Quiet! Seed Crystals Growing

A Crystal Growing Activity

Introduction

Like magic, a speck appears in a saturated solution. Soon it looks like a flock of freckles. Then, SUCCESS! Seed crystals are growing …

Concepts

- Crystal Growing
- Saturation/Supersaturation
- Solubility

Background

In this activity, crystals will be "grown" from saturated solutions. To completely understand how these crystals will "grow" out of a solution, the concept of saturation and supersaturation must first be addressed.

A *solution* is a homogeneous mixture of two or more substances. Generally, solutions are thought of as solutes dissolved in a solvent—usually water. The *solubility* of a given solute is the largest amount of that solute that will dissolve in a specified volume of solvent at equilibrium and at a particular temperature. Solubility is strongly temperature dependent; it generally increases with increasing temperature. Solubility also depends on the substance involved. Some salts are very soluble in water, while others are only slightly soluble.

A solution is said to be *unsaturated* if its solute concentration is less than its solubility. When a solute's concentration is equal to its solubility, the solution is said to be *saturated*. At that temperature, no more solid, not even a small grain, can be dissolved in the solution. However, if a saturated solution is heated, its solubility may increase, making it possible to dissolve more solid in that same solution. If additional solid is added and then the solution is cooled, it might be expected that the extra solid would precipitate out of solution. This does not always happen though. Instead, the extra solid may remain dissolved in solution even though its concentration has exceeded its solubility. In this case, the solution is said to be supersaturated. A *supersaturated* solution is one that is more concentrated than a saturated solution at the same temperature.

To clarify the difference between unsaturated, saturated, and supersaturated solutions, consider what happens when additional solute (in the form of a crystal) is added to each type of solution:

- If more solute is added to an unsaturated solution, it will dissolve because the solution can accept more solute and still have a concentration less than the solubility.
- When additional solute is added to a saturated solution, the crystal cannot dissolve because the concentration of the original solution is already equal to the solubility. Neither will the crystal grow, since there is no excess solute dis solved in solution to precipitate out on the crystals. In this case, the crystal will remain the same size.

If a crystal of solute is added to a supersaturated solution, it will not dissolve because the concentration of the original solution was already greater than the solubility. Instead, the crystal provides a lattice upon which the excess solid in solution (that which made the solution supersaturated) can precipitate. As a result, the crystal will grow until the concentration of the solution is equal to the solubility. The crystal of solute added is called a *seed crystal*—it is planted in the supersaturated solution and grows into a bigger crystal. The supersaturated solution in which the seed crystal grows is called a *growing solution*. Supersaturated solutions are fragile solutions. Not only will adding a crystal of solute cause them to precipitate, but disturbing the solution in other ways, such as stirring or scratching the walls of the container, may also cause the excess dissolved solute to precipitate out of solution until only a saturated solution remains.

The above scenarios apply when the solution is held at constant temperature—because as soon as the temperature changes, so does the solubility.

Material

Salt for growing crystals—good choices include:

Aluminum ammonium sulfate (ammonium alum), $AlNH_4(SO_4)_2 \cdot 12H_2O$
Aluminum potassium sulfate (alum), $AlK(SO_4)_2 \cdot 12H_2O$
Chromium potassium sulfate (chrome alum), $CrK(SO_4)_2 \cdot 12H_2O$
Copper(II) sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$

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Potassium sodium tartrate (Rochelle salt), $KNaC_4H_4O_6 \cdot 4H_2O$
Potassium ferricyanide, $K_3Fe(CN)_6$
Sodium chlorate, $NaClO_3$

Water, distilled or deionized
Beaker, small, Pyrex®
Bottle with cap for storing the stock solution
Container with a flat bottom (the growing container)
Microwave oven (not used for food) or hot plate
Pencil
Spatula
Transparent nylon thread or transparent fishing line
Tweezers

Safety Precautions

Aluminum ammonium sulfate, aluminum potassium sulfate, and potassium sodium tartrate are considered nonhazardous. Chromium potassium sulfate is a body tissue irritant. Copper(II) sulfate is moderately toxic by ingestion and inhalation and is a skin and respiratory irritant. Potassium ferricyanide is slightly toxic; however, if strongly heated or in contact with concentrated acids, poisonous fumes of hydrogen cyanide gas may evolve. Sodium chlorate is a strong oxidant, a dangerous fire risk, and is slightly toxic. Contact between sodium chlorate and organic material may cause fire. Avoid handling crystals with bare hands. Use caution when handling hot glassware. It is not recommended that students be allowed to take crystals home. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Please review current Material Safety Data Sheets for additional safety, handling, and disposal information.

