Molecular Structure Laboratory
Chemistry Department
University of Wisconsin-Madison

The 2017 Wisconsin Crystal Growing Competition

Handbook

February 6, 2017

http://xray.chem.wisc.edu/WICGC_2017.html

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Definition

The Molecular Structure Laboratory of the UW-Madison Chemistry Department has launched the 4th Wisconsin Crystal Growing Competition among Wisconsin **middle** and **high** school students to be held March-May 2017. The contest is **FREE** to all participants. This is an exciting scientific competition as well as a fun, hands-on lab experience. All participants will be provided the appropriate crystallization material free of charge (it is the intension of the organizers).

There are **four** separate contests:

1. **Middle school** students and home-schooled youths (ages 11-13) will grow crystals of **cupric sulfate** ($CuSO_4.5H_2O$, copper (II) sulfate pentahydrate, or 'bluestone').
2. **High school** students home-schooled youths (ages 14-18) will grow crystals of **potassium dihydrogen phosphate** (KH_2PO_4; KDP).
3. **Middle school teachers** will grow crystals of **cupric sulfate** ($CuSO_4.5H_2O$, copper (II) sulfate pentahydrate, or 'bluestone').
4. **High school teachers** will grow crystals of **potassium dihydrogen phosphate** (KH_2PO_4; KDP).

Instructions on how to grow crystals are provided on the web. The objective is *to grow the biggest and highest quality single crystal*. The contest is described in this Handbook.

Wisconsin prizes

The crystal growing period is from March 1 to April 30, 2017. The judging will take place on the 12th May 2017 by a panel of judges. The crystals will be judged in three categories:

1. Best Overall Crystal – 1st prize ($200), 2nd prize ($100), 3rd prize ($50)
2. Best Quality Crystal – 1st prize ($200)
3. Best Teacher's Crystal – 1st prize ($100)
4. Best crystal-inspired art (details on the "Crystal art judging" tab) - 1st prize ($100), 2nd prize ($50) and 3rd prize ($50).

There are two sets of identical prizes – one for the middle school contest and the other for the high school contest.

Students win cash awards for their school and will receive books and individual certificates as personal prizes.

All participants will be invited to a reception at University of Wisconsin-Madison and to receive prizes at the end of May 2017. The exact date is to be determined. There will be a special lecture for the audience and departmental tours.

In addition, the best crystals will become part of a permanent display at the Molecular Structure Laboratory at UW-Madison.

Who can participate?

- All Wisconsin middle and high school students or homeschooled youths ages 11-18.
- Individuals or teams of up to three students. There is no limit on the number of teams per school.
- Middle and high school science teachers.

The entries will be categorized in four different divisions as described below. Each division will be judged separately.

- Division 1: Wisconsin middle school students and home-schooled youths ages 11-13.
- Division 2: Wisconsin high school students and home-schooled youths (ages 14-18.
- Division 3: Wisconsin middle school teachers.
- Division 4: Wisconsin high school students.

How do I sign up?

1. Read the How to Grow Crystals section and decide that you are up to the task.
2. Register for the contest by filling out an on-line registration form (under the Who can participate tab at http://xray.chem.wisc.edu/WICGC_2017.html). Please state your full name, age, your science teacher's full name and contact information, and school name and address.
3. Grow crystals.
4. Fill out the official Entry form and mail it along with your crystals to the address below.

All forms, requests, questions, and crystals should be sent to

Ilia A. Guzei, Ph.D.
2124 Chemistry Department
University of Wisconsin-Madison
1101 University Ave
Madison, WI 53706, USA
Phone: 608-263-4694
iguzei@chem.wisc.edu (subject line: Wisconsin Crystal Growing Competition).

What is the crystallization material and how to get it?

Middle schools

For 2017 the material for the crystal growing competition is cupric sulfate, CuSO4.5H2O (copper (II) sulfate pentahydrate, or 'bluestone'). This material is chosen because it produces large beautiful blue crystals, which are neither too easy nor too difficult to grow. Cupric sulfate was also the compound used in *the very first X-ray diffraction experiment by Max von Laue in 1912*.

The organizers plan to provide 100 g of cupric sulfate free of charge to each participating team, which is enough for one crystallization. Should your team decide to set up more than one crystallization you are welcome to do so. Additional material can be ordered from Flinn Scientific (http://www.flinnsci.com/store/Scripts/prodView.asp?idproduct=17896). Many hardware stores carry cupric sulfate as a common pesticide.

