Crystallisation Resources

Further information and resources for students and teachers participating in the National Crystal Growing Competition

SSPC

iCRAG SFI RESEARCH CENTRE IN APPLIED GEOSCIENCES

Contents

Introduction to the National Crystal Growing Competition

What is the National Crystal Growing Competition?

Very simply, the National Crystal Growing Competition challenges participants to grow their own crystals! The aim of the competition is for an individual student to grow a single crystal using any of the materials listed in this booklet or on the website:

https://bit.ly/crystalcomp

Entries into the national competition will be judged by SSPC and iCRAG experts. The national finals will take place virtually at an online event hosted by SSPC and iCRAG after the Easter break.

Who can take part?

The competition is open to both primary and post-primary school students in Ireland. There will be one winner from each category (one primary and one post-primary winner). Teacher supervision is required for crystallisation of compounds other than salt and sugar. Please view the Material Safety Data Sheets for Health and Safety instructions and indications for materials available on https://bit.ly/crystalcomp.

Links to Curriculum:

Junior Cycle Science:
Nature of science: Investigating in Science, Communicating in Science
Chemical World: Building Blocks, Systems and Interactions
By taking part in this competition, students will learn about: the particulate nature of matter (atoms, bonding, molecules, compounds, mixtures), solutions, solubility, crystal structure, physical properties of matter.

Leaving Certificate:
Core: 1. Periodic Table and Atomic Structure, 4. Volumetric Analysis, 7. Organic Chemistry
Options: 2A Materials
By taking part in this competition, students will learn about: organic chemistry, physical properties of matter, recrystallisation, percentage yield.

Crystals

What are Crystals?

Crystals are solids that form by a regular repeated pattern of molecules connecting together. In some solids, the arrangements of the building blocks (atoms and molecules) can be random or very different throughout the material. In crystals, however, a collection of atoms called the Unit Cell is repeated in exactly the same arrangement over and over throughout the entire material. Because of this repetitive nature, crystals naturally take on strange and interesting looking forms.

Images clockwise from above: pharmaceutical crystals in close up(Anthony Maher), pharmaceutical crystals (SSPC), paracetamol crystals (SSPC)

Scientists have been interested in knowing and learning about crystals' inner structure and properties for numerous years. This branch of science is known as ***crystallography***, which has allowed us to study the chemical bonds that draw one atom to another. Crystal engineers can modify a crystal's inner structure, which then changes its properties and behaviour. Crystallisation is the process of forming (through natural or artificial growth and design) solid crystals.

A remarkable amount of materials around us, and indeed consumed by us in everyday life is made up of crystals. They are too small to be seen by the naked eye, but they are easily identifiable as crystals when viewed under a microscope. Examples of these include bones, shells, plastics, metals, paints, soils, minerals, sand and even, chocolate.

Crystals in real life - Geology

Crystals are at the heart of geology. Without crystals, there would be no rocks! Geological crystals come in two basic types:

- biogenic (from biological sources)
- abiogenic (non-biological)

Abiogenic crystals are produced by different Earth processes such as cooling of magma/lava, drying salty water, or chemical reactions when different materials mix. Crystals often form around a nucleation point, like how raindrops form around dust particles in clouds. The size of crystals produced is determined by how much time the crystal has to grow. For example, granite, which is formed from magma that slowly solidified underground has relatively large crystals. These are the white, grey, and black colours you see in a granite rock. This is in contrast with lava, which cools quickly at the surface, creating microscopic crystals. After formation, crystals can be put under additional stresses (temperatures and pressures) which can cause the atoms within the crystal to move around to form new crystals with a similar chemistry but different properties. This is exactly what happens when you squash the mineral graphite under high pressure to turn it into diamond.

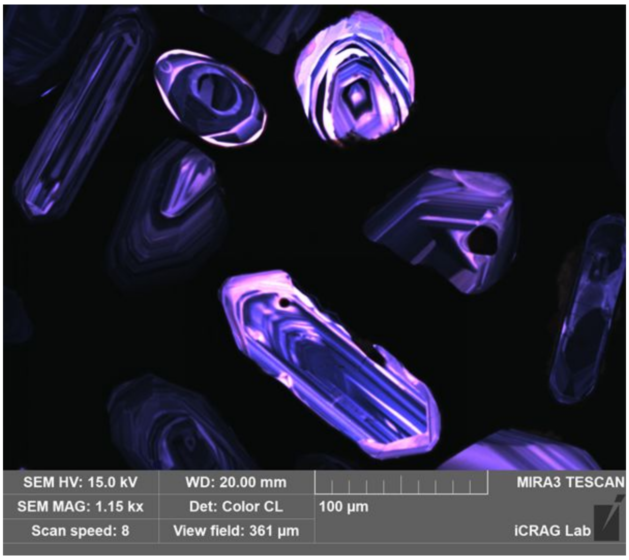
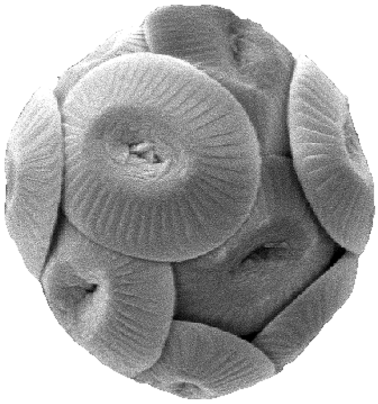


Image of zircon crystals photographed under an Electron Scanning Microscope. Zircon crystals are really interesting as geoscientists can use them to tell the age of rocks. Learn more about this in our resource booklet. Image courtesy of Martin Nauton-Forteu.

