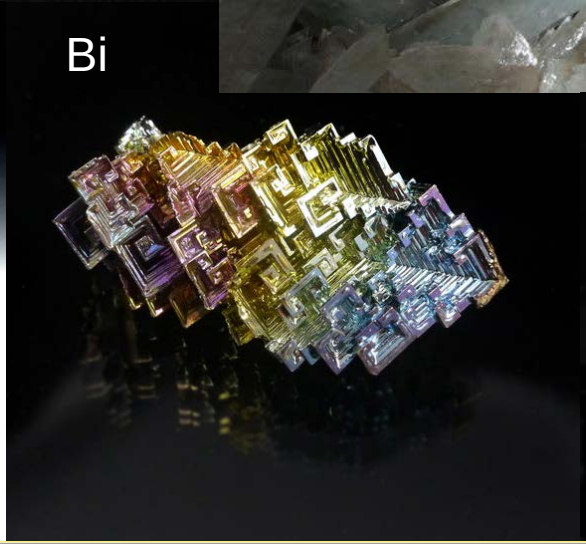
How to: Crystallization



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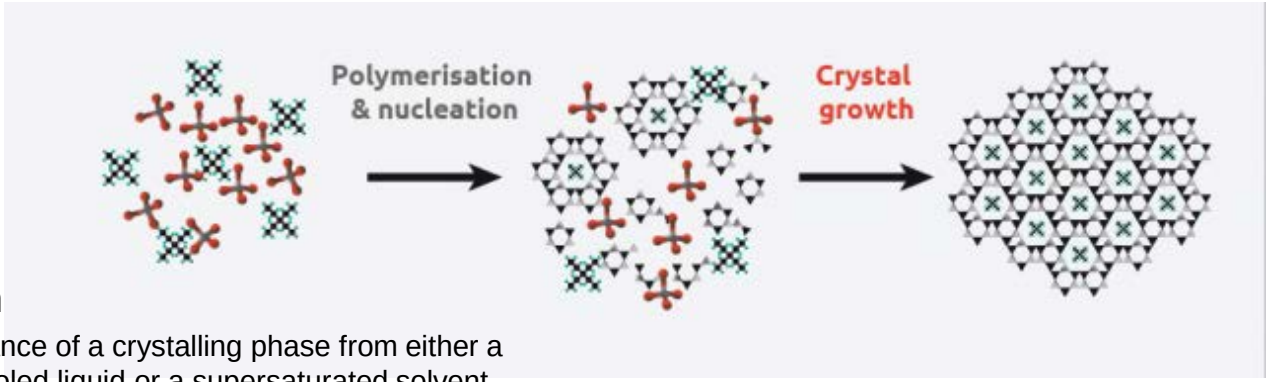
What is crystallization and how does it occur?



- Nucleation
  - Appearance of a crystalling phase from either a supercooled liquid or a supersaturated solvent

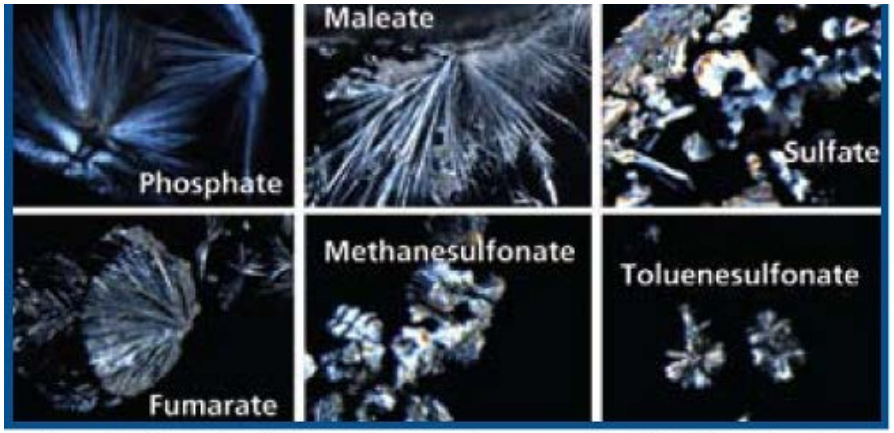
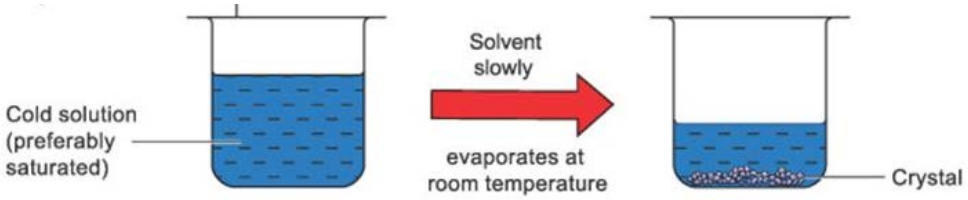


