
LABORATORY WORK No. 5

DISTURBED CRYSTALLIZATION AND SUBLIMATION

CRYSTALLIZATION

■ PRINCIPLE:

- Crystallization is a fundamental procedure for obtaining pure solid substances from their solutions. Solid substance is dissolved in a suitable solvent, impurities are filtered out and dissolved substance is let crystallize. Crystals are separated by filtration from the rest of solution - so called mother liquor.

- During crystallization, crystals exclude so that at first crystal nuclei are formed and from them crystals are increasing.

- We differentiate three kinds of crystallization:

- **by cooling of solution saturated in high temperature**

- **by evaporation of part of the solvent**

- **by adding the third substance**

- **ad a) Crystallization by cooling of solution saturated in high temperature**

- We prepare saturated solution in high temperature, filtrate it under this temperature and let it crystallize.

- During **free crystallization**, we let the solvent evaporate freely under gradual cooling. Small amount of crystal nuclei is created, crystals are bigger.

- During **disturbed crystallization**, we cool the saturated solution down rapidly, e.g. under running water. Big amount of crystal nuclei is created, with relatively small crystals on them.

- **ad b) Crystallization by evaporation of some part of the solvent**

- Solution is brought into crystallization by evaporation of a part of solvent, e.g. by evaporation in water bath or in desiccator.

- **ad c) Crystallization by adding the third substance**

- Solubility of substance can be (instead of evaporation of solvent) also influenced by adding the third substance (for example ethanol) which is mixed with solvent but creating crystals are not dissolved in the third substance.

■ **TASK No. 1 CLEANING TECHNICAL COPPER SULFATE BY CRYSTALLIZATION**

■ **CHEMICALS:**

Copper sulfate, pentahydrate $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ (technical), ethanol $\text{C}_2\text{H}_5\text{OH}$, distilled water

■ **AIDS:**

beaker 250 ml, glass rod, Erlenmeyer flask 250 ml, folding filter, filter for vacuum filtration, speedfiltering funnel, Büchner funnel, suction flask, seal, wash bottle with distilled water, laboratory scales, graduated cylinder 100ml, Bunsen burner, wire gauze, support stand, metal ring

■ **PROCEDURE:**

Weigh $m_1=10\text{g}$ of technical copper sulfate $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ (to three decimal places), dissolve it in 40 ml of distilled water.

Filter the solution in hot temperature by using the folding filter into the Erlenmeyer flask.

Make it thicker for crystallization by evaporation of solvent (between 10-20 ml).

Treat the solution by disturbed crystallization under running water.

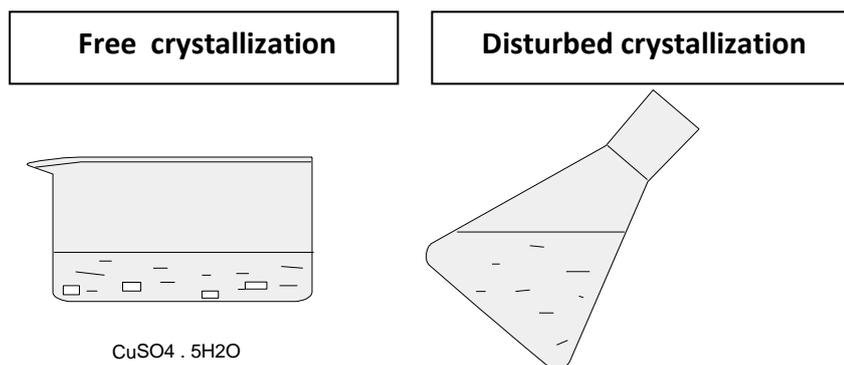
If the substance does not crystallize, add a few drops of ethanol (10-15 drops).

Filter the new crystals in the Büchner funnel, wash them with mother liquor.

Dry and weigh – you will get the weight of the product $m_2[\text{g}]$.

Calculate the yield of crystallization μ .

■ **Drawing of apparatus:**



■ **CALCULATIONS:**

The calculation of the yield μ for the crystallized $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ in percentages

(we use direct proportion):

$$\begin{array}{l} \uparrow \quad m_1 = 10,000 \text{ g} \dots\dots\dots 100 \% \quad \uparrow \\ \quad \quad \underline{m_2 \text{ g} \dots\dots\dots x \%} \\ \mu = \frac{m_2}{m_1} \times 100 = \dots\dots \% \end{array}$$

■ **CONCLUSION:**

We obtainedg of pure substance by crystallization of copper sulfate, i.e.....%.

SUBLIMATION

■ **PRINCIPLE:**

Sublimation is one of the fundamental methods of cleaning and separating substances in a laboratory.

The principle of this method is the transition of substance from the **solid state** into the **gaseous state** in hot temperature and then from the gaseous state into the solid by cooling.

Advantage of sublimation:

There are not a lot of losses during sublimation, the final product is without chemical impurities and time demands are smaller. The lower is the the temperature of the cooled area the smaller crystals are formed.

TASK NO. 2 CLEANING THE MIXTURE OF IODINE AND SAND BY SUBLIMATION

■ **CHEMICALS:**

Iodine I₂, sand (eventually mixture of I₂ and sand), ice, ethanol

■ **AIDS:**

beaker 250 ml, distilling flask, scoop, burner, wire gauze, a metal ring, watch-glass, stopper

■ **PROCEDURE:**

Take away carefully one spoon /cca 5g/ of polluted iodine and pour it into the dry and clean beaker.