Biogenic minerals are those produced by an organism during its life, such as bones, which are primarily composed of calcium phosphate (also known as apatite), or shells, which are most often made of calcium carbonate (also known as calcite or aragonite). Calcium carbonate is such an important mineral that vast accumulations of it, such as coral reefs (coral make their bodies out of calcite), can have big impacts on global seawater chemistry. When these organisms die, their skeletons tend to fall apart and can form rocks, producing limestone. The evolution of mineralised skeletons in organisms may have resulted in the

Cambrian explosion (a time in history about 520 million years ago when modern animal groups first appeared in the fossil record). It is unclear why these skeletons first evolved but it could have been for protection, to aid movement, or as storage for important minerals needed for metabolic processes.



The image on the right shows a *Coccolithophore*. These are microscopic organisms that are made up of plates of Calcium Carbonate. *Coccolithophores* are the main constituent of chalk rock. They accumulated in such large numbers that hundreds of thousands of metres of chalk were made when their shells were compacted together after they died! A good example of this is the White Cliffs of Dover in the UK.

Image credit: https://www.ucl.ac.uk/GeolSci/micropal/calcnanno.html

Natural crystals, which are called minerals, can be identified by their crystallographic structure, their physical characteristics (colour, hardness, shape), and/or their optical properties (how light passes through them).

Crystals in real life - Medicine

Crystals are particularly important when making medicines. Most marketed pharmaceuticals consist of molecular crystals. The arrangement or packing of the molecules in a crystal determines its physical properties and, in certain cases, its chemical properties, and so greatly influences the processing and formulation of solid pharmaceuticals, as well as key drug properties such as dissolution rate (how long it takes the medicine to dissolve in the body) and its stability or indeed if it has any therapeutic effect at all.

In medicine manufacture, the crystal is the main part of the Active Pharmaceutical Ingredient (API), which needs to be ‘formulated’ or packed into a tablet that can be taken orally. Therefore, the shape and size of the crystals are very important as the crystalline material still needs to undergo the rest of the manufacturing process. There is so much involved in the manufacturing process, you want to make sure that your crystalline material remains stable and does not change form.

Other work involves co-crystallization; this involves growing one crystal, the API, on top of another ‘carrier’ crystal, which may improve its ability to be taken up into the bloodstream.

Terms to revisit/introduce:

Abiogenic: Something that is non-biological in origin
Biogenic: Something that is biological in origin
Crystal: A solid whose atoms are arranged in a highly ordered microscopic structure that extended in all directions.
Crystal Lattice: A crystal lattice can be thought of as an array of small boxes infinitely repeating in all three spatial directions.
Crystallography: The branch of science that deals with the study of crystals.
Dissolving: particles of one substance move into spaces between the particles of another substance.
Mineral: A mineral is a naturally occurring inorganic element or compound having an orderly internal structure and characteristic chemical composition, crystal form, and physical properties.
(https://www.usgs.gov/faqs/what-difference-between-a-rock-and-a-mineral)
Nucleation: the initial process that occurs in the formation of a crystal from a solution, a liquid, or a vapour, in which a small number of ions, atoms, or molecules become arranged in a pattern characteristic of a crystalline solid, forming a site upon which additional particles are deposited as the crystal grows.
(https://www.britannica.com/science/nucleation)
Nucleation point: An original point that crystals “stick” to and start their growth around.
Polymorph: The word ‘poly’ means many, and the word ‘morph’ means shapes.
Recrystallise: This means to form crystals again, so to re-dissolve and thus recrystallise crystals again. This is a purification technique.
Solubility: The solubility of a substance is the amount of that substance that will dissolve in a given amount of solvent. Solubility is a quantitative term. Solubilities vary enormously. The terms soluble and insoluble are relative. A substance is said to be soluble if more than 0.1 g of that substance dissolves in 100 mL solvent. If less than 0.1 g dissolves in 100 mL solvent, the substance is said to be insoluble or, more exactly, sparingly soluble.
Solute: the substance that is of lesser quantity and is being dissolved (moving into spaces)
Solvent: the substance in greater quantity (the one with the spaces being filled)
Supersaturated: A solution that contains more of the dissolved material than could be dissolved by the solvent under normal circumstances. For example, you can dissolve more sugar in hot water than room temperature water, the abnormal circumstance is the temperature of the water.
Unit cell: This is the smallest unit of volume that contains all of the structural and symmetry information to build-up the macroscopic structure of the lattice by translation.

Misconceptions:

- all matter is made of discrete particles.
- particles are in constant random motion.
- the space between particles is empty.
- ‘bonds’ or forces exist between particles.
- Atoms actually touch each other.
- When a substance dissolves it disappears

An explanation for these misconceptions:

The invisibility of particles to the naked eye means our minds “see” materials as continuous. Even scientists themselves did not understand about particles until quite recently – they had been at work for nearly 2000 years before the idea of atoms was accepted in the early 19th century!

Particles are tiny! They are smaller than insects, and many bacteria and viruses! If you have access to a microscope, try crushing up a paracetamol tablet and looking at this under the microscope. You should be able to see the individual crystals in the crushed up powder - this is the active paracetamol, the bit that makes you feel better when you take the tablet.

