Name of the project:	**Digitization of chemistry experiments to improve the quality and support chemistry teaching in secondary schools**
Acronym:	**ChemIQSoc**
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Tittle: Free and disturbed crystallisation

Work instructions

Task:

1. Weigh 10 g of the blue vitriol, mix and dissolve and then filter the resulting suspension under normal pressure.
2. Concentrate the resulting filtrate and crystallise by evaporation of the solvent for future laboratory exercise.

Theory

Filtration is the most common method for separating solids from liquid or gaseous media. It is carried out by means of a suitable filter baffle, which is made of a porous material (in the laboratory this is most often filter paper, glass frit, or a filter sail, in operational practice a filter sail or sand).

Crystallisation is the most common method for separating and purifying substances. It is the separation of a solid from a solution or melt.

Crystallisation from solution can be done by:

a. free evaporation of a part of the solvent–so-called *free crystallisation***,** in which large crystals are formed by slow growth,
b. cooling of a hot saturated solution–so-called *disturbed crystallisation*, in which small, tiny crystals are rapidly formed.
c. addition of a substance with the same ion–so-called *salting out*.

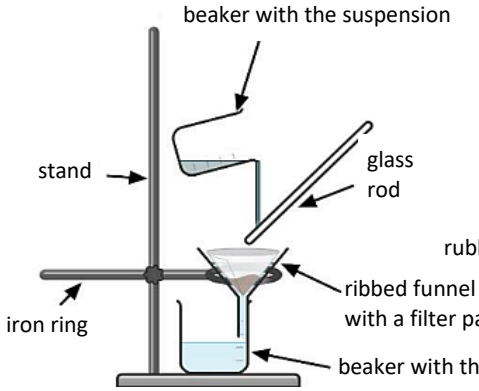
From aqueous solutions, crystals are usually precipitated in the form of so-called hydrates – i.e. substances in whose crystal lattice water molecules are bound, the number of which is given in the chemical formula (e.g. $CuSO_4 \cdot 5H_2O$). If this water bound in the crystals were removed, e.g. by heating and evaporation, the crystals would collapse and form a so-called anhydrous salt.

Copper sulfate crystallised from aqueous solutions forms deep blue crystals of the trigonal system. It loses crystal water on heating, changes to monohydrate at 100°C and becomes an anhydrous salt at 200°C. The anhydrous salt is a white hygroscopic powder. It decomposes with intense annealing:

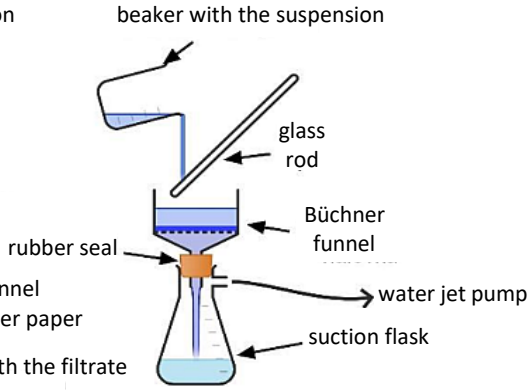
$$\mathbf{2\ CuSO_4 \rightarrow 2\ CuO + 2\ SO_2 + O_2} \qquad (1)$$

Technical $CuSO_4 \cdot 5H_2O$ is usually contaminated with a number of other substances (most commonly $CaSO_4$, $MgSO_4$, $ZnSO_4$, and $FeSO_4$) that can be removed by recrystallisation.

