Recrystallization

Environment, Nature

Most important method for the purification of organic solids ; Separation of compounds based on differences in solubility between the compound of interest and its contaminants ; Basic technique: 1 . Dissolve impure sample in an " appropriate" hot solvent Part A: Choosing a Solvent Part B: Purification of Phonetic 2. Cool solution slowly to induce crystal growth 3. Filter resulting mixture to isolate crystals Reading: Mooring, Hammond & Chats Chi. 15 pigs 183-197 Chi. 0 pigs 104-113 Chi. 14 pigs 174-182 ; Scale: 5-10 MGM coverer based research - a new material prepared in a lab 1, 000 keg + commercial applications - sugar refining, synthesis of pharmaceutical agents, etc. ; Molecular selection pure substance aggregation begins - based on size, shape, & functionality molecules deposit on growing surface in orderly manner, excluding those of different size of shape if deposition occurs too quickly, an impure substance can result crystal defects incorporated impurities Rationalization Steps 1 .

Choose an appropriate solvent - compound (solid) should be soluble when solvent is hot - compound should be insoluble when solvent is cold may require some trial & error 2. Dissolve impure compound in the minimum amount of hot solvent - too much solvent & compound may not come out when cool 3. Decolonize solution if needed with activated charcoal (Norris) - skip this step if no/ few colored impurities are present - be sure your compound is not supposed to be colored! 4. Filter off any insoluble materials - insoluble impurities and/or activated charcoal - done while solution is hot 5.

Slowly cool the resulting solution to induce crystallization temperature, then in an ice bath - if no crystals form: scratch flask with glass rod or ad a seed crystal to the solution - first cool to room - these methods provide a

nucleation point for crystallization 6. Collect and wash the crystals - collection typically by filtration (large quantities) - for small quantities can remove solvent with a pipette - wash crystals with a small amount of ice cold solvent - filtrate (" mother liquor") can be concentrated to get " 2nd crop" 7.

Dry the crystals thoroughly - apply vacuum & continue suction until crystals are dry - dry crystals further under vacuum in a side arm test tube - can also press solids between two pieces of filter paper Factors that Influence Melting Point ; Melting Point: point of equilibrium between crystalline & liquid states point at which a crystal goes from solid to liquid ; Temperature at which a compound melts is typically a range Factors that influence melting point temperatures: 1.

Intermolecular forces start: temperature at which first drop of liquid forms a. Van deer Walls interactions very weak end: temperature at which all solid has turned to liquid b. Dipole-dipole interactions e. G. 82-ICC ; Why do we care about melting point? 1. Can be used to help identify substances ampere pm of unknown substance with that of known substance result from popularization of bonds c. Hydrogen bonding compounds having O-H or N-H bonds d. Ionic forces take a " mixed" melting point 2. Is an indicator of purity pure samples have narrow pm ranges (0. - 2 co) impure samples melt over a broader range (> ICC) & are depressed very strong 2. Shape ; strength & nature of intermolecular interactions impact melting point temperature Melting Point as an Indicator of Purity ; In a pure sample, all surface molecules need the same energy to escape. Leads to a narrow melting point range. For melting to occur, surface molecules must have enough energy to

break free. Stronger intermolecular interactions = more energy required for molecules to " escape".

Translates to a higher pm. ; In an impure sample, intermolecular forces are disrupted in the region of the impurity. Less energy thus required for surface molecules to break free. Crystal begins to liquefy at a lower temperature ; structural features that influence how molecules pack together impact melting point temperature symmetrical compounds typically have higher melting points features that disrupt crystal lattice lower melting point Next Week Experiment 2: Rationalization & Melting Point A.

Choosing a Solvent identify an appropriate solvent for the rationalization of phonetic B. Purification of Phonetic purify the impure solid evaluate success by melting point & TTL Come prepared. You will get only one sample of phonetic DUE: Thin Layer Chromatography Lab Report (expo 1) Lab Reports are due at the beginning of your regular lab session ; Still some regions without impurities. Additional energy required for surface molecules in these regions to break free. End result is that melting point range is broadened

Experimental Details - Part A - prepare a hot water bath begin heating as soon as you arrive in lab - put a spatula tip of the impure compound into a small test tube no need to get an accurate mass - to the 1st tube, add 0. 5-1 ml of one of the solvents to be tested 10-20 drops (1 drop = ca. 0. Ml) - evaluate behavior: upon addition of solvent, when hot, when cold if compound dissolves upon addition, no need to go further if solids remain, heat in hot water bath to near boiling