GROWING ALUM CRYSTALS

INTRODUCTION

In nature, crystals are formed naturally from molten rocks (magmas), from hot aqueous solutions, or from hot gases. Well-formed crystals, frequently encountered in hydrothermal veins, are spectacular in form and color. Such crystals have always held a fascination for people. Throughout time, people have collected crystals, treasured them, studied them, and even ascribed magical powers to them. *Mineralogy*, the description and study of crystals, is one of the oldest scientific disciplines. Over the centuries, have learned how to cut and shape crystals, generally by trial and error, to enhance their beauty and value, but the actual understanding of crystal structures, and classification of them, did not occur until the early twentieth century.

In addition to jewelry, crystals are very important. Crystals of carbon, better known as diamonds, are the hardest naturally occurring material and, therefore, find applications as abrasives to cut and polish hard materials. Diamonds are valued as gemstones because of their optical properties, their hardness, and their rarity. Quartz (SiO_2) crystals serve as frequency generators in electronic applications; calcite ($CaCO_3$) crystals are used as polarizers for light waves in microscopes; and crystals of silicon (Si) are the starting material for computer chips.

A crystal is characterized by the orderly, repetitive arrangement in three dimensions of the ions, atoms, or molecules that make up the crystal. The structures of crystals can be determined by a technique known as single-crystal X-ray diffraction. One of the first crystals to be investigated by this technique approximately 70 years ago was sodium chloride (table salt). Sodium chloride crystallizes in the form of cubes. Each sodium ion is surrounded octahedrally by six chloride anions and each chloride by six sodium cations (See Figure 1).

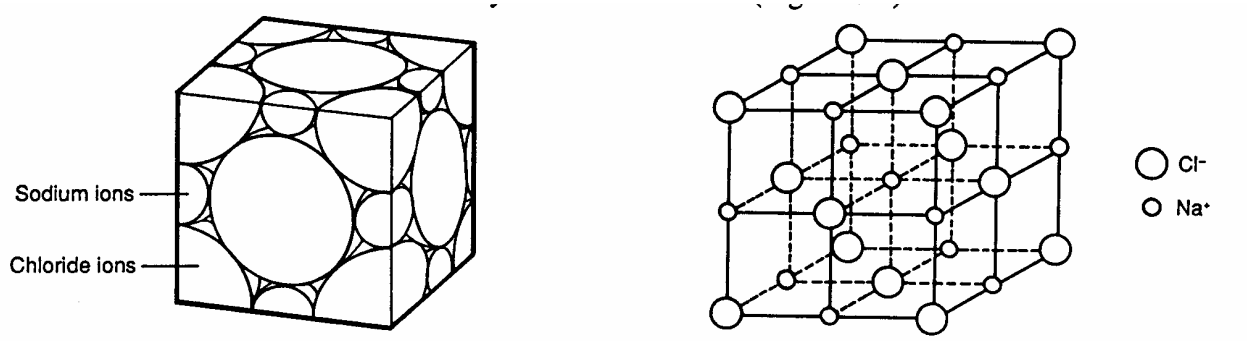


Figure 1. Sketch of a cubic sodium chloride crystal and the arrangement of sodium cations and chloride anions in the sodium chloride lattice.

Sodium chloride crystals can be seen by looking at the salt grains from a salt shaker with a magnifying glass or microscope.

The need for crystals now exceeds what nature can supply. The natural supply is either insufficient (quartz crystals) or the needed crystals do not occur naturally in nature (silicon crystals). Many crystals are now grown "artificially". Many gemstones, industrial grade diamonds, and crystals with special optical, electric, and magnetic properties are produced commercially. In addition, research is being conducted on growing crystals in space. Solid state chemistry, the branch of chemistry dealing with the preparation of crystalline materials and the relationships between composition, structure, and properties, is practiced by an ever-increasing number of chemists and physicists.

In the chemistry laboratory and in industry, crystals are grown for the purification of substances by a process called recrystallization. This method is used to produce the granulated sugar that you may use to sweeten foods or

dinks. To recrystallize a substance, the solid is generally dissolved in a suitable solvent at elevated temperature. After removal of any undissolved material and impurities by filtration, the filtrate (solution) is cooled slowly to allow crystals to form. The slower the crystals grow and the larger the crystals become, the purer the substance will be.

This experiment demonstrates crystal growth from an aqueous solution using potassium aluminum sulfate, known as alum. The substance crystallizes easily in the form of octahedra.