Procedure

Part 1. Preparing a Supersaturated Solution.

Note: Never discard any solutions. There certainly is a great temptation to do so when starting over is necessary, but there is no good reason to waste prepared solutions. If the chemistry of solutions is understood, it is easy to be successful in growing perfect gems.

1. Add about 30 mL of distilled or deionized water to a small, Pyrex beaker. Add a spatula-full of the crystal salt. Stir until it all dissolves. The result, at this point, is an unsaturated solution.
2. Add another spatula-full of the crystal salt and stir. Notice that the solution becomes cloudy as the small powdered particles are swirled into solution. If the cloudiness clears, the solution is still unsaturated. Keep adding the crystal salt until a layer of salt remains at the bottom of the beaker. At this point, the solution is saturated at room temperature.
3. Place the beaker in a microwave (not used for food) and heat for about 30 seconds. Swirl to see that all of the crystal salt is dissolved. If a microwave not used for food is not available, heat the solution on a hot plate or over a Bunsen burner until all of the salt is dissolved. The solution is once again unsaturated at this higher temperature.
4. While the solution is still warm, add one more spatula-full of the crystal salt and swirl to dissolve. It may be necessary to return the saturated solution to the microwave or hot plate to ensure that the solution is saturated at the higher temperature. It is a saturated solution when it is clear and no salt remains on the bottom of the beaker.
5. Set the solution aside undisturbed for about an hour and allow it to cool to room temperature. At that time, the room temperature solution should be a supersaturated solution.

Part 2. Growing Seed Crystals

6. Carefully pour the supersaturated solution into a growing container. It is important that the growing container have a flat bottom. Leave the supersaturated solution in the growing container uncovered. Do not disturb the solution for at least one hour after transferring it to the growing container. During this time, as the solvent evaporates, tiny seed crystals will begin to form. They appear as tiny dots or "freckles" in the solution. Once crystals are observed, do not disturb the growing container until the next morning.
7. By the next morning, two situations are possible:

   a. Success! A collection of seed crystals is present at the bottom of the growing container. These seed crystals should have grown to a size that can be easily handled. Ideally, a dozen or more seed crystals should be present. If so, choose three or four from the collection of seed crystals to grow individually in small containers.

b. Unfortunately, a collection of good seed crystals is not present. In this case, many tiny sand-like granules at the bottom of the growing container are observed, or there may be a large clump of crystals stuck together in a hard mass. If either of these situations exists, return the liquid and the sediment to the mixing container used in Part 1 and redissolve the salt. If necessary, use a spatula to scrape the sediment back into the mixing container. Add about 3 mL of distilled or deionized water and repeat Steps 3–7 until a collection of good seed crystals is produced. It really is a trial and error procedure, so be patient as this may take several tries. Above all, do not discard any of your solutions!

Part 3. Growing Crystals

Once growing seed crystals is perfected, larger crystals may be grown following one of two methods—suspended in solution or on a flat-bottomed surface at the bottom of a solution. Each method has its own advantages and disadvantages.