Figure 2: Different salt forms of a research compound exhibit different habits.

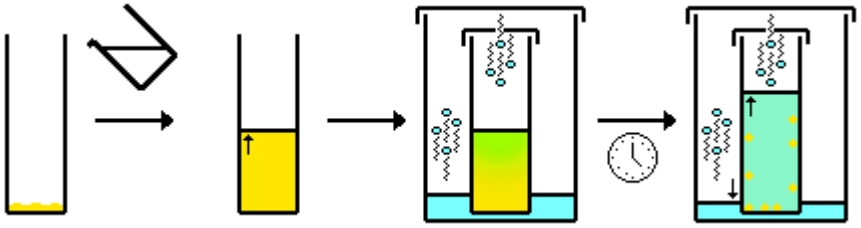
- Process by which a solid forms, where the atoms/molecules are highly organized into a crystal structure
- Extremely useful in research and industry as a means of purification

Methods of crystallization

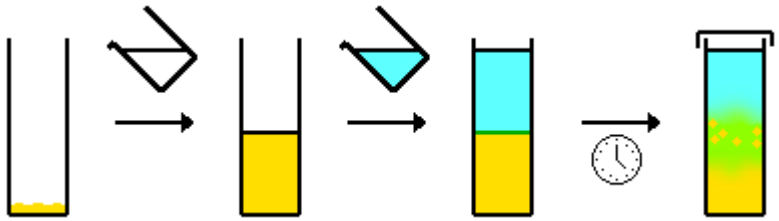


Evaporation

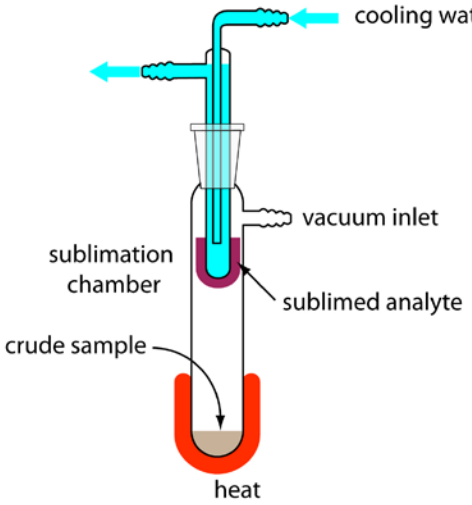
Concentrate & Cool



Vapor diffusion

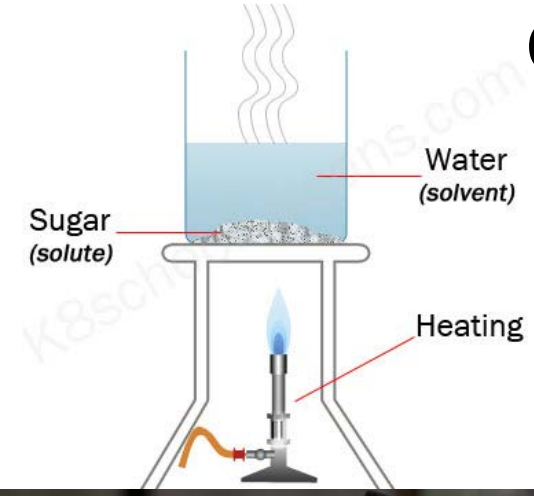