Apparatus:



Scheme 1 Apparatus for filtration under normal pressure



Scheme 2 Apparatus for filtration under reduced pressure

Equipment: beakers (150, 250, 400 ml), watch glass, graduated cylinder (100 ml), water jet pump, Büchner funnel, suction flask, ribbed funnel, iron ring, filter paper, scissors, glass rod, spatula, crystallising dish

Chemicals: copper(II) sulfate pentahydrate

Procedures:

Filtration under normal pressure

1. Weigh 10 g of the blue vitriol on a watch glass.
2. Transfer this amount into a 250 ml beaker.
3. Measure 100 ml of water into the beaker through a 400 ml beaker and a measuring cylinder (always pour water from the beaker into the measuring cylinder, never directly from the tap).
4. Stir with a glass rod and then dissolve all the blue vitriol.
5. Subsequently, filter the hot solution (approx. 50°C) under normal pressure. **Keep the filtrate for the second part of the work!**

Filtration under reduced pressure

1. Concentrate the filtrate by evaporation to a volume of about 50 ml, and then cool with cold water while stirring. This will eliminate large crystals creation–**disturbed crystallisation.**
2. Filter the resulting crystals under reduced pressure (aspirate on a Büchner funnel).
3. Place the filter cake on a watch glass and leave it to dry.
4. Transfer the remaining mother liquor to a crystallisation dish and leave until the next laboratory exercise–**free crystallisation**.
5. Evaporation of the solvent can take several days or weeks.
6. Weigh the dry crystals.

Management of chemical substances

Chemicals	Form	H-statements	P-statements
$CuSO_4 \cdot 5H_2O$	Solid	H302, H318, H410	P273, P280, P301 + 312, P305 + P351 + P338

Sources of risk and assessment of risk severity

Copper sulfate is harmful if ingested, irritates the skin, and penetrates the skin. Causes serious eye irritation. It is highly toxic to aquatic organisms, with long-term effects. Students use protective equipment when working with this substance. Weighing and handling of this substance is supervised by the teacher. Acceptable risk.

Waste management method

Residues of copper sulfate pentahydrate shall not be disposed of with municipal waste and shall not be discharged to sewers. In case of spillage, sweep up the substance and place it in a carefully labelled closed container designed for this purpose.

Risk reduction measures

Wear a face shield or safety glasses. Wear protective gloves. Gloves must be inspected before use. Use proper glove removal technique without touching the outer surface of the gloves to prevent skin contact with this product. Do not eat, drink, or smoke while working. Wash hands with soap and water after work or when work is interrupted or treat with a protective cream. In case of an accident or if you feel unwell, inform the teacher immediately. Prevent further leakage or spillage unless there is a risk involved. Do not allow to enter drains. Prevent discharge into the surrounding environment.

Worksheet

Calculations

1. Calculate the mass fraction of copper sulphate in the solution if you used 10.0 g of copper sulphate pentahydrate and 100 ml of water to prepare the solution.
   $\rho(H_2O, 20°C)$ = ... $g.cm^{-3}$

2. Calculate the mass of copper sulphate pentahydrate formed by cooling 50 g of copper sulphate solution saturated at 50°C to 20°C.
   $s(CuSO_4, 50°C)$ = ... g compound/g solution, $s(CuSO_4, 20°C)$ = ... g compound/g solution

Observation

1. Describe the difference between perturbed and free crystallization.

2. Search for the basic physicochemical properties of blue vitriol.

Feature	**Value**
Solubility in water (20°C)	
Solubility in water (50°C)	
Solubility in other solvents (20°C)	
Melting temperature	

Yield

1. Record the mass of blue vitriol obtained by disturbed and free crystallization.

   Disturbed crystallisation: m($CuSO_4.5H_2O$) = ... g
   Free crystallisation: m($CuSO_4.5H_2O$) = ... g

Questions

1. Explain the importance of crystallization in the preparation of inorganic compounds.

2. Describe how the solubility of copper sulphate in water changes as the temperature of the solution increases.

3. The solubility of substance X cold and hot is similar. Decide whether free or disturbed crystallization is preferable. Justify your statement.

4. Explain why it was necessary to filter the blue vitriol solution before evaporation.

5. Compare the size of the filter area and the filtration rate when using a plain and a pleated filter. Recommend the most suitable filter for blue rockfish filtration.

6. Compare the size of crystals obtained by perturbed and free crystallization.

7. Describe the use of blue vitriol.

Conclusion

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

Disclaimer

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