If you have questions about cupric sulfate - feel free to contact Dr. Ilia Guzei by mail or e-mail.

High schools

For 2017 the material for the crystal growing competition is potassium dihydrogen phosphate (KH_2PO_4; KDP). This material is chosen because it is safe and produces gorgeous crystals.

The organizers plan to provide 100 g of KDP free of charge to each participating team, which is enough for one crystallization. Should your team decide to set up more than one crystallization you are welcome to do so. Additional material can be ordered from Flinn Scientific (Potassium Phosphate, Monobasic: http://www.flinnsci.com/store/Scripts/prodView.asp?idproduct=20675).

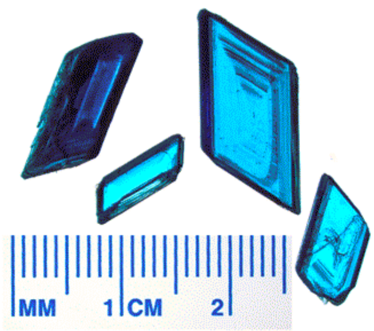
If you have questions about potassium dihydrogen phosphate - feel free to contact Dr. Ilia Guzei by mail or e-mail.

Safety

Both copper (II) sulfate pentahydrate and potassium dihydrogen phosphate are relatively safe, but the usual safety precautions should be exercised. Gloves and goggles are recommended.

The KDP and Cupric Sulfate Material Safety Data Sheet are available at the Sigma Aldrich (one of our sponsors) and at Flinn Scientific (see links above).

What is a crystal?



A crystal is a solid that consists of various atoms, ions, or molecules arranged in a uniform three-dimensional repeating pattern. This results in the material having a specific shape and color, and having other characteristic properties. Diamond (used in jewelry and cutting tools) is an example of a crystal. It is made of pure carbon. Salt and sugar are also examples of crystals.

Recrystallization is a process that has been used to purify solid material by dissolving the solid (called a solute) in an appropriate liquid (called a solvent) and then having the material precipitate out of solution in crystalline form. Depending upon conditions, one may obtain a mass of many small crystals or one large crystal.

Crystals are characterized by type, shape, form, clarity, and color.

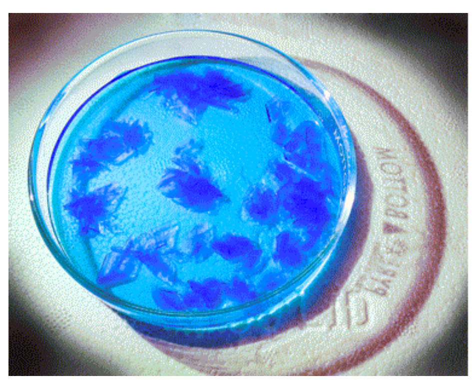
How to grow crystals

The crystal growing period is from March 1 to April 30, 2017. The judging will take place in May 2017 by a panel of judges.

RULE 1: The maximum amount of starting material that may be used for each given crystal is limited to 100 g.
RULE 2: So that all students have an equal preparation time, crystal production must conclude within six weeks after receipt of starting material.

First Stage: Grow a Seed Crystal

The idea is to grow a single crystal, not a bunch of crystals. You will first need to grow a small perfect crystal that will become your seed crystal, around which you will later grow a large crystal. It is therefore essential to avoid excessively rapid growth, which encourages the formation of multiple crystals instead of a single crystal.



What You Need

☐ Substance to be crystallized;
☐ Distilled or demineralised water;
☐ A shallow dish (e.g., Petri);
☐ Heating plate or stove;
☐ Fishing line (1 to 2 kg strength);
☐ A small wood rod (e.g., popsicle stick);
☐ A magnifying glass (optional).

Important Things to Know

☐ How much substance you have to work with, which you can determine by weighing it on a balance.
☐ The solubility of the substance in water at room temperature, which you can obtain from a chemistry reference book.
☐ It would also be useful to know the solubility of the substance at elevated temperatures, which is information that may also be available in a reference book such as Handbook of Chemistry and Physics, 45th Ed (1964-5).

