MICROGRAVITY

# Microscopic Observation of Crystal Growth

## Objective:

- To observe crystal nucleation and growth rate during directional solidification .

## Science Standards:

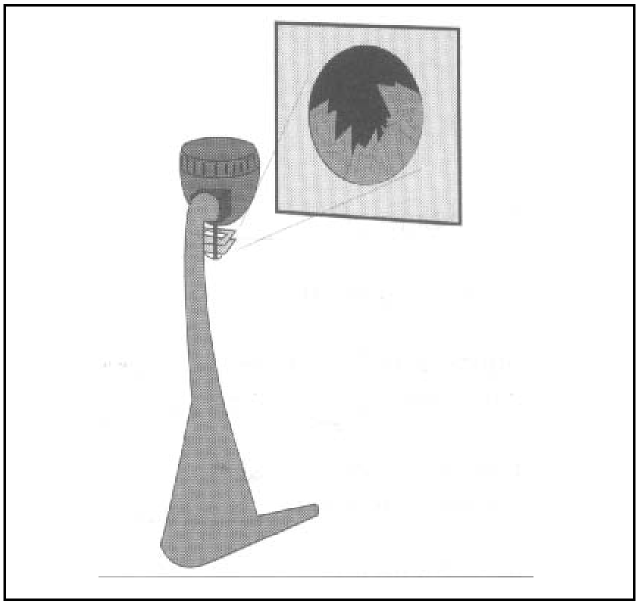
Science as Inquiry
Physical Science
- position and motion of objects
- properties of objects and materials
Unifying Concepts and Processes
Change, Constancy, & Measurement

## Science Process Skills:

Observing
Communicating
Investigating

## Activity Management

The mannite part of this activity should be done as a demonstration, using a microprojector or microscope with a television system. It is necessary to heat a small quantity of crystalline mannite on a glass slide to 168°C and observe its recrystallization under magnification. The instructions call for melting the mannite twice and causing it to cool at different rates. It is better to prepare separate samples so they can be compared to each other. Th~ slide that is cooled slowly can easily be observed under magnification as crystallizes. You may not have time to observe the rapidly chilled sample properly before crystallization is complete. The end result, however, will be quite apparent under magnification. If students will be conducting the second part of theactivity, it is suggested that you prepare several sets of mannite slides so they may be



A microprojector is used to observe crystal growth.

**MATERIALS AND TOOLS**

Bismarck brown Y
Mannite (d-mannitol) $HOCH_2(CHOH)_4CH_20H$
Salol (Phenyl salicylate) $C_{13}H_{10}O_3$
Microprojector
Student microscopes (instead of a microprojector)
Glass microscope slides with cover glass
Ceramic bread-and-butter plate
Refrigerator
Hot plate or desktop coffee cup warmer
Forceps
Dissecting needle
Spatula
Eye protection

distributed for individual observations. The salol observations are suitable for a demonstration, but because of the lower melting temperature (48°C), it is much safer for students to work with that the mannite. A desktop coffee cup warmer is sufficient for melting the salol on a glass slide. Because of the recess of the warmer's plate, it is best to set several large metal washers on the plate to raise its surface. The washers will conduct the heat to the slide and make it easier to pick up the heated slide with forceps. Point out to the students that they should be careful when heating the salol because overheating will cause excessive evaporation and chemical odors, and will increase the time it takes for the material to cool enough for crystallization to occur. The slide should be removed from the hot plate just as it starts melting. The glass slide will retain enough heat to complete the melting process.

Only a very small amount of bismarck brown is needed for the last part of the activity with salol. Only a few dozen grains are needed. Usually just touching the spatula to the chemical causes enough particles to cling to it. Gently tap the spatula held over the melted salol to transfer the particles. It will be easier to do this if the salol slide is placed over a sheet of white paper. This will make it easier to see that the particles have landed in the salol.

If students are permitted to do individual studies, go over the procedures while demonstrating crystallization with the d-mannitol. Have students practice sketching the crystallized mannitol samples before they try sketching the salol.

Refer to the chemical notes below for safety precautions required for this activity.

## Notes On Chemicals Used:

### Bismarok Brown Y

Bismarck brown is a stain used to dye bone specimens for microscope slides. Because bismarck brown is a stain, avoid getting it on your fingers. Bismarck brown is water soluble.

### Mannite (d-mannitol)

$HOCH_2(CHOH)_4CH_20H$ Mannite has a melting point of approximately 168°C. It may be harmful if inhaled or swallowed. *Wear eye protection and gloves when handling this chemical. Conduct the experiment in a well-ventilated area.*

### Salol (phenyl salicylate)

$C_{13}H_{10}O_3$

It has a melting point of 43° C. It may irritate eyes. *Wear eye protection.*

## Procedure: Observations of Mannite

1. Place a small amount of mannite on a microscope slide and place the slide on a hot plate. Raise the temperature of the hot plate until the mannite melts. Be careful not to touch the hot plate or heated slide. Handle the slide with forceps.
2. After melting, cover the mannite with a cover glass and place the slide on a ceramic bread-and-butter plate that has been chilled in a refrigerator. Permit the liquid mannite to crystallize.
3. Observe the sample with a microprojector. Note the size, shape, number, and boundaries of the crystals.
4. Prepare a second slide, but place it immediately on the microprojector stage. Permit the mannite to cool slowly. Again observe the size, shape, and boundaries of the crystals. Mark and save the two slides for comparison using student microscopes. Forty power is sufficient for comparison. Have the students make sketches of the crystals on the two slides and label them by cooling rate.

