Recrystallisation of copper(II) sulphate pentahydrate

Non-isothermal crystallization

- Build up an apparatus for atmospheric filtration. (Fig. 1) and make a folded (French) paper filter.
- Let heat a filter funnel in an oven to the temperature at least 100 °C.
- Weigh out 15,0 grams of "dirty" copper(II) sulphate pentahydrate.
- Dissolve it by the constant stirring with glass rod in a beaker in 18 ml of distilled water, measured with a graduated cylinder. Warning! Stir carefully because the sand can scratch the bottom of a beaker.
- Warm up the solution in a beaker to *cca* 90 °C.
- Filter out the hot solution in small parts through the filter funnel directly to an evaporating dish. After each portion heat the solution in a beaker again up to 90 °C.
- After filtration put a clean beaker below the funnel and penetrate the filter with a glass rod. Wash out the whole filter cake with water from the filter to the beaker. Wait for a while until sand seats on the bottom and carefully remove the slightly blue solution from the beaker. Add another portion of water. Repeat this step until there is only pure water in a beaker.
- After last removing of water put the beaker with wet sand to the oven heated over 120 °C. After drying weigh the sand.
- The evaporating dish with blue solution place over a water bath (Fig. 2) and wait until the first crystals appear on its surface. Using a glass rod try to dive the crystals back to the solution. If crystals are still present stop heating and immediately measure the temperature of the solution.
- Quickly pour hot solution from the evaporating dish to crystallization dish and place it in a coolest part of a laboratory. After the cooling to room temperature pale blue crystals appear. Measure the temperature of solution again (fig. 3).
- Build up an apparatus for vacuum filtration. (Fig. 4). Transfer the entire content of the crystallization dish to the round paper filter in Büchner funnel. Under vacuum exhaust the liquor to the suction flask to leave the crystals almost dry. Warning! To protect the pump, there must be a safety-flask connected between a suction flask and a pump (Fig. 4).
- Obtained crystals will be still wet, therefore dry them up between two filter papers applying the pressure of your hand. After drying weigh the product and calculate the theoretical yield of recrystallization.

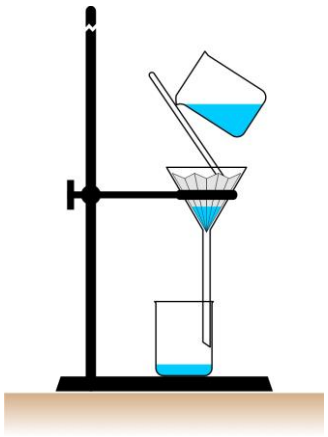

Fig. 1
Atmospheric (normal-pressure) filtration

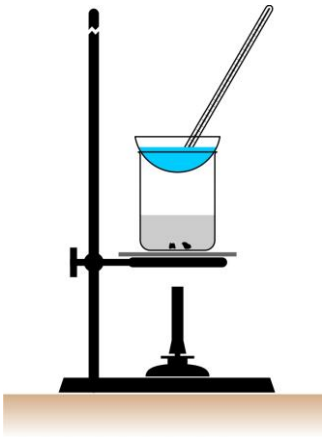

Fig. 2
Water bath
(Evaporation)

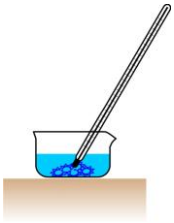

Fig. 3
Crystallization
(Cooling)

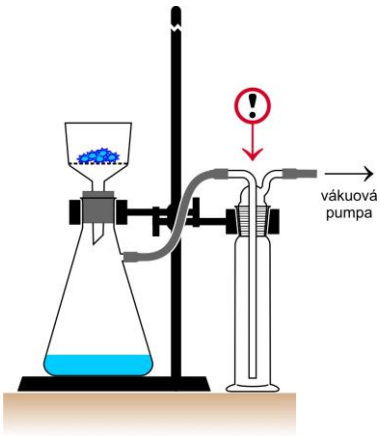

Fig. 4
Vacuum filtration
(use safety-flask)

★ *Note*

Even if we can measure the temperature of a solution above a water bath, it is rather difficult to estimate the moment of its saturation. It is a question of subjective feeling and only an experienced chemist is able to do it correctly. Just in this step we could make the great fault in determination of weight fraction (necessary for next calculations). However, the composition of the saturated solution can be determined much more exactly: **1.** Weighing out an empty evaporation dish (m_0) and then a dish with saturated solution (m) we obtain exact weight of the saturated solution ($m' = m - m_0$). **2.** Using the known weight (m^*) of pentahydrate ($w^* = 0{,}639$) used in the procedure we can then calculate the exact weight fraction (w) of copper sulphate in its saturated solution ($w = m^* w^* / m'$). We should compare the calculated weight fraction with a value obtained from the solubility curve for the measured temperature of the saturated solution.

Isothermal crystallization

Pour the obtained filtrate into a clean crystallisation dish, cover it by filter paper with small holes and let stay it for one week at room temperature to crystallize isothermally. Compare the size of the crystals obtained by both non-isothermal and thermal crystallization. (Fig. 5).

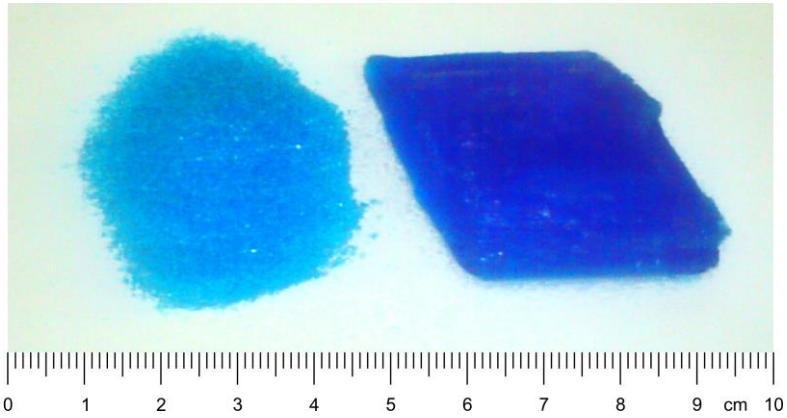

Fig. 5 Comparison of the size of the crystals obtained by non-isothermal (left) and isothermal (right) crystallization.

Safety instructions

***Copper(II) sulphate pentahydrate* – $CuSO_4 \cdot 5H_2O$**

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

Exercise

- Explain the words: dissolution, solution, saturated solution, saturation temperature, solubility curve, filtrate, filtride (filter cake), recrystallization, second crystallization, isothermal / non-isothermal filtration, atmospheric / vacuum filtration, reactant, product, theoretical / practical yield.