Method 1. Growing Crystals Suspended in Solution

8. Retrieve all seed crystals and solid pieces from the growing container so that all that remains in the growing container is a saturated solution.
9. Choose a seed crystal and tie a string around the crystal so that is can be suspended in the saturated solution. Avoid handling the crystal as much as possible as this can destroy the resulting crystal's shape. Transparent nylon thread or transparent fishing line can be used to tie the crystal. In either case, a second pair of hands is quite helpful when tying the thread around the crystal.
10. Once the seed crystal is securely tied, suspend it in the saturated solution so that it is completely surrounded by solution. Wrap the string around a pencil and place the pencil across the top of the growing container to hold the crystal suspended in solution.
11. Check regularly to make sure that the growing crystal is always completely submerged. As time passes, the solution will begin to evaporate. Add additional stock solution (see Part 4) if necessary to ensure that the growing crystal is always completely submerged. When adding additional stock solution, be careful that no solid particles are added. If necessary, carefully decant the stock solution into the crystal-growing container.
12. As the crystal grows larger, it may be necessary to transfer the crystal and its solution to a larger container. Again, add additional stock solution (see Part 4) if necessary to ensure the crystal is completely surrounded by solution.

Method 2. Growing Crystals on a Flat-Bottomed Surface in Solution

13. Retrieve all seed crystals and solid pieces from the growing container so that all that remains in the growing container is a saturated solution.
15. Place a single seed crystal at the bottom of the growing container. Avoid handling the crystal as much as possible as this can destroy the resulting crystal's shape.
16. Rotate the seed crystal at least once daily to ensure that the crystal grows evenly. As time progresses, the solution will begin to evaporate. If the solution level becomes low enough, add additional stock solution (see Part 4) to ensure the crystal is always completely submerged. When adding additional stock solution, be careful that no solid particles are added. If necessary, carefully decant the stock solution into the crystal-growing container.
17. As the crystal grows larger, it may be necessary to transfer the crystal and its solution to a larger container. Again, add additional stock solution (see Part 4) if necessary to ensure the crystal is completely submerged. It is important that any containers used for growing crystals have a flat bottom. A piece of window glass cut to fit the container is a good way to ensure the crystal is grown on a flat surface.

Part 4. Care and Feeding of a Stock Solution

18. As the crystals grow and the surrounding solution evaporates, it will be necessary to replace the lost fluid. This is done with a reserve or stock solution. Keep these five points in mind when working with stock solutions:

    a. Prepare the stock solution exactly as the original solution was prepared by following Steps 1–9.

    b. Keep the stock solution tightly capped when not in use to avoid evaporation.

    c. The stock solution should have a small layer of undissolved solid at the bottom to indicate that it is saturated.

    d. When adding stock solution to the crystal growing solution, always decant the stock solution slowly, pouring off the liquid while leaving all sediment behind.

e. Keep the stock solution near the crystal-growing container so that both solutions are always at the same temperature.

Part 5. Tips for Maintaining the Growing Crystals

19. Check on the appearance of the growing crystal daily. If "satellite" crystals are starting to grow on the crystal, remove them by carefully wiping with a spatula. Be careful not to harm the crystal in the process, however.
20. Avoid over-handling the crystals as this can destroy their shape. Always use tweezers (not fingers) when moving the crystal.
21. If the crystal-growing container develops a layer of sediment on the bottom, transfer the crystal to an empty container. Then slowly and carefully decant the liquid in which the crystal was growing into the new container so that the crystal is again submerged completely. Do not allow any of the solid sediment to be transferred to the new container. Instead, transfer the sediment to the stock bottle.
22. It is ideal to have a volume of stock solution readily available equal to the volume of the saturated solution covering the crystal.
23. One of the most important factors when growing crystals is changes in temperature. Because an increase in temperature increases the solubility of the solution, a temperature rise may cause the suspended crystal to dissolve in solution. If the temperature in your classroom varies throughout the day, place the growing container in a water bath.
24. After several weeks of growing, the crystal may develop a stringy-like growth due to collection of dust. To remove this growth, remove the crystal from the saturated solution. Transfer the saturated solution to the stock bottle. Reheat the stock solution in a microwave or on a hot plate until all of the solid is dissolved. Strain the warm solution through a towel or filter paper back into the stock bottle. Allow the solution in the stock bottle to sit overnight undisturbed. The next day, decant the solution in the stock bottle back into the crystal-growing container with the crystal. This procedure may have to be performed about every two weeks to keep the crystal looking good.

Part 6. The Final Crystal

25. When the crystal has grown to the desired size, remove it from the growing solution and allow it to dry. Its appearance will usually change once it has dried. To keep the air from altering the appearance of the crystal further, it may be coated with clear fingernail polish.

