copper chloride if we assume that the copper recovery is 90%.

3. Calculate the practical yield of copper chloride in %.

   PV = ... g, TV = ... g

   PV(%) = ... %

Observation

1. Describe the appearance of copper chloride.

2. Search for the basic physicochemical properties of copper chloride.

Feature	Value
Solubility in water	
Solubility in other solvents	
Melting point	

Yield

1. Record the mass of copper chloride prepared.

   m(CuCl) = ... g

Questions

1. Write the equations of the reactions for the preparation of copper chloride.

2. Using the example of the preparation of copper chloride, explain what is synproportionation.

3. Explain why the resulting solution of copper tetrachloride must not come into contact with air and must be protected from sunlight.

4. Explain the significance of the decantation of copper chloride with dilute sulphuric acid.

5. Explain why copper chloride is washed on a Büchner funnel with anhydrous ethanol.

6. Describe the use of copper chloride.
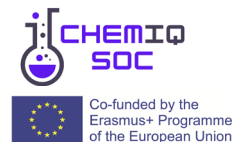
Conclusion

Briefly summarize the objective of the experiment, the main results and compare them with the expected values.

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