8.7 – Guide to Growing a Single Crystal

Overview:

As you may discover, growing single crystals takes patience as well as an artful hand. It also can be very sensitive to temperature and minor disturbances. Therefore, you will be encouraged to try several different temperatures, using otherwise identical conditions, and to always find a quiet undisturbed location to promote crystal growth. Here are some tried and true tips to get you started.

Tips - Option #1

• Sometimes nice crystals will grow simply by cooling your solution. You can also try supersaturating a solution by heating it until all of your material dissolves, then allowing it to cool down.

Option #2

1) Find a solvent that your compound is soluble in, and make a saturated solution.

2) If necessary, perform a filtration to remove insoluble impurities. For such small scales, a good filter can be made by plugging a disposable Pasteur pipet with glass wool (or even a bit of Kimwipe), then filling (about an inch) with a filtering aid such as Celite. Moisten the Celite with fresh solvent, then filter your solution by forcing it through the pipet with a pipet bulb.

3) Find another solvent, in which your compound is NOT soluble (or only slightly soluble), and which is miscible and less dense than the first solvent.

4) Carefully layer the second solvent onto your saturated solution in a small vial. You may see some turbidity at the interface. Your crystals should grow along this interface.

Option #3

• Another option is to place your saturated solution in a small vial that sits inside another larger vial. Add the second solvent to this outer vial and cap. The second solvent should slowly diffuse into the saturated solution, and your crystals should appear! To slow the process even further, place the diffusion set-up in the fridge.

Solvent systems to try:

CH₂Cl₂/ether or pentane toluene/ether or pentane CHCl₃/*n*-heptane water/methanol THF/ether or pentane 5.301 Chemistry Laboratory Techniques January IAP 2012

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