

Chemistry 832: Solid State Structural Methods, Spring 2000

Laboratory Exercise #L3, Systematic Approaches to Growing Single Crystals

Part I. Solubility of Organic Molecules. Before one can determine the optimum conditions for growing single crystals, one must determine their solubilities.

From the organic store room, select two compounds per group (having a range of structures, charges, and H-bonding groups). Obtain bottles of different solvents (water, dilute HCl, dilute NaOH, MeOH, ⁱPrOH, Acetone, EtOAc, THF, CH₂Cl₂, Toluene, and Hexanes as a representative range). Evaluate the solubility of your compounds in each of these solvents. Determine the identities of the “frontier solvents” (i.e. those where the solvent is just moderately soluble and only slightly soluble as opposed to either very soluble or insoluble).

Part II. Ideal Precipitation conditions. Working in the “solvent space” around the frontier solvents, find and **report** the boundary conditions for precipitation.

Part III. Single Crystal Growth. Working near the boundary conditions for precipitation, set up a series of single crystal growth trials as:

- a) layered solvents,
- b) solvent evaporation,
- c) solvent cooling.