GROWING CRYSTALS

To grow crystals suitable for single-crystal X-ray diffraction there are three easy techniques which will work in many cases. For samples that are not air-sensitive, solvent evaporation is an excellent method. For air-sensitive compounds, cooling is a good technique. Anti-solvent diffusion is suitable for any sample. In all cases it is desirable to grow the crystals **as slowly as possible**, and **quality is far more important than quantity.** The sample should be as pure as possible before you begin.

Solvent Evaporation

Place a small amount of the sample in a vial, dissolve in a minimum amount of solvent, cover the opening with lab-film and pierce **once** with a narrow needle. Leave the sample in a place that is free from vibrations and not in direct sunlight or next to a source of heat. **Do not keep picking it up.** As the solvent slowly evaporates, crystals should form. **Do not let all the solvent evaporate or your crystals may be of poor quality.** If no crystals form, even when all the solvent has evaporated, **try another solvent.**

Cooling

Concentrate your solution until the sample precipitates. Check with a hand lens or under the microscope to see if you have formed small crystals. If you have amorphous material, **try another solvent.** If you have crystals, redissolve them by heating the solution, and allow the solution to cool slowly suspended in a Dewar of hot water. If no crystals form, place your saturated solution in the fridge and leave it for at least a week. **Do not keep picking it up.** If no crystals form, even after several weeks, place the solution in the freezer and wait again (assuming the solvent does not freeze at this temperature). If this does not work, try further concentrating your solution, and repeat. If this method still does not produce crystals, **try another solvent.**

Anti-solvent Diffusion

Place a small amount of the sample in a small vial and dissolve in a minimum amount of solvent. Add a small volume of a volatile solvent, in which your sample is poorly soluble, to a large sample vial. **Carefully** place the small vial inside the large vial, taking care not to spill the solution, and stopper the large vial. Leave the sample in a place that is free from vibrations and not in direct sunlight or next to a source of heat. **Do not keep picking it up.** The volatile anti-solvent will slowly evaporate and diffuse into the solution, lowering the solubility of the sample. If no crystals form, even when all the anti-solvent has diffused into the solvent, **try another pair of solvents.**

Solvents

If you cannot get crystals using these methods, do not give up until you have tried every solvent you can. We routinely get excellent crystals grown from water, methanol, ethanol, acetonitrile, chloroform, DCM, toluene, ether and THF, to name only the most common. Persevere. If you still have no success, take a look at the references on our web page, www.ncl.ac.uk/xraycry/crystals.htm, or come and see us.