OCHEM LAB REVIEW

Lab 1: Extraction

- Polarity
    - Net dipole caused by polar bonds… type of bond is characterized by differences in electronegativity
        - If dif is less than 0.2, then nonpolar
        - If dif is between 0.2 and 1.5, then polar (yes C-H is slightly polar)
        - If dif is greater than 1.5, then ionic
- Hydrogen bonding: strong non-covalent interactions
    - Example of intermolecular attractions aka Van der Waals forces
    - Any molecule with N, O, F are polar enough to accept hydrogen bonds,
        - N-H and O-H are both donors and acceptors, =O and F and 3 amines are often only acceptors (no polar hydrogen)
- Polarity is important to solvation
    - Like polarities will associate with each other only
        - Nonpolar: alkanes, CCl_4
        - Slightly polar: ether, benzene, chloroform
        - Polar: alcohols, water, acetone, any N- compound
    - Miscibility: ability for two liquids/solvents to mix completely
        - If miscible, should see only one layer: immiscible solvents will form layers based off density
- Extraction
    - Separation of 2+ compounds based off their differences in solubility and polarity
    - Solid-liquid extraction:
        - Adding solvent to a solid and extracting only the soluble compounds… think coffee
    - Liquid-liquid extraction:
        - 2 compounds both dissolved in a solvent; must separate based off polarities.
        - Use a separatory funnel: swirl and vent to relieve any heat/pressure that may build because most of the time acid is added
        - Partition coefficient: K_c= [A organic]/[A aq.]
            - This is important because ratio is a constant; therefore doing two extractions is more favorable than one big one because the ratio extracted is proportional to the amount of org. solvent added and aq. solvent present.
    - Chemically active extraction
        - Manipulating polarity of a compound in order to separate it
            - Good to isolate amines, carboxylic acids; both are very active specimen
        - To isolate a carboxylic acid (or any acid)
            - Add a strong base; this deprotonates the base
                - Alkoxide (conj. Base) now more soluble in water, so can extract
            - Add strong acid back to alkoxide to protonate; separate using organic compound to isolate

        - To isolate an amine (basic)
            - Protonate with a strong acid; making compound more soluble in water
            - Deprotonate amine in aq solution to make soluble in org. layer again and extract
        - To extract a nonpolar: do both acidic and basic extractions and remaining original compound is purely nonpolar
- Important New techniques:
    - Steam bath: method of heating flammable organic solvents safely
        - Use some boiling stones to prevent bumping
            - Occurs when large bubbles form in solution and burst out, which can be messy
            - Boiling stones provide surface area for molecules to vaporize off of safely
        - Never use a full steam bath, this creates splashing
    - Vacuum Filtration:
        - Used to separate a solid from a liquid
        - Stabilize the filter flask so it doesn't fall over
        - Apply vacuum only after seal is made; terminate vacuum after breaking seal only
            - Terminating vacuum with seal present will cause rush of air to blow sample all over the place
    - Separatory Funnel for Extraction:
        - As explained above, swirl and vent several times to avoid build-up of heat/pressure
        - Always vent away from people and into hood
        - Analyze layers once allowed to settle; remember organic compounds are less dense and often settle atop aqueous layer

Lab 2: Recrystallization

- Basic recrystallization schematic
    - Collision: solvent interacts with solid
    - Dissociation: solvent breaks off part of solid bit by bit
    - Solvation: solvent completely dissolves solute
    - Recrystallization: allowing for solute to precipitate
- Free energy change dictates crystallization and solvation
    - Solvation is entropically favored: increases disorder
        - Therefore solvation can be favored with increase in temperature
    - Recrystallization is enthalpically favored; forming bonds releases heat
        - Therefore favored at low temperatures
- Recrystallization process
    - Taking a crude solid, dissolving it, filtering it, and then slowly cooling it to precipitate out a pure compound
    - Solvent choice

        - Compound of interest should only be soluble at high temperatures; called a high temperature coefficient
        - Boiling point of solvent needs to be less than melting point of solid so that when added, the solid doesn't melt instead of dissolving (called oiling out)
        - Should be inert, volatile, and inexpensive
            - Could mix solvents to create more desirable solvent
        - Impurities must either be completely insoluble or completely soluble in solvent
            - Allows for them to be separated either at filtration step or at recrystallization step
    - Note: adding charcoal will absorb any colored impurities and can easily be filtered away
    - Filtering step:
        - Must keep everything hot to maximize solubility, therefore perform hot gravity filtration over steam bath
    - Recrystallization step:
        - Need to spark nucleation:
            - Spontaneous process where atoms coagulate into crystals
            - Can be facilitated by scratching glass (surface area) or adding a seed of desired compound (surface area)
            - Icing solution will further favor recrystallization
        - Crystal Lattice Theory
            - Forming crystal lattice will only accept like molecules assuming process is slow
                - Fast nucleation = impure crystals due to impurities incorporated accidentally
- Technique review: N/A

Lab 3: Melting point/Boiling point and Distillation

- Physical properties: those that can be observed/measured without changing the substances identity
    - *Intrinsic* properties refer to those *independent* of amount of substance present, like melting point, solubility, polarity, density etc.
    - *Extrinsic* properties refer to those based on external factors, like mass, volume, entropy change
- Physical changes or phase changes occur when compound goes from solid to liquid to gas or any step in between; no bonds or broken and no change in chemical composition