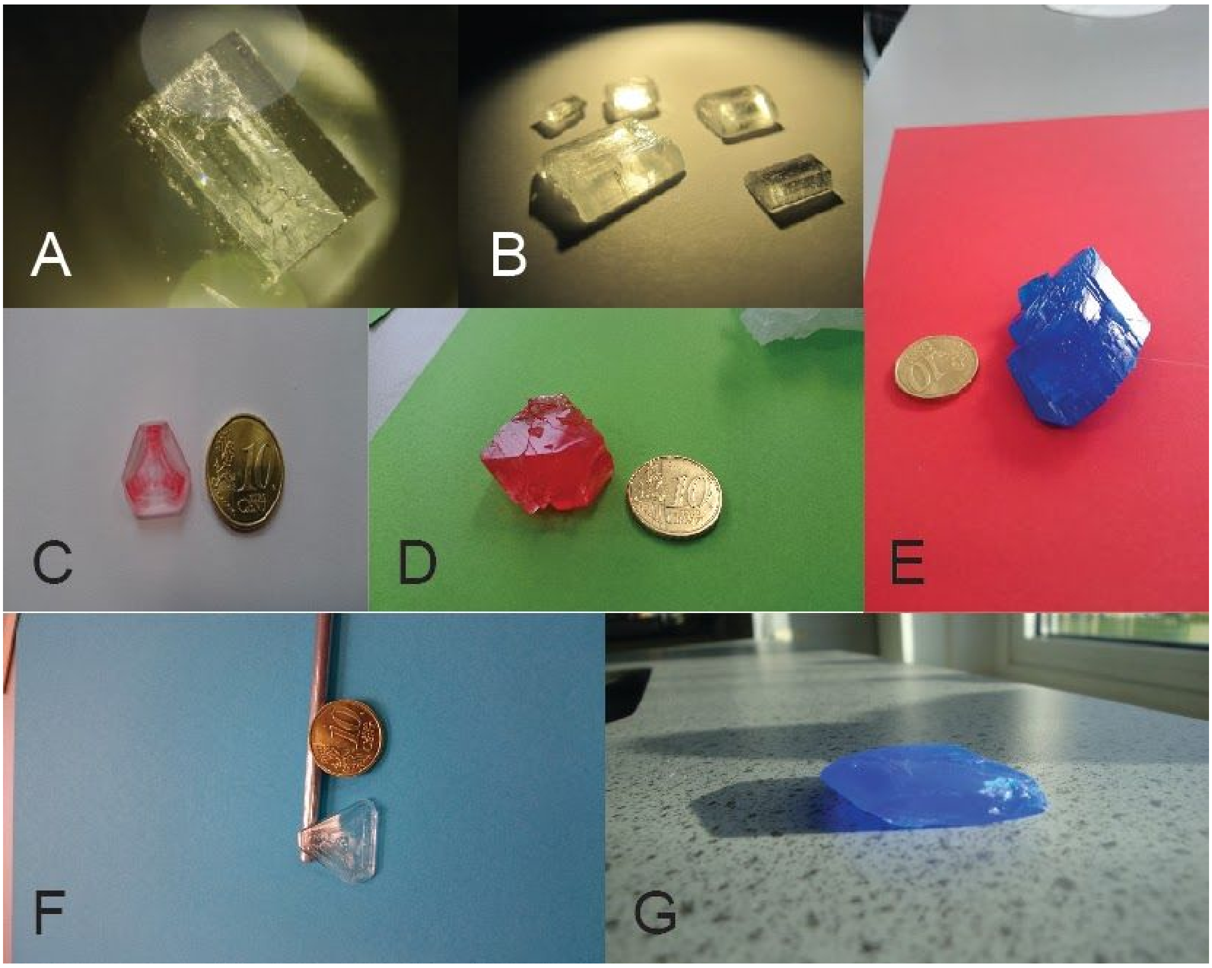
Note for teachers: If you are teaching particle theory, you can integrate this into other topics; it doesn’t have to be treated in isolation. Use the terminology when discussing chemical reactions, changes of state and introduce simple symbolic equations as soon as possible, rather than the word equations which emphasise the bulk materials. Also, Pupils may think that atoms are touching due to the way we represent these structures. It is important when using molecular representation to stress that it is a model for how we imagine the structures to be.

How to grow a crystal

N.B. This procedure has been adapted from:
https://www.iycr2014.org/__data/assets/pdf_file/0011/85457/CG_brochure.pdf

Introduction:

Growing a beautiful crystal takes time and an almost daily follow-up. The idea is to grow a single crystal, not a bunch of crystals. You will first need to grow a small perfect crystal, your seed crystal, around which you will later grow a large crystal. It is therefore essential to avoid excessive rapid growth, which encourages the formation of multiple crystals instead of a single crystal.



Images: A&B: by Youbeel Hagi, C: Alum crystal with red food colouring by Jason Folan, D: Alum crystal with red colouring by Jason Folan, E: Copper Sulphate crystal, F: Alum crystal by Oisín Tobin, G: Copper sulphate crystal by Ross Moloney

How to grow your crystal 1: you will need

Apparatus

- A small wood rod, popsicle or sate stick
- A shallow dish (*e.g.* Petri dish)
- Thermometer
- Balance
- Plastic or glass container
- Heating plate
- Beaker of 2 to 4 litres volume
- Fishing line (1 to 2 kg strength)/ fine strong thread/fine strong string
- Styrofoam box or picnic cooler
- A magnifying glass

Chemicals:

- Substance to be crystallized (see compounds to crystallise on page 15)
- Distilled or demineralized water
- Superglue

How to grow your crystal 2: procedure

Stage 1: Grow a seed crystal

1. Warm about 50 mL of water in a glass container.
2. Dissolve a quantity of the substance to produce a saturated solution at the elevated temperature.
3. Pour the warm solution into a shallow dish.
4. Allow the solution to cool to room temperature.
5. After a day or so, small crystals should begin to form as in Figure 1.
6. Remove some of the crystals.
7. With a magnifying glass select a beautiful and transparent small crystal. This will be your seed crystal. Weigh the crystal.