What to Do

☐ Warm about 50 mL (1/4 cup) of water in a glass container.
☐ Dissolve a quantity of the substance to produce a saturated solution at the elevated temperature.
☐ Pour the warm solution into a shallow dish.
☐ Allow the solution to cool to room temperature.
☐ After a day or so, small crystals should begin to form.
☐ Remove some of the crystals.
☐ With a magnifier select a beautiful and transparent small crystal. This will be your seed crystal.
☐ Tie the seed crystal with the fishing line by using a simple overhand knot.
☐ Suspend the seed crystal in a shallow (1 to 2 mm deep) small volume (about 1 to 2 mL) saturated solution (for example, in a cover or a Petri dish) for some time (1 to 2 days).
☐ Check with the magnifier that the seedling crystal is well-fixed to the line by its beginning growth. This step is very important because one can lose several days of growth if the 'beginning growth' is not regular or not along the structure of the seedling crystal. It is worth checking properly before going on with the regular crystal growth.

Second Stage: Grow a Large, Single Crystal

Now you are ready to proceed with the preparation of a large single crystal.

Once you have mastered this step, you may be interested in trying to grow single crystals in the presence of introduced 'impurities" that may give different crystal colors or shapes. 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. 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. 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.

Thus, if you heated the solvent to near the boiling point to get a highly supersaturated solution on cooling back to room temperature, crystals may start to form before the solution had completely cooled.
This is where the "art" of science comes into play. One has to experiment a bit to get the right conditions.
A second way to get supersaturation is to start with a saturated solution and let the solvent evaporate.
This will be a slower process.

The above will apply to most situations. It is necessary to match the proper solvent with a given solute.

WARNING: the solubility of some salts is quite sensitive to temperature, so the temperature of recrystallization should be controlled as best you can. There have been reports in the past of students having 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. Consider insulating your crystallization vessel inside a Styrofoam box.

What You Need

☐ Substance to be crystallized;
☐ A seed crystal of the substance to be crystallized on a fishing line;
☐ Distilled or demineralized water;

☐ A small wood rod or popsicle stick;
☐ Thermometer;
☐ Balance;
☐ Plastic or glass container;
☐ Heating plate;
☐ Beaker of 2 to 4 liters volume;
☐ Thermostated bath (optional);
☐ Slow revolution motor (1 to 4 rotations per day) (optional).

Important Things to Know

☐ How much substance you have to work with, which you can determine by weighing it on a balance.
☐ The solubility of the substance in water at room temperature, which you can obtain from a chemistry reference book.
☐ It would also be useful to know the solubility of the substance at elevated temperatures, which is information that may also be available in a reference book.

How to Prepare a Supersaturated Solution

To grow your large, single crystal, you will need a supersaturated solution.
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).

Method One

☐ 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 (about 1 oz) of X dissolves in 100 g (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.
☐ Stir the mixture until it appears that no more will go into solution.
☐ Continue stirring the mixture while gently warming the solution.
☐ Once all of the substance has gone into solution, remove the container from the heat.
☐ Allow the solution to cool to room temperature.
☐ You now have a supersaturated solution.

Method two

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

Now you can grow your wonderful crystal

Since the solubility of a substance varies a lot with temperature, it is very important to control the temperature carefully.

If the room temperature is stable then you might be able to leave your apparatus out in the open. If it can vary by even only a degree or two, then it may be necessary to place the apparatus into a thermostated bath set to a few degrees above room temperature (if available, but this is not mandatory). You could also place the growing apparatus inside a Styrofoam or picnic cooler.

Also, for the seed crystal to grow, it is absolutely necessary that the solution never be unsaturated at the temperature of the experiment (usually the room temperature).

Getting Started

1. Carefully suspend your seed crystal from the stick into the supersaturated solution, being careful not to let the crystal touch the bottom of the container.

2. Cover the container in which the crystal is growing. This is to:
☐ keep out dust, and
☐ reduce temperature fluctuations.
This can be done with plastic wrap or aluminum foil. If you want to allow the solvent (typically water) to evaporate (see step #4a below), then use porous paper (e.g., filter paper or filter paper or coffee filter).

3. 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. A couple of different things can happen at this stage.

The questions and answers below can help you.
☐ Why does the crystal stop growing?
A crystal will only grow when the surrounding solution is supersaturated with solute. When the solution is exactly 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 unsaturated and the crystal material went back into solution. Unsaturation 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?
Step 4 below will give you the details.

