

Single Crystal X-Ray Diffraction - A Guide to Crystal Growing

Remember:

1. PURIFY your compound as much as is practically possible. Also filter the solution you will grow the crystals from, to remove any dust etc., that could cause the crystals to be small and grow in clumps - in which case, start again.
 2. Be as certain as possible that you have the RIGHT STUFF - also TEST THE CRYSTALS when they appear - remove a few to see if they lose solvent and examine them by infrared, nmr etc. It has been known many times that the crystals you get are not the compound you expect them to be!
 3. Grow the crystals SLOWLY and with the MINIMUM of vibration - a fridge is a good place. You need crystals bigger than 0.2mm in all 3 dimensions.
 4. Grow the crystals in a vessel from which they can be EASILY REMOVED such as a small Schlenk tube, not a round bottomed flask!
 5. PERSEVERE - you may need to try several methods and different combinations of solvent, and concentration, before diffraction-quality crystals are obtained.
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Methods:

1. **Solvent diffusion** - here you use one solvent in which your compound is soluble and a second in which it is not. The two solvents must be miscible however. CH₂Cl₂/hexane is a common combination. Nmr tubes have been used successfully as containers.
 - a. Dissolve your compound. Filter if necessary, e.g. through a pipette with a glass wool plug.
 - b. Carefully layer the second solvent, in which your compound is not soluble, on top of the pure solution of your compound (if the second solvent is lighter). Let the second solution dribble down the side of the tube by syringing it down the side slowly. If the lower layer is disturbed a lot, powder will crash out - avoid this. Don't worry if it's cloudy at the interface - that's quite usual.
 - c. Keep the tube upright and either clamp it or put it in a test tube rack in the fridge.
 - d. It usually takes between 3 hrs and 3 days for crystals to appear - depending on concentration, rate of diffusion etc.
2. **Vapour Diffusion**
 - a. A solution of your compound is put in a small vial (which can have a loose bung in it). This vial is placed in a larger vial or beaker containing the precipitating solvent which is then sealed.
 - b. Diffusion is slower so crystal growth will take longer.
3. **Slow Evaporation**

- a. Leave a nmr sample in a rack on the bench - the solvent will evaporate slowly through the top. or ...
- b. Have a vial of solution covered in NESCO film.