Liquid/liquid diffusion



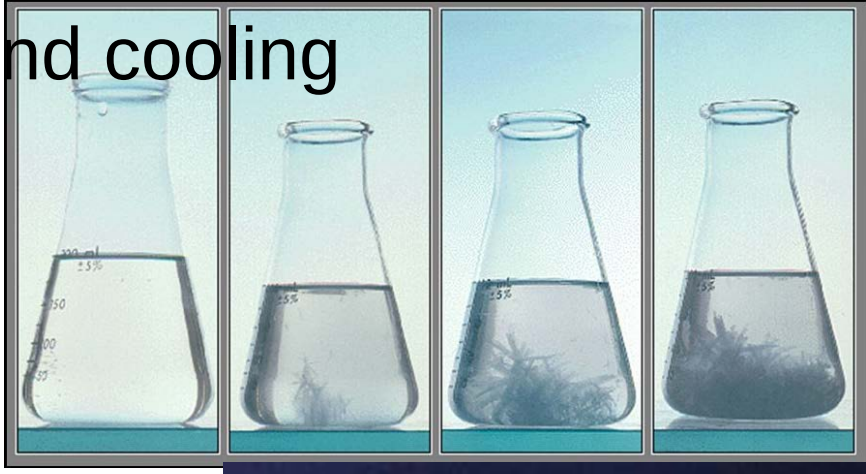
Sublimation

Concentrating and cooling



Saturate solution

Cool hot to RT

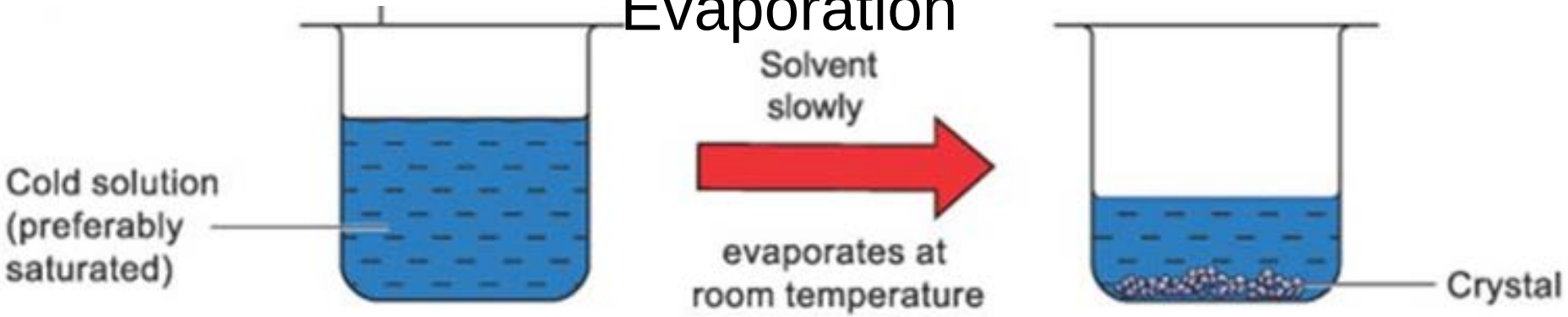


Pump down

Cool in freezer/ ice bath

- Simplest method but pretty effective
  - Crystal growth relies on the cooling of a saturated solution of the compound to be crystallized
  - Any solvent is fair game so long as the compound is soluble in it
    - With the exception of water and benzene if planning on using a freezer