Put the beaker on the wire gauze placed on tripod or ring which is fixed on the stand.

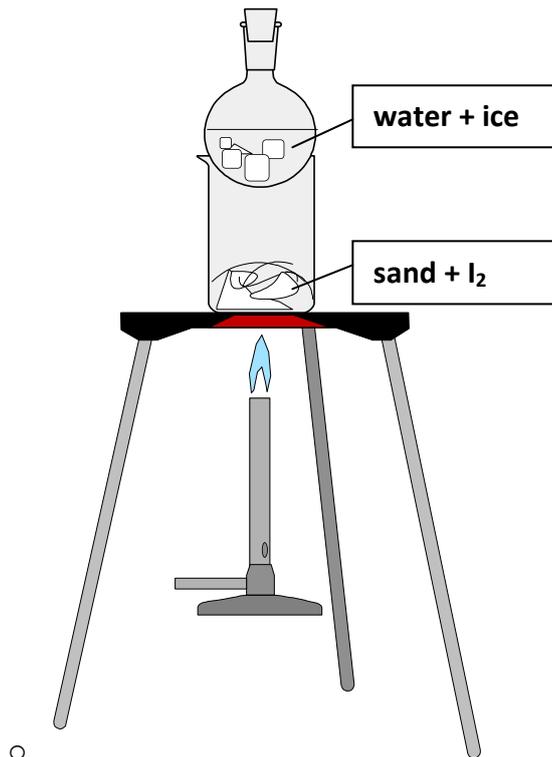
Pour water into the distilling flask and add ice. Close the flask with the stopper. Put the distilling flask carefully into the beaker with polluted iodine.

Seal up the spout of the flask by cotton wool.

Heat up the flask with moderate flame until iodine vapours start to release After finishing of sublimation, scrape off carefully the iodine crystals from the bottom of the cooling vessel and the walls of the beaker and put them on the watch glass. Remove by ethanol the rest of iodine from the beaker and the distilling flask.

Pour the clean iodine from the watch-glass into the prepared vessel and describe its appearance.

Sublimační aparatura



■ CONCLUSION:

We repurified the mixture of iodine by sublimation so that we obtained clean iodine with its characteristic appearance (describe appearance of repurified substance).

■ SAFETY:

It is necessary to heat the mixture up carefully and slowly to avoid huge development of vapours out of cooled area. Let the apparatus cool down after you finish the work and then reassemble it.



STUDENT'S SHEET No.5
CRYSTALLIZATION AND SUBLIMATION

1. Translate these vocabulary:

rozpouštědlo
ochlazení
rušená krystalizace
krystaly
třetí látka
skleněná tyčinka
síran měďnatý
odpaření
tekoucí voda
zahustit
výtěžek
laboratorní váhy
ovlivnit
krystalizace
krystalizovat
hmotnost

1. Use these words into sentences:

crystallization
crystals
crystallize
beaker
calculate

2. Complete the text with these words:

method

solid

crystallizes

Crystallization is the fundamental _____ for obtaining clean and _____ substances from their solution. The solid substance are dissolved in the solvent, impurities are filtered out and the dissolved substance _____. Crystals are separated by filtration from mother liquor.

gaseous state

sublimation

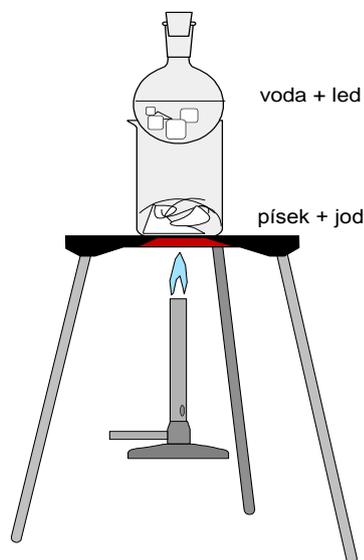
cooling

_____ is one of the fundamental methods of cleaning and separating substances in a laboratory.

The principle of this method is the transition of substance from the **solid state** into _____ and then _____ from the gaseous state into the solid one.

3. Mark the apparatus with these words – **sand + iodine, beaker, stopper, water + ice, distilling flask, burner, wire gauze,**

Sublimační aparatura



4. Put the words into the right order:

a/ with/the/flame/Heat/up/moderate/flask.
 b/ dissolved/solvent/Solid/are/in/substances/suitable.
 c/ Cleaning/crystallization/technical/sulfate/by/cooper.
 d/ on/gauze/the/Put/beaker/the/wire.
 e/ one/methods/fundamental/of/is/Sublimation/the.

5. Find these words in these letters:

Evaporation, sublimation, solid, solvent, crystals.

KLABCSOLLOIDSCRYSTALSGSDRTZUNMBONITAROPAVEBBEAKERHEAAAT

FLASSSKBULISUBSTTANCECUERTUSKJVMNBSOLIDHZTRJPELSTVUSMNBUL

SOLVENTNARUSTUVLKMPRUTSSSOOLLIIDDDSUBLIMATIONNNTTIIOOMM