At-Home Chemistry Experiment

Crystallization of Borax

Objective

The objective of this at-home chemistry experiment is to qualitatively introduce the purifying technique of crystallization.

Introduction

Chemists perform chemical reactions to change one compound into another. Sometimes when multiple products are formed, the chemist may want to separate one compound from the others. This can be done through a process called crystallization (or recrystallization), where a mixture of compounds can be dissolved in a minimum amount of hot solvent and then cooled. As the solution cools, one substance crystalizes (becomes crystals) and can be removed from the rest of the liquid. The other compound(s) remain in solution. The key aspect to crystallization is to choose a solvent where the desired compound will readily dissolve at elevated temperatures, but has reduced solubility at lower temperatures.

It is impossible to obtain a 100% recovery of the desired compound after a crystallization. Sometimes there is experimental loss, the original sample was not 100% pure, or some of the product remains solubilized in the crystallizing solvent even at 0°C. In this latter situation, the liquid remaining after crystallization has taken place is described as the **mother liquor**. Occasionally, it is worth isolating a second crop of crystals from the mother liquor, especially if you have performed a synthesis requiring many hours of work and the amount of product formed is relatively small. This can be accomplished by heating the mother liquor to evaporate some of the solvent and then cooling the resulting solution to induce a second crystallization. However, the purity of the second crop will not be as high as the first crop because the concentration of the impurities will be greater in the mother liquor after some of the solvent has been evaporated. Some of the impurities tend to crystallize with the desired product.

You should be aware that not all crystallizations occur in the same way. Crystals have many different shapes and sizes, and the amount of mother liquor visible at the end of the crystallization may vary significantly.

Materials

- Water
- Ice cubes
- Borax (household cleaner)
- Large bowl
- Two identical glass containers (jars or large drinking glasses)
- Measuring tablespoon
- Cooking pot
- Two pipe cleaners (any color except dark red)
- Two pencils (or chop sticks, dowels, etc.)
- Waterproof cover (aluminum foil, saran wrap or Ziplock)

***Caution*:** Borax is harmful if swallowed, inhaled or has come in contact with the eyes. On rare occasion, touching it can result in rashes.

Figure 1: *Materials (pipe cleaners are yellow in this image, but any color except dark red works)*

Procedure

1. Fill the large bowl half full with ice cubes and then add water until the bowl is about three quarters full.
2. Cut two pipe cleaners at least as long as the height of the glass containers. Tie the end of one pipe cleaner around a pencil. Tie the end of the second pipe cleaner around the other pencil. Adjust the pipe cleaners' lengths so that when the pencil is laid across the top of one of the jars, the end of the pipe cleaner hangs down to just above the bottom of the jar. Make sure the pipe cleaners are equal in length.

Figure 2: *Pipe cleaner tied to chop stick over an empty glass*

3. Set the pipe cleaners tied to the pencils aside so they are no longer in the jars.
4. Fill a cooking pot with enough water to fill both jars nearly full. Then bring that water to a boil on the stove. Once the water is boiling, turn the burner off so that the water stops boiling. (Because Borax is harmful if inhaled or contacts eyes it is advised to not dissolve it with boiling water. On the other hand, you want the water to stay as hot as possible).
5. Add one tablespoon of Borax to the water and stir until it dissolves. Continue to add one tablespoon of Borax at a time until no more dissolves. You should have a small amount of solid Borax at the bottom to ensure the solution is saturated when very hot. Don't add so much Borax that the water becomes cloudy. You may need to heat the water again if dissolving the Borax takes more than 3 minutes.

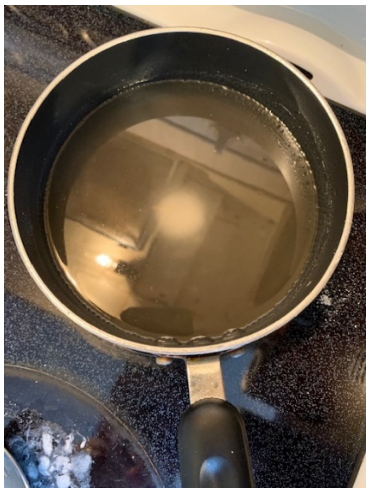

Figure 3: *Dissolving Borax in hottest water possible until saturation*

6. Carefully pour equal amounts of the very hot saturated Borax *solution* into the two jars. Each jar should be about three fourths full. It is important to avoid losing time, as the saturated solution must be as hot as possible when you add the pipe cleaners.
7. Lay a pencil across the top of each jar so that the pipe cleaner hangs down into the saturated solution, not touching the walls or bottom. Shaking them a little to get rid of any air bubbles. The part closest to the bottom usually ends up with more crystals than the part hanging near the surface of the solution.