BACKGROUND INFORMATION

Many substances will dissolve in water forming aqueous solutions. The mass of a substance, also termed the *solute*, that will dissolve in a given quantity of water, usually set at 100 grams, is not unlimited. A solution that contains the maximum amount a substance as will dissolve a given amount of water is called *saturated*. The mass of substance present in a saturated solution prepared with 100 g of water is known as the *solubility of the substance*. The solubilities of many substances are compiled in handbooks of chemistry. For instance, the solubility of sodium chloride is 35.6 grams NaCI in 100 g of water at 20°C. The solubility changes with temperature. Most substances are more soluble at higher temperatures than at lower temperatures. When a solution saturated at a higher temperature is cooled to a lower temperature, one of two things occur. Some of the excess solid will crystallize out of the solution, forming crystals on the bottom of the container, or, all the solid may remain in the solution. The cool solution, where everything remains dissolved, will be *supersaturated*, that is, it contains more solute than allowed by its solubility at that temperature. Supersaturated solutions are unstable and will, in most cases, deposit the excess solute in crystalline form when disturbed. An example of this are chemical hot packs, available in many pharmacies, which look like plastic packets of liquid with a small metal disk in them. When the metal disk is “clicked”, it sends vibrations through the liquid, a supersaturated solution of sodium acetate, causing the excess solute to crystallize, giving off heat. The hot pack is regenerated by boiling it in water for 15 to 30 minutes.

Another way to force a solute to crystallize is by evaporation of the solvent. When a saturated aqueous solution of a substance is allowed to stand in an open container at room temperature, the water will slowly evaporate forcing the solute in excess of the solubility to crystallize.

In an aqueous solution of $KA1(SO_4)_2$ the K^+, $A1^{3+}$, and SO_2^{2-} are surrounded by molecules of water (they are hydrated). These ions do not have an orderly arrangement in solution. When the compound is forced to crystallize, the ions must begin to join each other in their characteristic order. This process of nucleation may occur spontaneously when the ions of alum collide with appropriate orientation and with sufficiently low kinetic energy to permit them to "stick" to each other and prevent them from rebounding. Occasionally, some "foreign" solids (irregularities on the wall of the container, dust particles) will serve as nuclei (or starting points) for the formation of crystals. Once a tiny crystal has formed, ions in their random motion through the solution will hit the faces of the crystal, join the orderly array of ions, and make the crystal grow. To keep the crystal growing, the solution must be cooled to even lower temperatures or solvent must be evaporated continuously. To obtain large crystals, small seed crystals are first prepared. A well formed seed crystal is then suspended in a saturated growing solution and the solvent slowly evaporated. By replenishing the growing solution a huge, perfectly octahedral crystal of alum can be obtained.

SAFETY

Alum is non-toxic. Alum solutions can cause eye irritation. Wear goggles or safety glasses when working with the solution.

Store your growing solutions in a place in which they will not be disturbed.

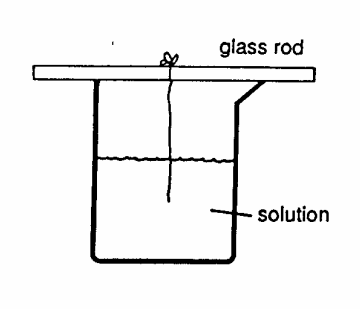
DISPOSAL

The materials used in this experiment are non-toxic and can be safely disposed of in the trash or liquids can be poured down the drain.

PROCEDURE

Weigh out approximately 10 g of alum, $KA1(SO_4)_2 \cdot 12H_2O$. (NOTE: 1 Tbs. alum has a mass of approximately 10 g) Place the alum into a clean 250-mL beaker (or 1/4 cup container such as a small glass or jar). For each gram of alum in the beaker add 7 mL of water. (Note: one teaspoon of water = 5 mL; one tablespoon = 15 mL) Heat the mixture to approximately 60ºC. (Heating can be done in a microwave oven.) Stir until all the alum has dissolved. Should the mixture remain cloudy, let it stand for a few minutes to allow the suspended matter to settle. Carefully decant the clear solution in to another clean 250-mL beaker. If necessary, heat the mixture, briefly, to effect complete solution of the alum crystals.

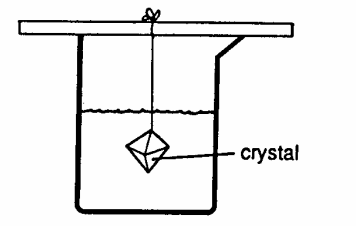
Tie a piece of thread to a glass rod, popsicle stick, pencil, large toothpick, or other support object. Adjust the length of the thread so that not more than 1/2 inch (1.3 cm) is submerged. Smear grease (such as Vaseline) or oil (cooking oil or mineral oil) on the part of the thread above the solution to prevent the solution from creeping up the thread. Place the rod on top of the beaker, cover the beaker with a piece of paper towel held in place with a rubber band, and store the beaker in a safe place. Crystals should form on the submerged thread, at the bottom of the beaker, or in both places within a few days or by the end of one week.