Figure 1. Seed crystals of alum. image: alum seeds cup (Credit: Cohise College)

Stage 2: Grow a large Single crystal

1. Glue the seed crystal at the end of a piece of fishing line/thread by using superglue (be careful not to glue your fingers together!).
2. Check with the magnifier that the seed crystal is well-fixed to the line.
3. To grow your large, single crystal, you will need a supersaturated solution.

*We all know that sugar dissolves in water. At 40°C 250g of sugar will dissolve in 100mL of water. This value is called the **SATURATION Solubility** of sugar in water at 40 °C. If you add more sugar at this temperature is will not dissolve, but remain in suspension, even with vigorous stirring.*

BUT, how do you get sugar to crystallise or recrystallize?

The trick is to quickly cool the saturated solution. Suppose we cool it quickly to 15° C. At this temperature the water is only able to dissolve 200g of sugar in 100mL of water. However, the extra dissolved sugar, in this case 50g per 100mL, remains dissolved for a short time, and during this time we have a supersaturated solution. The “extra” sugar (50g per 100mL) crystallises out of solution and the sugar-water mixture returns to its saturated state. This technique produces a large number of small crystals.

- *A **saturated solution** is one in which no more solute can be dissolved in the solvent. For example if you add sugar to water until no more will dissolve you then have a saturated solution*
- *So then **a supersaturated solution** is one where there are more particles or solutes than solvent in the solution.*

So how can we make one of these?

- *If we add sugar to water until no more can dissolve we will have a saturated solution, to make this solution supersaturated we can heat the solution to a certain temperature and then continue to add sugar.*
- *This is because as we increase the temperature in this case it will allow more sugar to dissolve, thus making our solution supersaturated.*
- *The amounts of substance and water to be used will depend upon the solubility at room and elevated temperatures. You may have to determine the proper proportions by trial and error (just like the first scientists did!).*

Procedure for supersaturated solution:

1. Place about double the amount of substance that would normally dissolve in a certain volume of water at room temperature into that volume of water. (e.g. If 30 g of X dissolves in 100 mL of water at room temperature, place 60 g of X in 100 mL of water.) Adjust the proportions depending upon how much material you have. Use clean glassware.
2. Stir the mixture until it appears that no more will go into solution.

3. Continue stirring the mixture while gently warming the solution.
4. Once all of the substance has gone into solution, remove the container from the heat.
5. Allow the solution to cool to room temperature.
6. You now have a supersaturated solution.

An alternative method to create a supersaturated solution is:

1. To start with a saturated solution and let the solvent evaporate. **This will be a slower process.**

A third method to create a supersaturated solution:

1. Select an appropriate volume of water.
2. Warm this water to about 15–20° C above room temperature.
3. Add some of your substance to the warm water and stir the mixture to dissolve completely.
4. Continue adding substance and stirring until there is a little material that won’t dissolve.
5. Warm the mixture a bit more until the remaining material goes into solution.
6. Once all of the substance has gone into solution, remove the container from the heat.
7. Allow the solution to cool to room temperature.
8. You now have a supersaturated solution.

Now you are ready to grow a large single crystal starting from your seed crystal.

9. Carefully suspend your seed crystal from the stick into the cold supersaturated solution in the middle of the container with supersaturated solution **(Figure 2).**
10. Cover the container in which the crystal is growing with plastic wrap, aluminium foil or a piece of cardboard in order to keep out dust, and reduce temperature fluctuations.

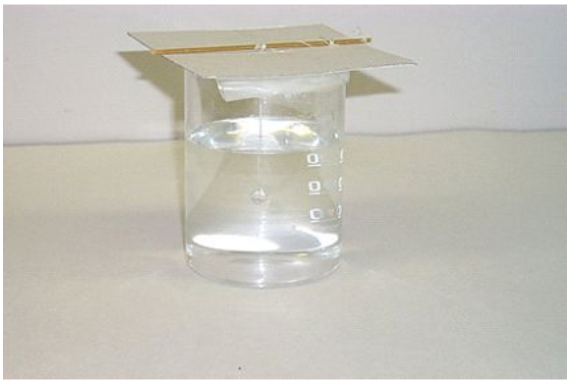


Figure 2. Seed crystal of alum suspended in saturated solution. (Credit: picture by Luc Van Meervelt)

The solubility of some salts is quite sensitive to temperature, so the temperature of recrystallization should be controlled as best you can. It is possible that you have a nice big crystal growing in a beaker on a Friday, the room temperature rising in a school over the weekend, and by Monday morning the crystal had totally gone back into solution.
So it is a very good idea to place your growing crystal inside a Styrofoam box (**Figure 3)** or picnic cooler!

Figure 3. Styrofoam or isomo box.

IMAGE: #18028934 Open Styrofoam Storage Box on White..

