

Experiment 6: Growing crystals

How do crystals form? Suppose that you wanted to grow crystals of a chemical AB (a solid at room temperature) where A^+ is the cation and B^- the anion.

You start by preparing a solution of a certain concentration of AB in a given solvent. Most of the time the ions A^+ and B^- see only solvent molecules surrounding them. Once in a while they get close to other A^+ and B^- ions and when that happens there is an attractive force between them (remember that AB was a solid). Very often the AB pairs get pulled apart by interactions with the solvent but sometimes they stay close together. The process repeats itself until several AB units are able to stay together, reaching a “critical size”. This is what we call the nucleation process. As this very small AB crystal moves around in solution it gathers more AB units, increasing its size. At some point the crystal is large enough that it “precipitates” to the bottom of the container where it continues to act as a nucleation site until it reaches equilibrium with the surrounding solution.

Several factors affect the nucleation process: the concentration of the starting solution, the temperature of the surroundings as the crystallization proceeds, the presence of a pre-formed nucleation site (such as a “seed” crystal) and whether the solution’s container is open or closed.

Starting with a very concentrated solution tends to produce imperfect small crystals: the nucleation process happens too fast, not allowing the AB pairs to get oriented in the proper way.

The temperature of the surroundings determines the temperature of the solution, setting the rate at which the solvent evaporates. Also, if the surroundings are too hot the molecules in the solution have a high thermal energy and the formation of durable AB nucleation sites is more difficult.

The presence of a pre-formed nucleation site usually helps the formation of larger crystals. New AB pairs are attracted to the seed crystal.

When the solution is kept in an open container (often covered with a watch glass or Al foil with a few holes) the solvent can evaporate and the concentration of AB slowly increases, favoring the formation of nucleation sites. If the container is closed the temperature of the surroundings has to be manipulated so that the solubility of AB decreases with time.

Part A: What do crystals look like under the microscope? To answer this question different groups will prepare different crystals starting with the following solutions: NaNO_3 , $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{K}_3\text{Fe}(\text{CN})_6$ and $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$. We will also observe how a supersaturated solution of sodium nitrate crystallizes on a slide under the microscope.

Background

Part A. Obtaining crystals

Experimental Procedure

1. You will be working in pairs of students. Ask your instructor to assign you a compound. Prepare the corresponding solution by dissolving in a clean and dry paper cup one of the following:

- NaNO_3 : 40 g of NaNO_3 in 50 mL of distilled water.

- $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$: 10 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 50 mL of distilled water.

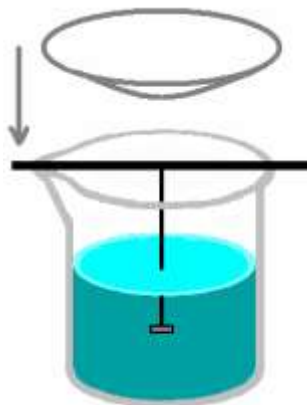
- $\text{K}_3\text{Fe}(\text{CN})_6$: 20 g of $\text{K}_3\text{Fe}(\text{CN})_6$ (potassium ferricyanide or “red prussiate of potash”) in 50 mL of distilled water.

- $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$: 20 g of $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ (potassium aluminum sulfate or “alum”) in 140 mL of distilled water.

2. Place your beaker with a stir bar on a hot plate in a medium setting until all the solid has dissolved. You need to remove any undissolved solid or sediment by either filtering or decanting.

3. Place your beaker in one of the fume hoods and cover it with a watch glass or parafilm. Make sure there is a small opening left to allow for the water to evaporate slowly.

4. Do not disturb the beaker for at least one week.



The following week:

5. Examine the beaker to see if any large crystals have formed. If this is the case, use a pair of tweezers to pick up a good looking crystal (ask for your instructor’s advice), preferably around 3-5 mm long, one with smooth surfaces. You will use it as a seed crystal to obtain even larger crystals. If there are no crystals at all in your beaker you should wait for one more week leaving the beaker uncovered.

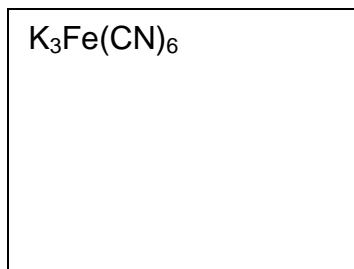
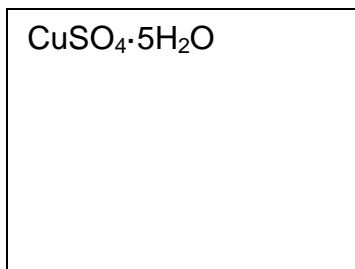
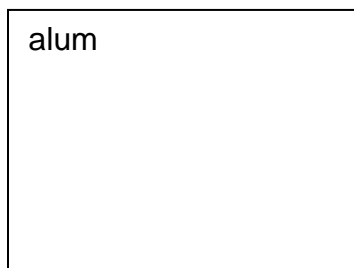
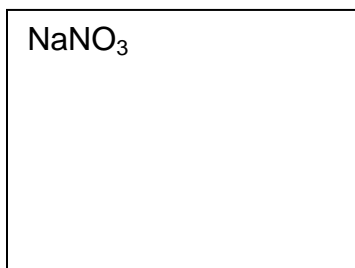
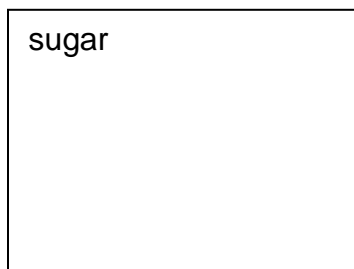
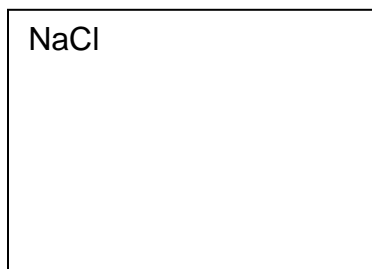
If your beaker is full of very small crystals you should warm the beaker, redissolve the crystals and without covering it leave it under the hood for one more week.

6. Using your seed crystal.

Prepare again a solution containing the salt you are trying to crystallize. You must glue the seed crystal to a piece of string provided by your instructor and attach it to a piece of wire or an inoculator (as shown in the figure to the right) so that the crystal is hanging in the beaker without touching any surfaces. Cover the beaker with a watch glass so that the solution will slowly evaporate. Check the beaker after a week to see how large a crystal you got. Observe it under the microscope.

Part B: The following crystals will be available for looking under the microscope: NaCl, $C_{12}H_{22}O_{11}$ (sucrose or table sugar), $NaNO_3$, $CuSO_4 \cdot 5H_2O$, $K_3Fe(CN)_6$ and $KAl(SO_4)_2 \cdot 12H_2O$.

Take a few crystals and put them on a microscope slide. Observe them carefully and draw them in the spaces below.



Part C: Fill a small dropper with hot supersaturated solution of $NaNO_3$. Squirt a few drops of the liquid on the microscope slide and watch carefully as the crystals grow. Draw what you see in the space below.



Part B.
Observing
crystals under
the microscope

Part C.
Watching a
crystal form
under the
microscope