

# The National Crystal Growing Competition Handbook

## Including:

- Information on the competition
- A guide to crystal growth

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## Information on the National Crystal Growing Competition

## What is the Chemical Institute of Canada?

The Chemical Institute of Canada (CIC) is a Canadian, not-for-profit association of professionals involved in the field of chemistry, chemical engineering and chemical technology. The CIC association organizes outreach activities promoting chemistry to students and the general public. The National Crystal Growing Competition, which has been going on for more than thirty years, is one of these.

## How does this competition work?

The competition aims at growing the largest and highest quality crystal possible. It takes place in Canadian high schools during the fall semester. Winning crystals from each school are sent for evaluation, first to a regional coordinator, and regionally winning crystals are sent for judging at the national level.

## Competition schedule

The 2019 competition schedule appears below. In case of abnormal delays in postal services, or delivery of the substance to be crystallized, please contact your regional coordinator, who will adjust dates if required.

September 3 to 6	Competition announcement sent to high schools by regional coordinators
September 3 to October 9	Schools register with their regional coordinator
September 11 to October 1	Schools order/purchase their chemicals
October 9 to November 12	Crystal growth
November 13 to 15	Schools choose their representing crystals
November 18	Deadline to send your two best crystals to regional coordinator
November to December	Crystals are judged and winners sent to National level.
Early January	National judging and results sent to regional coordinators

#### What can I win?

Students and their teachers can win cash prizes for their school and will receive individual certificates for the national prizes.

National prizes include:

- Best Overall Crystal 1st prize (\$300), 2nd prize (\$200), 3rd prize (\$100)
- Best Quality Crystal 1st prize (\$200)
- Best Teacher's Crystal 1st prize (\$200)

## Who can participate?

All Canadian high school students or homeschooled youth between the ages of 13 and 18 (included) can participate. Students can participate individually or as teams. There are no fixed limits to the number of participants per school.

The competition is also open, although in a separate division, to high school teachers and lab technicians.

## Whom am I up against?

Participants are classified in the following four divisions:

- Division 1: High school students within regions that have coordinators. Crystals are submitted to the regional coordinator for regional judging.
- Division 2: "Outlier" high school students are those living outside regions that have coordinators. These crystals will be submitted to the national coordinator for "regional" judging.
- Division 3: Home-schooled students. These crystals will be submitted to the national coordinator for "regional" judging.
- Division 4: High school teachers. High school teacher crystals are judged for overall quality on a national basis only, separate from crystals submitted by students. These are submitted directly to the national coordinator for judging.

Winning crystals in each regional section of Division 1, and in Divisions 2 to 3 are pooled against one another for the final, national judging.

## How do I sign up?

All you need to do is contact your regional coordinator to let him/her know that you are participating. If you are unsure of who your coordinator is, contact Josée Brisson, national coordinator at <a href="mailto:josee.brisson@chm.ulaval.ca">josee.brisson@chm.ulaval.ca</a>. Please do not overlook this step, as it allows regional coordinators to organize their event and contact participants if their crystals do not arrive.

Parents of home-schooled students should register directly with the national coordinator, at josee.brisson@chm.ulaval.ca.

If you cannot find your regional coordinator in the list annexed, please contact Josée Brisson, national coordinator, at <a href="mailto:josee.brisson@chm.ulaval.ca">josee.brisson@chm.ulaval.ca</a>.

## Important contacts

#### **National coordinator**

Josée Brisson, MCIC
Department of chemistry
Université Laval
1045 Avenue de la Médecine
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Tel.: 418-656-2131, ext. 403536

josee.brisson@chm.ulaval.ca

#### CIC Office

Gale Thirlwall Chemical Institute of Canada Ottawa, Ontario

Tel.: 613-232-6252, ext. 223 gthirlwall@cheminst.ca

## To order your material:

In 2019, the crystals will be grown from copper sulfate (II) 5-hydrate.

- Contact Boreal Science by phone at 1-800-387-9393, by fax at 1-800-668-9106 or by e-mail at borealcs@vwr.com, between September 10 and October 1.
- Use Promo Code NCGC2019 National Crystal Growing Competition item No. 470300-856 when ordering. The company will ship one 500 g bottle of the material directly to you. The cost is \$17.25 for 500 g, plus shipping charges. Payment must be made by credit card, and the shipping address will be that of the school.
- If you need more material, you can order one extra bottle at the same time. Quantity is limited due to the supply available. You may, of course, purchase additional supplies from your own sources.
- Home schools need to identify themselves accordingly. Parents can pay directly, but the shipping address provided must be that of a school.