1. Observe the crystal growth. Depending upon the substance, the degree of supersaturation and the temperature, this may take several days before the growth slows down and stops.
2. Re-supersaturate the solution. This may need to be done on a daily basis, especially when the crystal gets larger. But first, remove the crystal.
3. Determine the weight of the crystal and compare it to the previous weight. Make your solution again supersaturated by adding the amount the crystal grew.
4. Warm and stir the solution until everything is gone into solution.
5. Cool the solution to room temperature!
6. Each time the solution is saturated, it is a good idea to ‘clean’ the monocrystal surface, by
    - making sure the crystal is dry;
    - not touching the crystal with your fingers (hold only by the suspending line if possible);
    - removing any ‘bumps’ on the surface due to extra growth;
    - Removing any small crystals from the line.
    - It is a good habit to clean your hands after each manipulation.
7. Re-suspend the crystal back into the newly supersaturated solution.
8. Repeat the previous steps as needed.

Worked Example: Copper Sulphate Crystals

Introduction:

Copper(II) sulphate, also known as cupric sulphate or copper sulphate, is the chemical compound with the chemical formula $CuSO_4$. This salt exists as a series of compounds that differ in their degree of hydration. The anhydrous form is a pale green or grey-white powder, whereas the pentahydrate ($CuSO_4 \cdot 5H_2O$), the most commonly encountered salt, is bright blue. Copper(II) sulphate exothermically dissolves in water to give the aquo complex $[Cu(H_2O)_6]^{2+}$, which has octahedral molecular geometry and is paramagnetic. Other names for copper(II) sulphate are "blue vitriol" and "bluestone".

A crystal is a solid material whose constituent atoms, molecules, or ions are arranged in an orderly repeating pattern extending in all three spatial dimensions. Crystal growth is a major stage of a crystallization process, and consists in the addition of new atoms, ions, or polymer strings into the characteristic arrangement of a crystalline Bravais lattice. The growth typically follows an initial stage of either homogeneous or heterogeneous (surface catalysed) nucleation, unless a "seed" crystal, purposely added to start the growth, was already present.

The action of crystal growth yields a crystalline solid whose atoms or molecules are typically close packed, with fixed positions in space relative to each other. The crystalline state of matter is characterized by a distinct structural rigidity and virtual resistance to deformation (i.e. changes of shape and/or volume). Most crystalline solids have high values both of Young's modulus and of the shear modulus of elasticity. This contrasts with most liquids or fluids, which have a low shear modulus, and typically exhibit the capacity for macroscopic viscous flow.

HEALTH & SAFETY: Copper sulphate is harmful if swallowed and can irritate skin and mucous membranes. In case of contact, rinse skin with water. If swallowed, give water and call a physician

Chemicals:

- copper sulphate
- water

Method:

Make a Saturated Copper Sulphate Solution

1. Stir copper sulphate into very hot water until no more will dissolve.
2. Pour the solution into a jar and wait a few days for crystals to grow
3. However, if you grow a seed crystal, you can get much larger and better-shaped crystals.

Grow a Seed Crystal

1. Pour a little of the saturated copper sulphate solution into a saucer or shallow dish.
2. Allow it to sit in an undisturbed location for several hours or overnight.
3. Select the best crystal as your 'seed' for growing a large crystal.
4. Scrape the crystal off of the container and tie it to a length of thin string (or dental floss)

Growing a Large Crystal

1. Suspend the seed crystal in a clean jar that you have filled with the solution you made earlier (the saturated copper sulphate solution).
2. Do not allow any undissolved copper sulphate to spill into the jar.
3. Do not let the seed crystal touch the sides or bottom of the jar.
4. Place the jar in a location where it won't be disturbed. You can set a coffee filter or some kitchen roll over the top of the container, but allow air circulation so that the liquid can evaporate.
5. Check the growth of your crystal each day. If you see crystals starting to grow on the bottom, sides, or top of the container then remove the seed crystal and suspend it in a clean jar. Pour the solution into this jar. You don't want 'extra' crystals growing because they will compete with your crystal and will slow its growth.
6. When you are happy with the size of the crystal, you can remove it from the solution and allow it to dry.

Tips & Safety

- Even a small increase in the temperature of the water will greatly affect the amount of copper sulphate ($CuS0_4 . 5H_20$) that will dissolve.
- Copper sulphate pentahydrate crystals contain water, so if you want to store your finished crystal, keep it in a sealed container. Otherwise water will evaporate from the crystals, leaving them dull and powdery. The grey or greenish powder is the anhydrous form of copper sulphate.
- Copper sulphate is used in copper plating, blood tests for anaemia, in algaecides and fungicides, in textile manufacturing, and as a desiccant.

Compounds to Crystallise

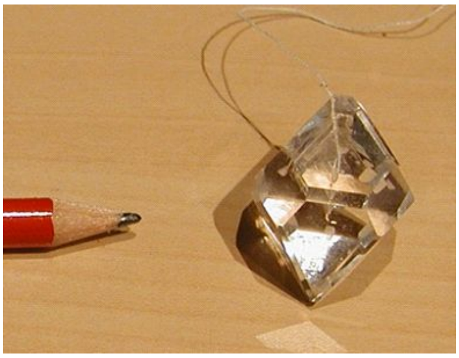
N.B. This section has been adapted from:
https://www.iycr2014.org/__data/assets/pdf_file/0011/85457/CG_brochure.pdf

The compounds listed below are safe to grow at home and can be made with ingredients readily available at chemists or in hardware shops. Other compounds require teacher supervision to crystallise. The recipes for these are listed in the pdf linked above.