## Observations of Salol

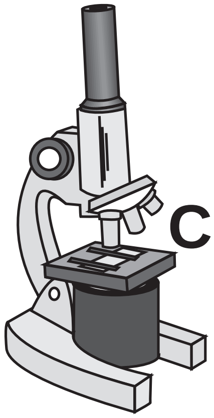
5. Repeat the procedure for mannite (steps 1–4) with the salol, but do not use glass cover slips. Use a desktop coffee cup warmer to melt the salol. It may be necessary to add a seed crystal to the liquid on each slide to start the crystallization. Use a spatula to carry the seed to the salol. If the seed melts, wait a moment and try again when the liquid is a bit cooler. (If the microprojector you use does not have heat filters, the heat from the lamp may remelt the salol before crystallization is completed.)
6. Prepare a new salol slide and place it on the microprojector stage. Drop a tiny seed crystal into the melt and observe the solid-liquid interface.
7. Remelt the salol on the slide and sprinkle a tiny amount of bismarck brown on the melt. Drop a seed crystal into the melt and observe the motion of the bismarck brown granules. The granules will make the movements of the liquid visible. Pay close attention to the granules near the growing edges and points of the salol crystals.

## Assessment:

Collect the student data sheets.

## Extensions:

1. Design a crystal-growing experiment that could be flown in space. The experiment should be self-contained and the only astronaut involvement that of turning a switch on and off.
2. Design a crystal-growing experiment for spaceflight that requires astronaut observations and interpretations.
3. Research previous crystal-growing experiments in space and some of the potential benefits researchers expect from space-grown crystals.



# Crystal Growth

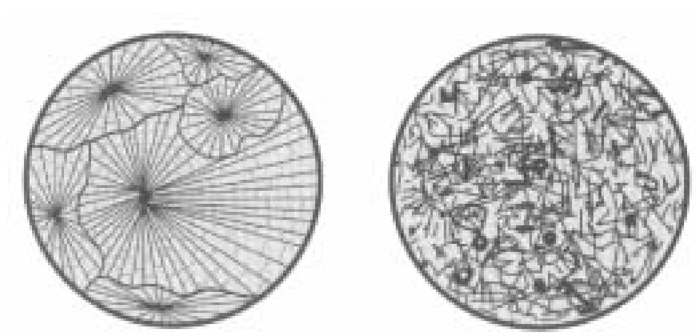
Directional solidification refers to a process by which a liquid is transformed (by freezing) into a solid through the application of a temperature gradient (a temperature difference over a specified distance such as 1 0oC/cm) in which heat is removed in one direction. The heat travels down the temperature gradient from hot to cold. A container of liquid will turn to a solid in the direction the temperature is lowered. If this liquid has a solute (something dissolved in the liquid) present, typically some of the solute will be rejected into the liquid ahead of the liquid/solid interface. However, not all of the solute can be contained in the solid as it forms; the remaining solute is pushed back into the liquid near the interface. This phenomenon has many important consequences for the solid including how much of the solute eventually ends up in the solid. The concentration of solute in the solid can control the electrical properties of semiconductors and the mechanical and corrosion properties of metals. As a result, solute rejection is studied extensively in solidification experiments.

The rejected material tends to build up at the interface (in the liquid) to form a layer rich in solute. This experiment demonstrates what happens when the growth rate is too fast and solute in the enriched layer is trapped.

Fluid flow in the melt can also affect the buildup of this enriched layer. On Earth, fluids that expand become less dense. This causes a vertical flow of liquid which will interfere with the enriched layer next to the growing solid. In space, by avoiding this fluid flow, a more uniform enriched layer will be achieved. This, in turn, can improve the uniformity with which the solute is incorporated into the growing crystal.

**Sample Microscope Sketches**

Mannite Crystallization



Slow Cooling    Fast Cooling

# Microscopic Observation of Crystals

Name: ______________________________

1. Study the mannite crystallization slides. Sketch what you observe in the two circles below. Identify the cooling rate for each slide and the magnification you used for your observatlons.

## Mannite (d-Mannitol)

Cooling Rate: ______________________

Magnification: ______________________

Cooling Rate: ______________________

Magnification: ______________________

Describe below the difference between the two mannite samples.

How can you explain these differences?

2. Prepare the salol samples according to instructions provided by your teacher. Remember to wear eye protection as you handle the chemical. Study the salol crystallization slides. Sketch what you observe in the two circles below. Identify the cooling rate for each slide and the magnification you used for your observations.

## Salol (Phenyl Salicylate)

Cooling Rate: ______________________

Magnification: ______________________

Cooling Rate: ______________________

Magnification: ______________________

Describe below the difference between the two salol samples.

How can you explain these differences?

3. Prepare a third salol sample according to instructions provided by your teacher. Remember to wear eye protection as you handle the chemical. Adjust the sample on the microscope stage so you can observe the interface between the growing crystals and the melted chemicals. In particular, look at what happens to the bismarck brown particles as the growing crystals contact them. Sketch what you observe in the circle below.

Cooling Rate: **Slow**

Magnification: ______________________

What happens to the resulting crystals when impurities (bismarck brown) exist in the melt?

What caused the circulation patterns of the liquid around the growing crystal faces? Do you think these circulation patterns affect the atomic arrangements of the crystals? How?

How do you think the growth of the crystals would be affected by growing them in microgravity?