Remove the thread from the solution. Remove all the crystals except the best formed one from the string. If a suitable crystal has not formed on the string, decant the solution, from the crystals at the bottom of the beaker, into a clean container. Inspect the crystals and select one that has a regular octahedral shape and smooth faces. Loop a fine thread around the selected crystal and tie it with a knot. Save several of the remaining crystals as reserve, in case anything happens to your first choice seed crystal. (Wrap the reserve crystals in plastic wrap or place in a small plastic bag to keep them from drying out.)

Redissolve the remaining alum crystals in the alum solution, heating to about 60°C, as described in the first step of this procedure. If all the alum does not dissolve, you may have to add a minimum amount of water. Add 5 mL (1 teaspoon measure) of water and warm the solution back to about 60°C. If necessary, add additional water in 5 mL increments, warming the solution between additions, until all the alum dissolves. Allow this solution to cool to room temperature.

Suspend your choice seed crystal in the cool solution, cover the beaker with a paper towel, and keep the beaker undisturbed for a week. If your solution is not saturated at the time you add your seed crystal, the crystal will begin to dissolve and may be lost. To prevent such an undesirable occurrence, observe the solution in the vicinity of the seed crystal after its placement into the solution. Should the solution be unsaturated, causing the crystal to begin to dissolve, the part of the solution in contact with the crystal will become more concentrated and denser than the solution more remote from the crystal. The denser solution will flow toward the bottom of the beaker. Should you see such a density current, remove the seed crystal, cool the solution further, dissolve more alum, or let the solution sit undisturbed for about a day to allow for some evaporation of the water.



Inspect the alum crystal and solution on a regular basis. If the crystal has stopped growing, and other crystals have formed on the bottom of the solution, remove the crystal and warm the solution to dissolve the additional crystals. If necessary, add small amounts of water (about 2 mL or 1/2 teaspoon) and warm until all the crystals dissolve. Cool the solution and add the large crystal.

If no crystals have formed, your solution is too dilute. Warm the solution and allow it to stand for up to 24 hours, so that some evaporation of water takes place. Try to grow another crystal.

You may try to increase the size of your crystal by removing it from the solution each week and either redissolve any crystals that have formed, or suspend it in a fresh, saturated alum solution. To keep large crystals completely submerged you might have to prepare larger volumes of alum solutions always maintaining the ratio of about 1 g of alum to 7 mL of water. (If additional alum is needed, request some from your instructor.) If a balance is available, weigh your crystal each week and describe its color, size and shape. (If a balance is not available, weigh your final crystals in the laboratory before giving them to your instructor for evaluation.)

Crystal growing is an art and can sometimes be frustrating. If you were unsuccessful in growing a large crystal, turn in any small crystals that formed. You will still get credit for growing crystals.

Your instructor will evaluate and grade your crystal(s) when you have turned them in. You may keep the crystal after your work has been graded. To preserve the crystal and prevent it from crumbling to a white powder through loss of water, keep it in a plastic bag or cover it with a clear plastic spray available from your instructor.

OPTIONAL: As an additional project, a layered, colored octahedron can be grown by substituting a solution of "chrome alum," $KCr(SO_4)_2 \cdot 12H_2O$ for an additional alum growing solution. Dissolve 8 g $KAl(SO_4)_2 \cdot 12H_2O$ in 56 mL distilled water and 15 g $KCr(SO_4)_2 \cdot 12H_2O$ in 25 mL of water as described at the beginning of this procedure. Hold the beaker with the colorless alum solution toward a light. Slowly pour the chrome alum solution into the colorless alum solution. Mix well and check whether you can still see through the mixed solution. Stop adding the chrome alum solution when it has become difficult to see through the mixed solution. Suspend your crystal in this mixed solution. For subsequent growing stages, use solutions of $KAl(SO_4)_2 \cdot 12H_2O$.

Your instructor will have a collection container for all waste solutions containing chromium.

Maintain records of the appearance and, if available, the mass of the crystal at each growing stage. Record how much alum and water you used at each stage. Complete the Report Form using your notebook records.

REPORT FORM

GROWING ALUM CRYSTALS

Name _____ Course/Section _____

Date _____

DATA AND RESULTS

Record your operations in preparing and growing your alum crystal(s). Include a description of the stages of the crystal growth. (Use additional paper as needed.)

Draw a diagram of your crystal (or one of the smaller crystals if you do not have one large one.)

Mass of final alum crystal(s) _____ g