## How the competition works

Crystal growing must occur between October 9 and November 12. Two main rules apply:

- RULE 1: Participating crystals must weight between 0.5g to 100 g. This corresponds to the use of a maximum of 100 g chemicals per student or team.
- RULE 2: So that all students across the country have an equal preparation time, crystal production must conclude within five weeks after the receipt of starting material.
  - As soon as the growth period is finished, teachers should select the two best crystals that will represent the school at the regional level and send them to the regional coordinator:
- 1) The best quality crystal, notwithstanding size (minimum mass must nevertheless be 0.5 g)
- 2) The best overall crystal, which combines a large size to a good quality (single crystal, well-formed, sharp edges, faces, good transparency, etc.), which must not have a mass higher than 100 g.

Each of these two crystals should be well dried and placed in a small plastic bag (Ziploc or similar) labelled with the name of students having grown the crystal and the participating school.

We know that several crystals from a school may be of roughly equivalent overall quality, and it is sometimes difficult for the teacher to make a choice. In such a case, it may be necessary to submit several crystals. Of these crystals from a given school, only two will be the "official" crystals to be considered for all prize(s) awarded.

## How and when are the crystals judged?

The best crystal from each region will be sent for judging at the national level. Prizes will be attributed to the students having submitted the best crystals. Results will be announced in January.

## Judging criteria

One single crystal will be judged based only on quality, as outlined below. The other single crystal will be judged on mass and quality criteria, as also outlined below.

Experts will rank 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 form known for the chemical.

The crystal is weighed, and the mass Mo recorded. Crystals must weight between 0.5 and 100 g.

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 2)
- presence/absence of occlusions (out of 2)
- intact, clean and well-defined/broken, rounded edges (out of 2)
- well-formed/misformed faces (out of 2)
- clarity/muddiness (out of 2)

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

The **Total Score** is then determined as follows:

Total Score = 
$$[log (Mo+1)] \times Qo$$

The logarithm of the mass is chosen so that large poor quality crystals do not 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 ( $M_{MAX}$ ) that scores a perfect 10 on quality ( $Q_{MAX}$ ) would get a theoretical maximum of:

$$[\log (100+1)] \times 10 = 20.01$$

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

A perfect crystal weighing 100 g would get a score if 100%.

Example: The best overall crystal in the 2001 contest with 150 g starting material ( $M_{MAX}$  = 150 g) weighed 46.53 g and had a quality of 8.65. Its overall score was:

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

## Guide to crystal growth

## What is a crystal?

A crystal is a solid that consists of the various atoms, ions, or molecules organized in a uniform repeating pattern. This results in the material having a specific shape and colour, and having other characteristic properties. Diamond (used in jewellery and cutting tools) is an example of a crystal. It is made of pure carbon. Graphite (used in pencils and lubricants) is also a crystal made from carbon. Salt and sugar are also examples of substances that can form 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 come out of solution in crystalline form. Depending upon conditions, one may obtain a mass of many small crystals or one large crystal. Click on these links for more detailed information:

https://www.otago.ac.nz/chemistry/outreach/crystals/growing/index.html

https://uwaterloo.ca/earth-sciences-museum/resources/crystal-shapes

https://www.youtube.com/watch?v=gsC039jpOT0

http://www.ccp14.ac.uk/ccp/web-mirrors/paulboyle/student\_xtal.html

## Which substances crystallize well?

Many substances crystallize. At home, table salt (sodium chloride) is often used to grow crystals. For the competition, we change the chemical used each year. Thus far, the National Crystal Growing Competition has used three different substances:

- Cupric sulfate pentahydrate (copper (II) sulfate pentahydrate, or "bluestone");
- Aluminum potassium sulfate (or "alum"). This substance seems to be a popular one with which to experiment;
- Potassium sodium tartrate (or "Rochelle Salt").

The choices were made based on relative safety, availability, and good crystal growth. The first two chemicals are available from most laboratory chemical supply houses. The third may be bought at a grocery store, but make sure to use real cream of tartar to do your experiment.

Other chemicals that are known to give good crystals include:

- Potassium ferricyanide,
- Copper acetate monohydrate, and
- Calcium copper acetate hexahydrate.

## How to grow crystals

To obtain large single crystals, three steps are usually followed:

- 1) A small seed crystal is obtained
- 2) A supersaturated solution is prepared

3) Increasing the size of the seed crystal is performed by suspending this seed crystal into the supersaturated solution (more details later).