1. **Potassium Aluminium Sulfate**

Also known as "Alum" or "Potassium Alum"
Chemical formula: $KAl(SO_4)_2 \cdot 12H_2O$
Crystals: Transparent, octahedral
Solubility: 118 g/L (20°C, water)

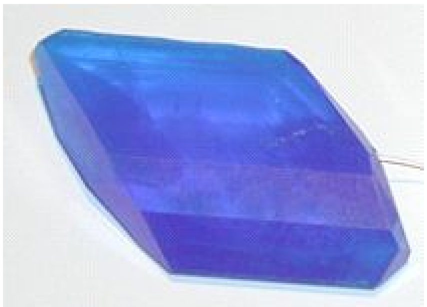
Credit: picture by Luc Van Meervelt



2. **Copper(II) Sulfate Pentahydrate**

N.B. This compound is suitable for post primary level students only.
Chemical formula: $CuSO_4 \cdot 5H_2O$
Crystals: Blue
Solubility: 390 g/L
(anhydrous, 20°C, water).

Credit: picture by Luc Van Meervelt



3. **Ammonium Magnesium Sulfate Hexahydrate (or Epsom salts if you do not have access to ammonium sulfate and magnesium sulphate)**

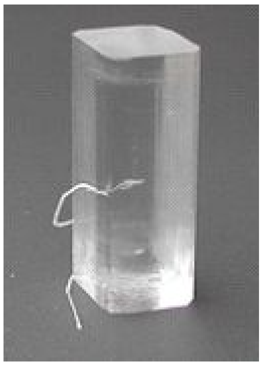
Chemical formula: $(NH_4)_2Mg(SO_4)_2 \cdot 6H_2O$
Crystals: Transparent, long

To make this crystal, you will need:

- Ammonium sulfate and magnesium sulfate

- Disolve equal amounts of both salts in water (e.g. 0.4 mol of both compounds in 45-48 mL of water)
- Stir until everything is dissolved
- Add small amounts (2-5 grams) of both salts to the solution under a gentle heat (max 30-40°C). This will make your solution supersaturated.
- Cover the beaker and allow to cool at room temperature
- Proceed with crystal growth as detailed in the 'How to grow a crystal' section

Credit: picture by Luc Van Meervelt



4. **Potassium Sodium Tartrate**

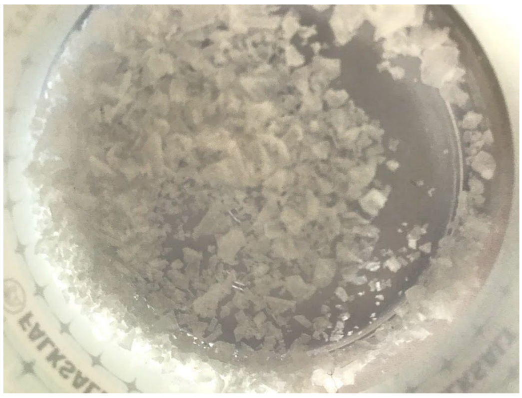
Also know as "Seignette salt" or "Rochelle salt",

Chemical formula: KNaC4H4O6.4H2O

Crystals: Transparent, long

Solubility: 630 g/L (anhydrous, 20°C,water).

Credit: picture by Luc Van Meervelt



5. **Sodium Chloride**

Chemical Formula: NaCl. Also known as table salt.

Solubility: 35.9 g/100 mL (20°C, water)

Crystals: Normally small, not well formed (because the solubility barely changes as a function of temperature)

At 20°C, one can dissolve 35.9g of NaCl in 100g of water, and at 100°C, just 39.2g per 100g of water. The easiest way to grow sodium chloride crystals is by evaporation of a saturated solution. Small (sub-millimetre) clear cubes with smooth faces will grow on the bottom of your glass dish or jar, or on any thread suspended in the jar. Larger crystals tend to develop hopper faces, or even more erratic growth habits. One interesting experiment to try is to see how the growth morphology changes if you add small quantities of other substances - a smidge of copper sulfate perhaps, K-alum, or sodium nitrate - to your saturated salt solution.

6. **Sucrose**

Chemical Formula: C12H22O11. Also known as saccharose or table sugar.

Solubility: 211.5g/100 mL (20°C, water).

Cane sugar produces great crystals without too much trouble, provided you can be patient. To make this crystal, you should:

- Dissolve ~500 grams of sugar per 100 mL of hot water, and leave to cool.
- When supersaturated, the solution will appear pale/silvery-yellow and will be very viscous
- It can take anywhere from a week to over a month to start producing crystals, depending on how big a container you're using.
- Crystals shoot from smaller volumes of liquid quite quickly and can grow to a length of a few millimeters.

Use these as seeds to grow much larger crystals. You can grow very pretty single crystals over a period of just a few weeks. Sucrose is slightly deliquescent; in other words, the crystals 'sweat' a bit. You'll find the crystals become slightly moist and sticky, even when you've dried them, particularly in a warm room.

7. **Fructose**

Chemical Formula: $C_6H_{12}O_6$. Also known as fruit sugar.
Solubility: 3750 g/L (20°C, water).

Solutions are made up in the same way as for sucrose. As with sucrose, patience is everything. An 80 wt. % fructose solution, if unseeded, may take several weeks to start crystallising at room temperature; the first sign is the appearance of little flat squares floating on the surface of the solution. If you instead seed the solution with some of the tiny crystals from your original box, then white 'blotches' - like cotton wool - appear in the liquid. These might be mistaken for bits of mould; in fact they are aggregates of very very fine hair-like crystals of fructose hemihydrate ($C_6H_{12}O_6 \cdot 1/2H_2O$). The hemihydrate will also appear if you seed a concentrated solution kept in the fridge (at 1 - 4°C). Vigorous stirring of the solution breaks up the hemihydrate crystals and causes crystals of fructose dihydrate ($C_6H_{12}O_6 \cdot 2H_2O$) to nucleate.
credit: https://www.sciencephoto.com/media/95969/view/fructose-crystals-sem.