General Tips for Growing Crystals

- A good seed crystal is the key to success in crystal growing and each solid varies in its propensity to form seed crystals. A good seed crystal is about ¼ to ⅛ inch long. It must be a single crystal so that the crystal growing from it will also be a single crystal. If the seed crystal is too small, it will dissolve more easily once it is hung in solution, it will be more difficult to tie on the string, and it may float on the surface of the water instead of hanging in solution. If the seed crystal is too large, it may have a greater chance of having irregularities in its structure, or may have grown too fast, causing it to be cloudy. Growing containers may be placed in a water bath to help them stay at a constant temperature.
- Make sure the jars or beakers used for growing crystals are clean and free from scratches. Scratches may cause too much precipitate to form, or may cause the seed crystals to take on an imperfect shape.
- Some solids are more difficult to grow seed crystals with than other crystals. Alum and copper(II) sulfate are the easiest to grow, while chrome alum and potassium ferricyanide are among the hardest to grow.
- In general, the amount of solid to add to 30 mL of water to prepare the initial solution for growing crystals is about twice the solubility in 30 mL of cold water. See the Characteristics of Crystals table below for solubilities.

Characteristics of Crystals*

Chemical Formula	Color	Shape	Solubility (g/100 mL cold water)
Ammonium alum	Colorless	Hexagonal	Very soluble
Alum	Colorless	Octahedral	11.4
Chrome alum	Purple	Octahedral	24.4
Copper(II) sulfate	Blue	Triclinic	31.6
Rochelle salt	Colorless	Orthorhombic	47.4
Potassium ferricyanide	Red	Monoclinic	33
Sodium chlorate	Colorless	Cubic	79

**CRC Handbook of Chemistry and Physics, 69th ed.; Weast, R. C., Ed.; CRC: Boca Raton, 1988.*

Disposal

Please consult your current *Flinn Scientific Catalog/Reference Manual* for general guidelines and specific procedures, and review all federal, state and local regulations that may apply, before proceeding. Aluminum ammonium sulfate, aluminum potassium sulfate, copper(II) sulfate, and potassium sodium tartrate crystals may be disposed of in the trash according to Flinn Suggested Disposal Method #26a and their solutions may be flushed down the drain with an excess of water according to Flinn Suggested Disposal Method #26b. Dispose of chromium potassium sulfate crystals and solution according to Flinn Suggested Disposal Method #27f. Dispose of potassium ferricyanide crystals and solution according to Flinn Suggested Disposal Method #14. Dispose of sodium chlorate crystals and solution according to Flinn Suggested Disposal Method #12a.

Connecting to the National Standards

This laboratory activity relates to the following National Science Education Standards (1996):

Unifying Concepts and Processes: Grades K–12
Constancy, change, and measurement
Form and function
Content Standards: Grades 5–8
Content Standard A: Science as Inquiry
Content Standard B: Physical Science, properties and changes of properties in matter
Content Standards: Grades 9–12
Content Standard A: Science as Inquiry
Content Standard B: Physical Science, structure and properties of matter, chemical reactions, interactions of energy and matter

Acknowledgment

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Reference

Holden, A.; Morrison, P. *Crystals and Crystal Growing;* MIT: Cambridge, MA, 1995.

Materials for *Quiet! Seed Crystals Growing* are available from Flinn Scientific, Inc.

Catalog No.	Description
A0224	Aluminum Ammonium Sulfate, 100 g
A0265	Aluminum Potassium Sulfate, 100 g
C0205	Chromium Potassium Sulfate, 100 g
C0102	Copper(II) Sulfate Pentahydrate, 100 g
P0084	Potassium Sodium Tartrate, 100 g
P0050	Potassium Ferricyanide, 100 g
S0216	Sodium Chlorate, 100 g
AP8447	Bottle, Ointment Jar Style, 16 oz
AP2260	Crystals and Crystal Growing
AP4846	Crystal Growing Kit
AP4682	Giant Crystal Growing Kit

Consult the Flinn Scientific website for current prices.