Evaporation

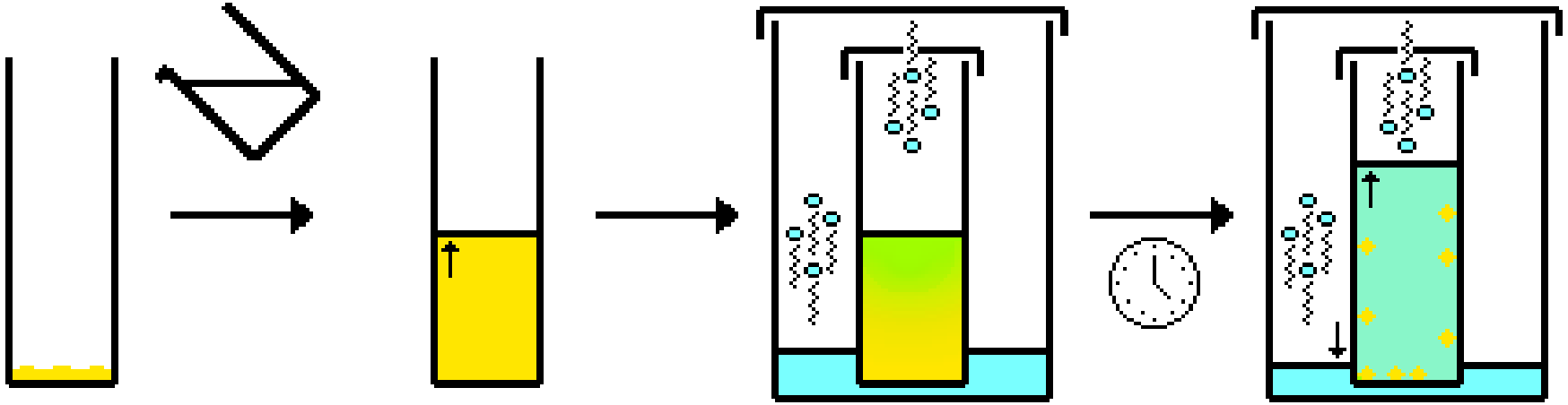


https://www.youtube.com/watch?v=1Ou1bIDn1xE

Look up how to insert youtube video above

- Most common crystal growth method for air stable compounds
  - Crystallization depends on simply evaporating solvent from the solution until saturation is reached and crystals form
    - Not the best and often leads to ugly crystal (poor diffraction)
  - Can slow down crystal growth by placing in freezer

Vapor diffusion

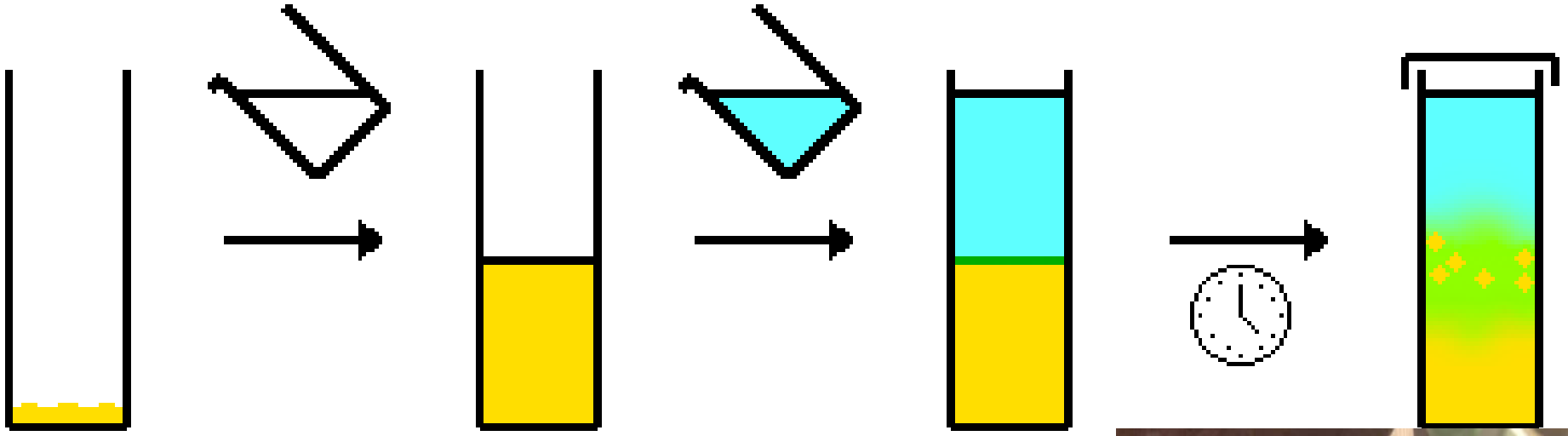


Lower vapor pressure (inside)

Higher vapor pressure (outside)

- Usually most successful for beautiful crystals
  - Two vials are needed (one fitting inside the other)
  - Inner vial
    - Contains compound to be crystallized dissolved in small quantity of solvent
  - Outer vial (capped)
    - Contains volatile solvent in which compound is insoluble