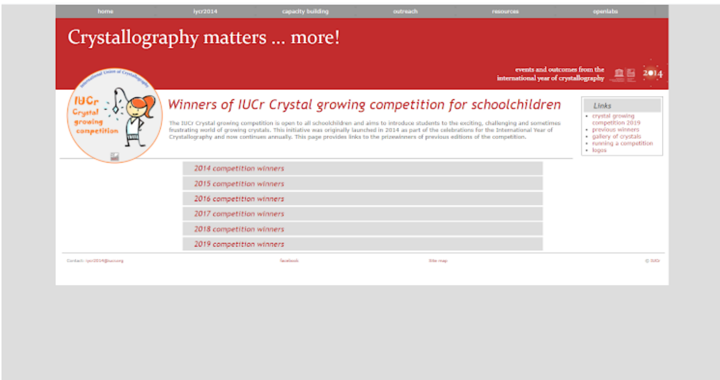
Further Resources

Here is a list of websites that may be useful to students and teachers taking part in the crystal growing competition.

1. https://www.xtal.iqfr.csic.es/Cristalografia/index-en.html

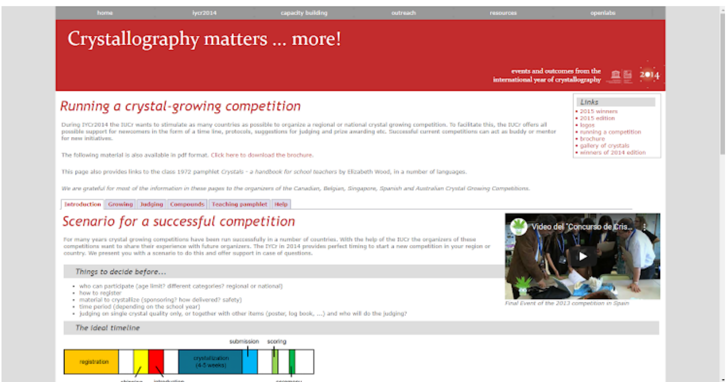
A fascinating website about all things crystals! Suggested target age: Second Level or advanced upper Primary Level students.

2. https://www.iycr2014.org/participate/competition-winners



Learn information about what past entrants and winners of the Crystal Growing Competition have done. Suggested target age: any

3. https://www.iycr2014.org/participate/crystal-growing-competition-2015/info-for-newcomers



Useful information for new participants of the Crystal Growing Competition

Frequently asked questions

Why does the crystal stop growing?

A crystal will only grow when the surrounding solution is supersaturated with solute. When the solution is completely saturated, no more material will be deposited on the crystal. (This may not be entirely true. Some may be deposited; however an equal amount will leave the crystal surface to go back into solution. We call this an equilibrium condition.)

Why did my crystal shrink/disappear?

If your crystal shrank or disappeared, it was because the surrounding solution became under saturated and the crystal material went back into solution. Under saturation may occur when the temperature of a saturated solution increases, even by only a few degrees, depending upon the solute. (This is why temperature control is so important.)

How do I get crystal growth restarted?

Make the solution supersaturated again!

Help, my crystal has lost its transparency!

When removing the crystal from the solution, clean it very quickly in water to rinse the thin layer of solution on the crystal surface away. Otherwise this thin layer would leave an amorphous (non-crystalline) precipitate on the surface after evaporation. This will decrease the transparency of the crystal, and you will not be able to harvest a perfect transparent crystal as in Figure 4.

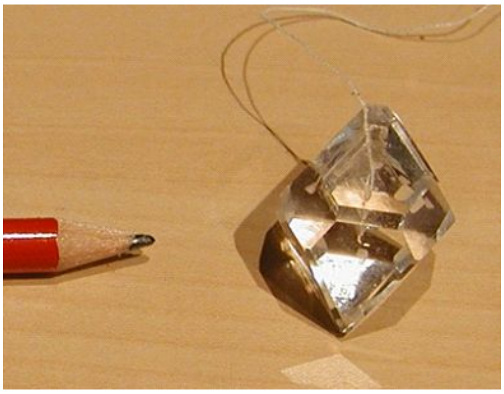


Figure 4. Transparent alum crystal. (Credit: picture by Luc Van Meervelt)

Image: Alum crystal transparent(Credit:www.inriodulce.com/links/alum)

What is the difference between an under saturated, saturated and supersaturated solution?

In recrystallization, one tries to prepare a solution that is supersaturated with respect to the solute (the material you want to crystallize). There are several ways to do this.

One is to heat the solvent, dissolve as much solute as you can (said to be a "saturated" solution at that temperature), and then let it cool. At this point, all the solute remains in solution, which now contains more solute at that temperature than it normally would (and is said to be "supersaturated").
This situation is somewhat unstable. If you now suspend a solid material in the solution, the "extra" solute will tend to come out of solution and grow around the solid. Particles of dust can cause this to occur. However, this growth will be uncontrolled and should be avoided (thus the recrystallization beaker should be covered). To get controlled growth, a "seed crystal", prepared from the solute should be suspended into the solution (Figure 5).