4. 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.
One way to resupersaturate the solution is to reduce the amount of solvent. This may be done by heating the solution for a while and then cool it to the original temperature. Or, you can just let the solvent evaporate from the solution (this may be a slow process, but has the advantage of getting a better quality crystal.) One can also supersaturate the solution by warming it somewhat, then adding and dissolving more solute, and finally cooling it.

5. 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.

6. Re-suspend the crystal back into the newly supersaturated solution.

7. Repeat steps 4-6 as needed.

8. 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.

How Are the Crystals Judged?

The Wisconsin competition judging will take place on the 12th of May, 2017.

Each school is encouraged to submit one crystal for best quality and one for best overall. It is recognized that several crystals from a school may be of roughly equivalent overall quality. If this is the case each school may submit several crystals.

Judging Criteria

One single crystal will be judged only on the basis of quality as outlined below. The other single crystal will be judged on the basis of combining mass and quality factors as outlined below.

The quality is judged by experts who will rank the crystals on a scale of 0 to 10. A score of 10 will be given to a perfect gem-quality crystal that fits the ideal crystal structure known for the chemical.

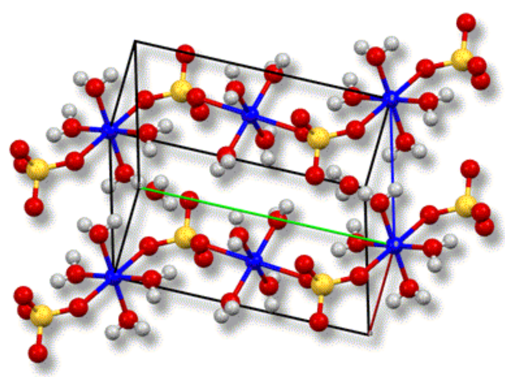
1. The crystal is weighed, and the mass M recorded. The crystal must be a minimum of 0.5 g to be eligible for judging.

2. The quality of the crystal is judged on a scale of 1 to 10, with 10 representing a perfect crystal.

The following factors will be considered in judging quality:

☐ match/mismatch with crystal type (out of 4)
☐ presence/absence of occlusions (out of 4)
☐ intact/broken edges (out of 4)
☐ well formed/misformed faces (out of 4)
☐ clarity/muddiness (out of 4)

Total Observed Quality Qo = x.xx (out of 10)



3. The Total Score is then determined as follows:

Total Score = [log (Mo+1)] x Qo

The logarithm of the mass is chosen so that large poor quality crystals don't swamp out smaller good quality crystals. The value 1 is added to the mass so that crystals weighing less than 1 g get a positive score. A 100 per cent yield crystal made from 100 g (Mt) that scores a perfect 10 on quality (Qt) would get a theoretical maximum of:

[log (100+1)] x 10 = 20.01

The actual score is expressed as a percentage of the maximum. The crystal with the highest Overall Score is the winning crystal.

100 x {[log (Mo+1)] x Qo} / {[log (Mt+1)] x Qt} = Overall Score %

For example, the best overall crystal in the 2001 contest with 150 g starting material weighed 46.53 g and had a quality of 8.65. Its overall score was:

100 x {[log (46.53+1)] x 8.65} / {[log (150+1)] x 10} = 66.6%

This score is nearly an absolute score that could be used to judge different types of crystals grown from differing amounts of starting material.

Crystal Art Judging

This is broadly defined category - it can be a photo, video (no more than 2 minutes), essay (no more than 2 pages), a poem, a haiku, multi-media product or a drawing. The works must be inspired by the crystal-growing contest. They can be scientific (such as an experiment account), descriptive, or fictional, hand drawn or computer-based. Submission of such art work is absolutely optional. The judges will select the best works and award the prizes based on the following criteria: creativity, aesthetic value, clarity of explanations, and scientific background. The participants are encouraged to express themselves by growing crystals and by creating art. Who knows, maybe your work will be published in a scientific journal as it happened in 2014.

If you have questions – contact Dr. Ilia Guzei.

(Example of a haiku:
There are molecules
Inside crystals.
Don't you want to see?)

Acknowledgements

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Contact

With questions of any type regarding this competition contact

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