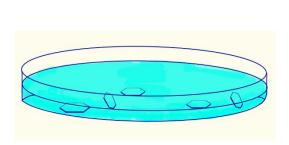
It is relatively easy to obtain nice small crystals. Obtaining larger, almost perfect crystals is almost an art, and requires constant attention to details. The rest of this guide will teach you the basics of this art.

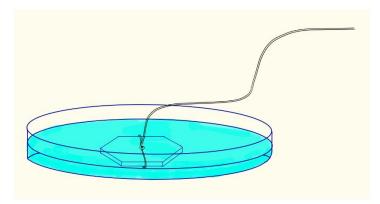
What you need to know before starting

- 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 the *Handbook of Chemistry and Physics*, section *Aqueous Solubility of Inorganic Compounds in Mass % as a Function of Temperature*.

## First step: growing a seed crystal

The aim of the competition is to obtain a single crystal and not a bunch of crystals all stuck together and intergrown, no matter how nice this may look. In order to do this, you will need a small, well-formed crystal, which will serve a "seed" to start crystal growth. You will then let this small crystal slowly grow bigger, as a rapid crystallization often leads to multiple crystals instead of a single, well-formed crystal.





## What you will need

- Substance to be crystallized
- Distilled or demineralized water
- A shallow dish (e.g., Petri)
- Heating plate or stove
- Beaker or other small container, 100 to 250 mL capacity
- Fishing line (1 to 2 kg strength)
- Small stick (Popsicle stick works fine)
- A magnifying glass (optional).

## What you should do

- Warm about 50 mL (1/4 cup) of water in a glass container.
- Dissolve enough of the substance to produce a saturated solution at 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 magnifying glass, select a regular, small transparent crystal. This will be your seed crystal.

- Tie the seed crystal with the fishing line by using a simple overhand knot. Make sure the line is long enough (approximately half of the height of the beaker used in Step 3).
- Suspend the seed crystal in a shallow dish (1 to 2 mm deep) containing a small amount (about 1 to 2 mL) of supersaturated 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, and that the crystal grows over the line. 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 step: Preparation of a supersaturated solution

To grow a large crystal, you will need to suspend it in a supersaturated solution. This solution is not in an equilibrium state: you want it to contain more of the crystallizing substance (the solute) than normally presented. In order to do this; you will first prepare a saturated solution, which is at equilibrium and which contains the maximum of the substance that can dissolve in water. Afterwards, you will change the conditions in order for the substance to be too concentrated as compared to equilibrium conditions and the solute will need to crystallize out. This, however, takes time, and meanwhile, your solution is supersaturated!

Many methods exist to obtain a supersaturated solution. We have given below three different methods.

Please note: Quantities of the growing substance and of water depend on solubility at room temperature and at a slightly higher temperature. Handbook data will guide you, but you may also need to proceed by trial and error to determine the exact proportions, just like any scientist would do when beginning a new experiment.

## What you will need

- Substance to be crystallized
- Distilled or demineralized water
- Thermometer
- Balance
- Plastic or glass container (0.5 to 1 L)
- Heating plate

#### 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.

Note: The supersaturation method, as well as the second one, 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. Some substances are even more soluble at low than at high temperatures.

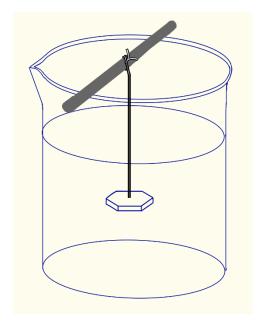
## Method Two

- Select an appropriate volume of water.
- Warm this water to about 15–20° above room temperature.
- Add some of your substance to the warm water and stir the mixture to dissolve completely.
- Continue adding the substance and stirring until there is a slight amount of material that won't dissolve.
- Warm the mixture a bit more until the remaining material goes into solution. The solution must be entirely transparent, with no haziness whatsoever.
- 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 Three

- Prepare a saturated solution, using the solubility value (mass of substance / volume of water) found in a chemistry handbook.
- Let the water evaporate (this can take a few hours up to a few days, depending on how well saturated the initial solution is).

## Third step: Growth of a large crystal



You are now ready for the final growing step. You will suspend your seed crystal in the supersaturated solution prepared in Step 2. This solution contains more than the maximum solubility of the solute, it is not at equilibrium, and the solute will "want" to get out of the solution. It will crystallize onto any surface that can serve as a seed, including your seed crystal (and this is what you want) or any dust particle or solid impurity present (which you want to avoid, as it will give irregular, badly shaped crystals).