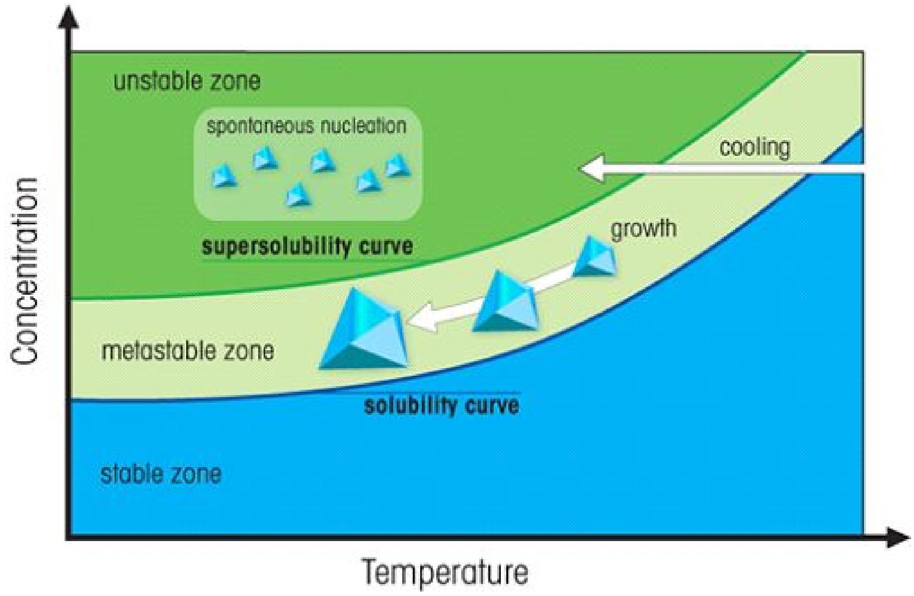


Figure 5. The region above the solubility curve is called "supersaturated". In the unstable zone (green) spontaneous nucleation occurs. A crystal suspended in the metastable zone will grow further.

The supersaturation method works when the solute is more soluble in hot solvent than cold. This is usually the case, but there are exceptions. For example, the solubility of table salt (sodium chloride) is about the same whether the water is hot or cold.

I am a perfectionist; can I do anything else?

To get improved symmetry and size, slowly rotate the growing monocrystal (1 to 4 rotations per day). An electric motor with 1 to 4 daily rotations might be difficult to find (consider one from an old humidity drum-register or other apparatus). This option becomes useful only when a monocrystal gets rather big. You can also place the beaker into a thermostated bath set to a few degrees above room temperature.

Slow or fast growing, what is the best?

The rate at which crystallization occurs will affect crystal quality. The more supersaturated a solution is, the faster growth may be. Usually, the best crystals are the ones that grow slowly.

What is the effect of impurities?
Once you have mastered crystal growth, you may be interested in trying to grow single crystals in the presence of introduced ‘impurities'. These impurities may give different crystal colours or shapes.

Does this method also work for proteins?
No, it is not possible to make a supersaturated protein solution by dissolving protein into a hot solvent. The protein will denature and lose its regular folded structure. A special set-up is needed here. In the hanging drop method for example, a droplet containing protein, buffer and precipitant is hanging above a larger reservoir containing buffer and precipitant in a higher concentration. As water evaporates from the droplet it will transfer to the reservoir where it is bound to the precipitant. During this process the protein is concentrated. Once supersaturation is reached, nucleation and crystal growth is starting. Growing crystals is a long process, and takes a number of days/weeks. This activity can be turned into a project over the course of this module, to be re-visited towards the end of the module. When re-visiting the crystals that your class has grown, have pupils compare their newly grown crystals with the chart below to determine what type of crystals they have grown.

About

SPCC

SSPC is the SFI Research Centre for Pharmaceuticals. SSPC is a world-leading hub of Irish research expertise developing innovative technologies to address key challenges facing the pharmaceutical and biopharmaceutical industry.

Our unique and innovative culture of collaboration is helping the global pharmaceutical and biopharmaceutical industry to develop more environmentally sustainable methods for drug manufacturing; to increase the range of medicines available to the public and to reduce drug manufacturing costs. SSPC's advanced research programme extends from **Molecule**, **Materials** and **Medicine** into the **Manufacturing** and **Modelling** space. Our research addresses four significant challenges faced by the pharmaceutical sector:

- Reducing time to market in drug development
  Advanced manufacturing processes
- Improved Efficacy of Drug Products
- Addressing the needs of new more complex active ingredients

SSPC, the world leading SFI Research Centre in pharmaceuticals is hosted by the University of Limerick (UL). The SSPC comprises approximately 150 researchers across eight universities and institutions. SSPC is supported by Science Foundation Ireland, the European Regional Development Fund, and industry partners.

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About iCRAG

iCRAG is the SFI Research Centre in Applied Geosciences. We are a team of researchers creating solutions for a sustainable society. We develop innovative science and technologies to better understand the Earth's past, present, and future and how people are connected to it. We drive research in areas that are critical to society and the economy, including:

- Sustainable discovery of energy resources and raw materials required for decarbonisation.
- Securing and protecting groundwater and marine resources.
- Protecting society from Earth's hazards such as flood and landslides.

iCRAG, the world leading SFI Research Centre in applied geosciences hosted by UCD, comprises 150 researchers across eight universities and institutions including NUI Galway. iCRAG is supported by Science Foundation Ireland, the European Regional Development Fund, Geological Survey Ireland and industry partners.

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IUCr

The IUCr is a scientific union adhering to the International Science Council (ISC). Its objectives are to promote international cooperation in crystallography and to contribute to all aspects of crystallography, to promote international publication of crystallographic research, to facilitate standardization of methods, units, nomenclatures and symbols, and to form a focus for the relations of crystallography to other sciences.