#### **Oxalato complexes**

The oxalate anion is derived from oxalic acid, systematically named ethanedioic acid HOOC–COOH. As a ligand, it acts commonly as a tetradentate ligand, bridging two metal atoms (Fig. 1). In mononuclear complexes it is bidentatly coordinated, forming five-membered chelate rings (Fig. 2). Oxalate ligand is also rarely tridentatly coordinated, where the oxygen atom of chelate ring is simultaneously bonded to another metal atom.



Fig. 1 Schematic representation of tetradentatly coordinated oxalate anion.



Fig. 2 Schematic representation of bidentatly coordinated oxalate anion.

## Preparation of potassium bis(oxalato) cuprate(II) dihydrate

Potassium bis(oxalato) cuprate(II) dihydrate  $K_2[Cu(C_2O_4)_2] \cdot 2H_2O$  is a blue crystalline substance insoluble in common organic solvents, but soluble in aqueous solutions of oxalate salts. Its solubility increases rapidly at higher temperature. In water the substance slowly decomposes forming copper(II) oxalate. When heating over 50 °C crystal water is eliminated, and over 260 °C it is totally decomposed. In the complex anion  $[Cu(C_2O_4)_2]^{2-}$  copper atom has coordination number 4 (Fig. 1) and a square-planar geometry.

Potassium bis(oxalato) cuprate(II) dihydrate may be prepared by a reaction of copper(II) oxalate with potassium oxalate solution, according to the equation 1:

$$\operatorname{CuC}_2\operatorname{O}_4(s) + \operatorname{K}_2\operatorname{C}_2\operatorname{O}_4(\operatorname{aq}) \longrightarrow \operatorname{K}_2[\operatorname{Cu}(\operatorname{C}_2\operatorname{O}_4)_2](\operatorname{aq}) \tag{1}$$

Copper(II) oxalate precipitates from the copper(II) sulphate solution after adding equimolar quantity of potassium oxalate solution, according to the equation 2:

$$CuSO_4(aq) + K_2C_2O_4(aq) \longrightarrow CuC_2O_4(s) + K_2SO_4(aq)$$
(2)

The overall equation 3 for the preparation of  $K_2[Cu(C_2O_4)_2]$  could be expressed as follows.

$$CuSO_4(aq) + 2 K_2C_2O_4(aq) \longrightarrow K_2[Cu(C_2O_4)_2](aq) + K_2SO_4(aq)$$
(3)

Prepare potassium bis(oxalato) cuprate(II) dihydrate from 5,00 g of copper(II) sulphate pentahydrate.

## **Chemicals**

- copper(II) sulphate pentahydrate,  $CuSO_4 \cdot 5H_2O$ , or another water-soluble copper(II) salt,
- potassium oxalate monohydrate,  $K_2C_2O_4 \cdot H_2O$ .

# Procedure

Prepare 30 wt% copper(II) sulphate solution and 25 wt% potassium oxalate solution. Heat both solutions up to 90 °C. Pour the copper(II) solution slowly under continuous stirring to the oxalate solution. Let stand the mixture freely to cool to room temperature and subsequently continue cooling it in ice water. Filter out crystals formed by cooling on a filter funnel with glass frit, wash them with small volume of cold water and dry them up at 50 °C.

## Decomposition of potassium bis(oxalato) cuprate(II) dihydrate

Mix 0,5 g of potassium bis(oxalato) cuprate(II) dihydrate with small amount of cold water. Heat the suspension to a boil. Add cold water stepwise in small portions until the solution turns to blue. Let cool the blue solution freely to room temperature. The complex compound slowly decomposes and a precipitate of copper(II) oxalate is excluded from the solution (eq. 4).

$$K_2[Cu(C_2O_4)_2](aq) \xrightarrow{\Delta T} K_2C_2O_4(aq) + CuC_2O_4(s)$$
(4)

### Safety instructions

#### <u>Copper(II)</u> sulfate pentahydrate – CuSO<sub>4</sub> <u>· 5H<sub>2</sub>O</u>

R22	Harmful if swallowed.
R36/38	Irritating to eyes and skin.
<b>S22</b>	Do not breathe dust.

#### <u>Potassium oxalate monohydrate – $K_2C_2O_4 \cdot H_2O$ </u>

- **R21/22** Harmful in contact with skin and if swallowed.
- **S24/25** Avoid contact with skin and eyes.

#### <u>Potassium sulfate – K<sub>2</sub>SO<sub>4</sub></u>

R22	Harmful if swallowed.
<b>S36</b>	Wear suitable protective clothing.