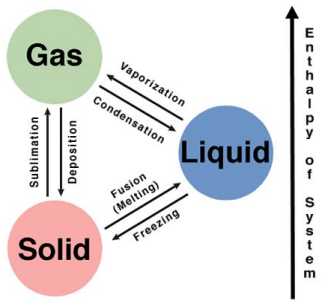


Sublimation

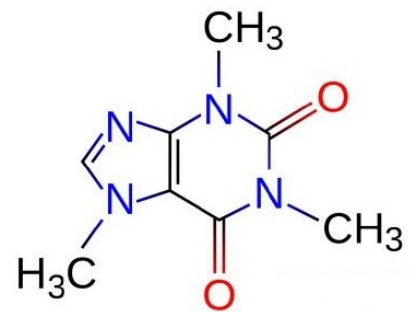
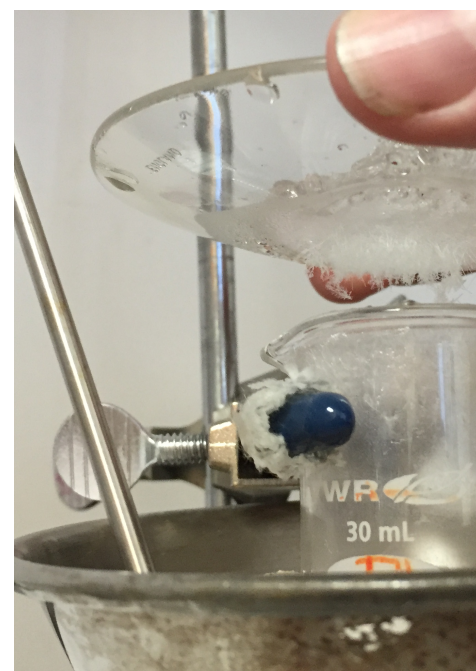
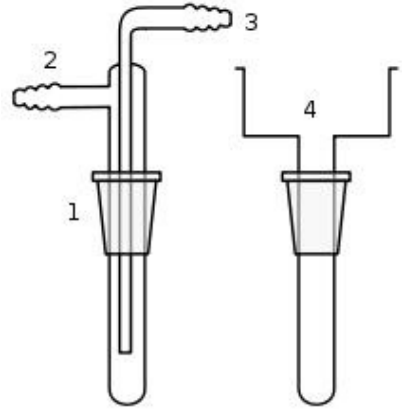


Sublimation

- ▶ Property
- ▶ Process
- ▶ Techniques

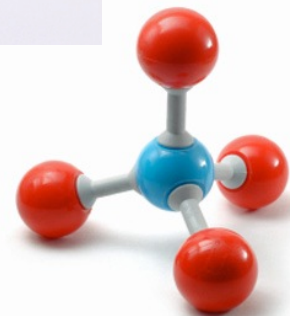
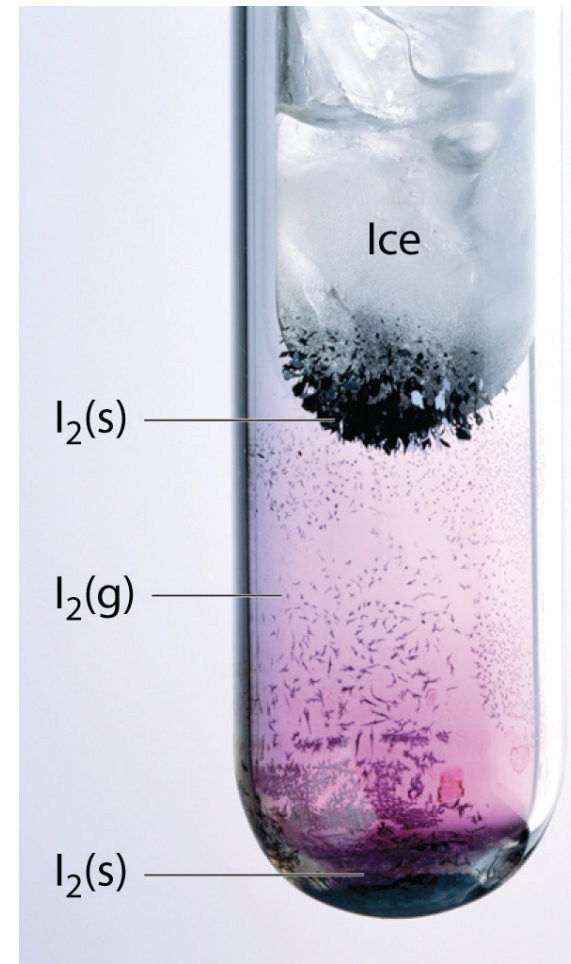
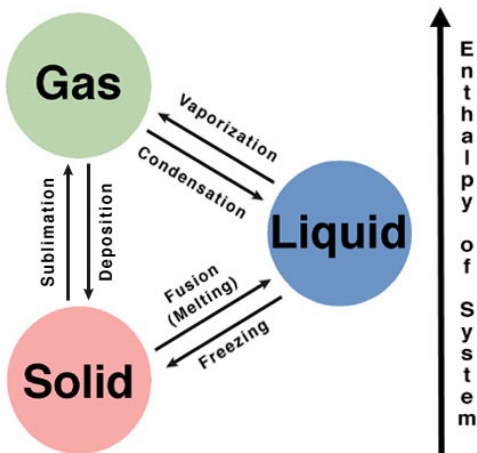