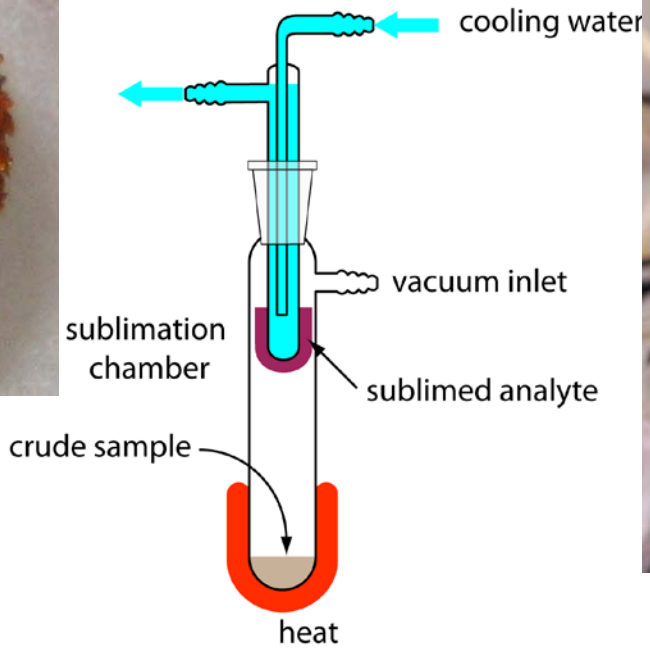
Liquid/liquid diffusion



- Similar to vapor diffusion
  - Simply involves **carefully** layering a solvent on top of another one (both solvents must be miscible)
    - Bottom solvent contains the dissolved compound
    - Top solvent is one in which the compound is insoluble

Sublimation

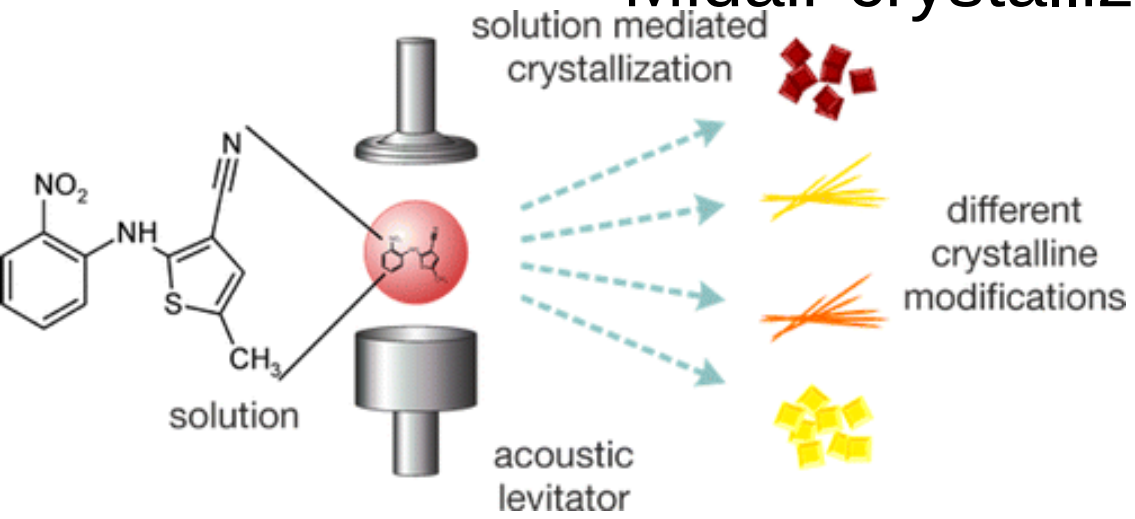
Ferrocene



Nickelcene

- Will only work if your compound is sufficiently volatile
  - Crystallization relies on heating the compound (generally under vacuum) and collecting crystals on a cooled cold-finger
  - Major advantages
    - Material collected is highly crystalline
    - By the nature of the method the crystals grown are free of solvent impurities

Midair crystallization



Polymorphic compound studied in situ by combined time-resolved XRD and Raman spec.

Any influences of solid surfaces, temperature, and humidity on the crystallization were omitted by the use of specially designed acoustic levitator

- Acoustic levitation
  - Two metal probes emit and reflect sound waves that trap liquid droplet in midair
    - Eliminates interference from container walls
  - Begin with an under saturated solution
    - As solvent evaporates saturation increases leading to nucleation and crystallization