The speed at which crystallization occurs will greatly influence final crystal quality. The more supersaturated the solution (the higher the excess in solute as compared to equilibrium conditions), the faster the crystal will grow. However, the best crystals are always obtained with a slow growing speed.

One word of warning: as solubility varies with temperature, it is of utmost importance to avoid temperature fluctuations during crystal growth. Further, your solution must always remain saturated at the chosen growth temperature (usually room temperature), or else your crystal could dissolve partially or entirely.

Unfortunately, many schools have had the disappointment of admiring a big crystal on a Friday, only to come back on Monday to an empty fishing line. The reason? A rise in room temperature in the school over the weekend, which caused the crystal to dissolve completely. The worst period for this is around October, when schools turn on the heating systems. For this reason, if available, growing the crystals in a thermostated bath set to a few degrees above room temperature is highly recommended. If unavailable, placing the growth set up inside a cooler (Styrofoam or other) will limit temperature fluctuations, and can also save the day.

## What you will need

- Substance to be crystallized
- Seed crystal of the substance to be crystallized, attached on a fishing line
- Small wood stick (e.g., Popsicle stick)
- Distilled or demineralized water
- Thermometer
- Balance
- · Heating plate or other heating device
- Beaker of 2 to 4 litres volume
- Styrofoam of picnic cooled (optional)
- Magnifying glass or microscope (optional)
- Thermostated bath (optional)
- Slow revolution motor (1 to 4 rotations per day) (optional)

## What you should do

- 1. Take the fishing line on which you have attached your seeding crystal. Attach a wooden stick (Popsicle stick) to the other end, making sure the crystal will be immersed approximately in the middle of your supersaturated solution in the growing vessel.
- 2. Carefully suspend your seed crystal from the stick into the supersaturated solution, taking care not to let the crystal touch the bottom of the container.
- 3. Cover the container in which the crystal is growing. This is to:
  - keep out dust, and
  - reduce temperature fluctuations.

This can be done using plastic wrap or aluminum foil. If you want to allow the solvent (typically water) to evaporate (see step #4 below), then use porous paper (e.g., filter paper).

4. Observe the crystal growth. Depending upon the substance, the degree of supersaturation, and the temperature, it may take several days before the growth slows down and stops.

WHY DOES THE CRYSTAL STOP GROWING?

A crystal will only grow when the surrounding solution is supersaturated with the 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?

You must resupersaturate your solution. Step 5 will give you the details.

5. Resupersaturate 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. Alternatively, 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.) You can also supersaturate the solution by warming it somewhat, then adding and dissolving more solute, and finally cooling it.

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.
- 6. Resuspend the crystal back into the newly supersaturated solution.
- 7. Repeat steps 5 and 6 as needed.
- 8. To get improved symmetry and size, especially if the crystal gets very big, better results will be obtained if you slowly rotate the growing monocrystal (1 to 4 rotations per day) while it is immersed in the supersaturated solution. An electric motor with 1 to 4 daily rotations might be difficult to find (reusing one from an old humidity drum-register or a similar apparatus is suggested).
- 9. Remove your crystal from the bath once the growth period is finished, and wipe it thoroughly with absorbent paper. Do not touch your crystal with your fingers! Once well dried, put your crystal in a small plastic bag (such as a Ziploc bag), close it, and put a label on the bag stating your name and school name, and give the crystal to your teacher or lab technician. He or she will choose the best two crystals and will send them to the regional coordinator for judging. It is important to verify, before sending any crystal, if their mass falls between the 0.5 to 100 g thresholds for acceptable crystals: if not, the crystals are automatically disqualified.

The teacher or technician should consult sections "Competition schedule", "How the competition works" and "Judging criteria" for more information on how the crystal are judged, and when and how to send your two selected crystals to the regional coordinator.

At this stage, some people varnish their crystals. This insures that the crystals do not dry out, and maintains their general shape longer. This may hide some defects present on the crystal faces, but on the

other hand, edges appear rounder, less well defined. Although we have decided not to disqualify these varnished crystals, these will invariably lose many points due to loss in perfection of the edges, and will invariably fall down the ranking. We do not recommend that you varnish the crystals that will be submitted to the competition.

10. Finally, when manipulating chemicals, is it essential to always thoroughly wash your hands with soap and water afterwards.

For professional instructions on how to grow crystals, you may also want to consult an article written by Paul Boyle "Growing crystals that will make your crystallographer happy", available on the web.

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 colours or shapes. These will, however, not be accepted for the Competition.

Last revision: August 4, 2019.