8. Cover the top of the jars loosely with plastic wrap, aluminum foil or other material.
9. Leave one jar (referred from now on as jar A) undisturbed on a countertop or table at room temperature.
10. Place the second jar (referred from now on as jar B) in the bowl full of ice that you previously prepared. If needed, adjust the water level in the bowl so that the water reaches at least three fourths the way up the jar, but is not so high that it goes into the jar.

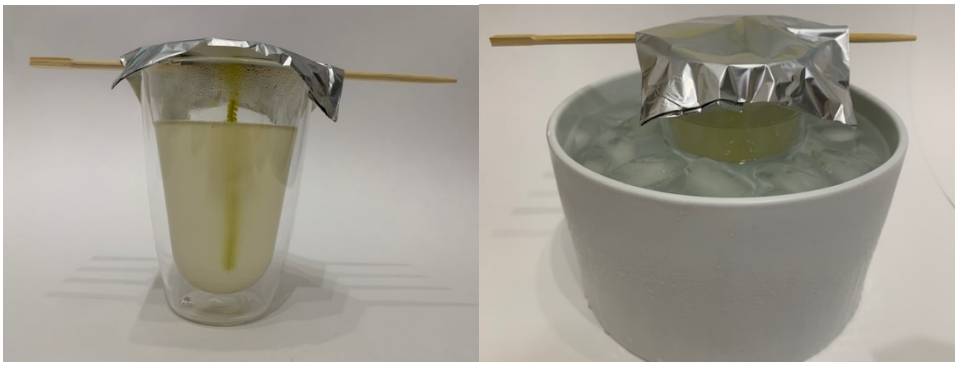

Figure 4: *Jar A (left) and Jar B (right) to be left undisturbed*

11. Do not disturb the jars for at least five hours. Check the bowl of ice regularly and add ice if it has melted.
12. Check on the jars about once an hour to see how the crystals are forming. It may be difficult to observe the jar in the bowl—try looking at the pipe cleaner through the plastic wrap cover.
13. After at least five hours carefully remove the pencils and observe the crystals on the pipe cleaners.
14. Take a picture of the crystals from jar A (identified) and B (identified) and include them in your lab report.
15. Put the pipe cleaner of jar A back in the jar for at least 24 hours.
16. Take a picture of the crystals from jar A.
17. Very carefully clean all the materials. Do not leave any trace of Borax on your kitchen utensils.

Name _____ Section _____

Date _____

Crystallization of Borax

Data

1. How do the number, size, and shape of crystals on each pipe cleaner compare with one another? Answer using the following table.

Crystal Characteristics (after 5 hours)	**From Jar A**	**From Jar B**
Number (total on pipe cleaner)		
Size (of each crystal)		
Shape (of each crystal)		

2. How does allowing the Borax mixture to cool at different temperatures affect crystal formation?

3. Do crystals form in one jar before the other?

4. How do the results change if you grow your crystals for a longer period of time?

Post-Lab Questions

1. Why are crystalline solids typically purer than solids that are not crystalline?

2. What can you do if you do not see crystals forming in your cooled solution?

3. Benzyl alcohol solvent (bp 205°C) was selected by a student to crystallize fluorenol (mp 153°C–154°C) because the solubility characteristics of this solvent are appropriate. However, this solvent is not a good choice. Explain why.

4. A student performs a crystallization on an impure sample of biphenyl. The sample weighs 0.4 g and contains about 5% impurity. Based on his knowledge of solubility, the student decides to use benzene as the solvent. After crystallization, the crystals are dried and the final mass is found to be 0.01 g. Assume that all steps in the crystallization were performed correctly, there were no spills, and the student lost very little solid on any glassware or in any of the transfers during the experiment. Why is the recovery so low?

References

- Experiment 2 Crystallization in *Introduction to Basic Laboratory Techniques - small scale approach*, 2nd ed., by D.L.Pavia, G.M. Lampman, G.S. Kriz and R.G. Engel, Thomson Brooks/Cole, **2005**, page 16-24.
- Solubility Science: How to Grow the Best Crystals, By Science Buddies, May 2, 2013 https://www.scientificamerican.com/article/bring-science-home-crystals/

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