- ▶ The Experiment
 - ▶ Preparation
 - ▶ Sublimation
 - ▶ Analysis
- ▶ For Next Week



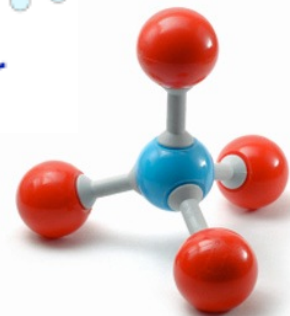
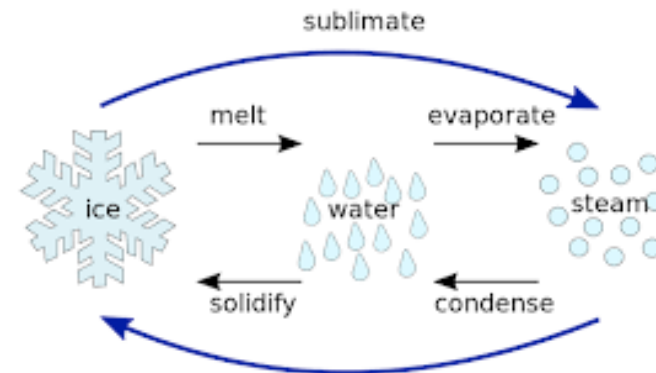
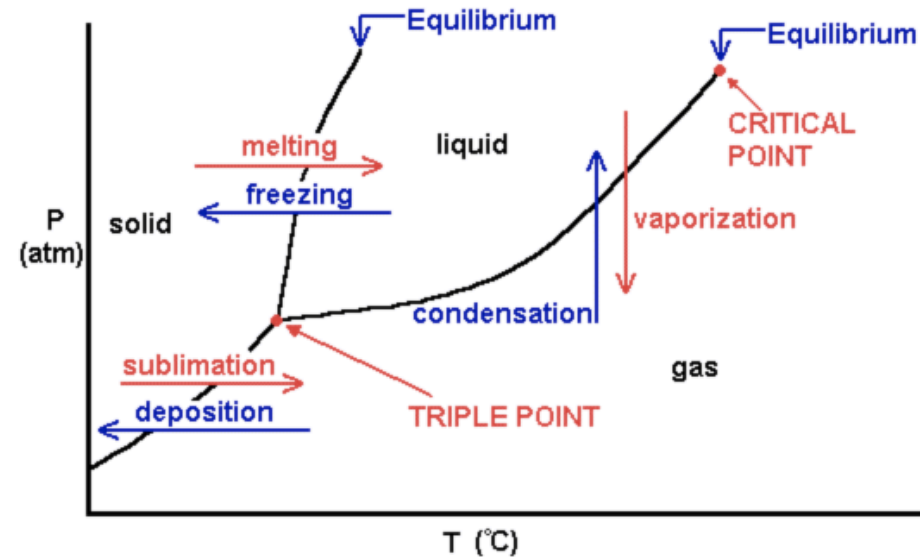
Sublimation

- ▶ **Sublimation** is the property of a substance to go directly from the solid to gas phase.
- ▶ Not all substances significantly sublime.
 - ▶ Sublimation is a property most often observed in non-polar substances with symmetric structures.
 - ▶ More polar and less symmetric substances often have too strong intramolecular forces.
- ▶ Not all substances that sublime do so at atmospheric pressure.
- ▶ Sublimation is a physical property, like boiling or melting. It neither changes nor requires change in the chemical structure of substances.



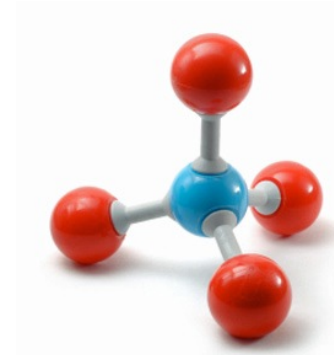
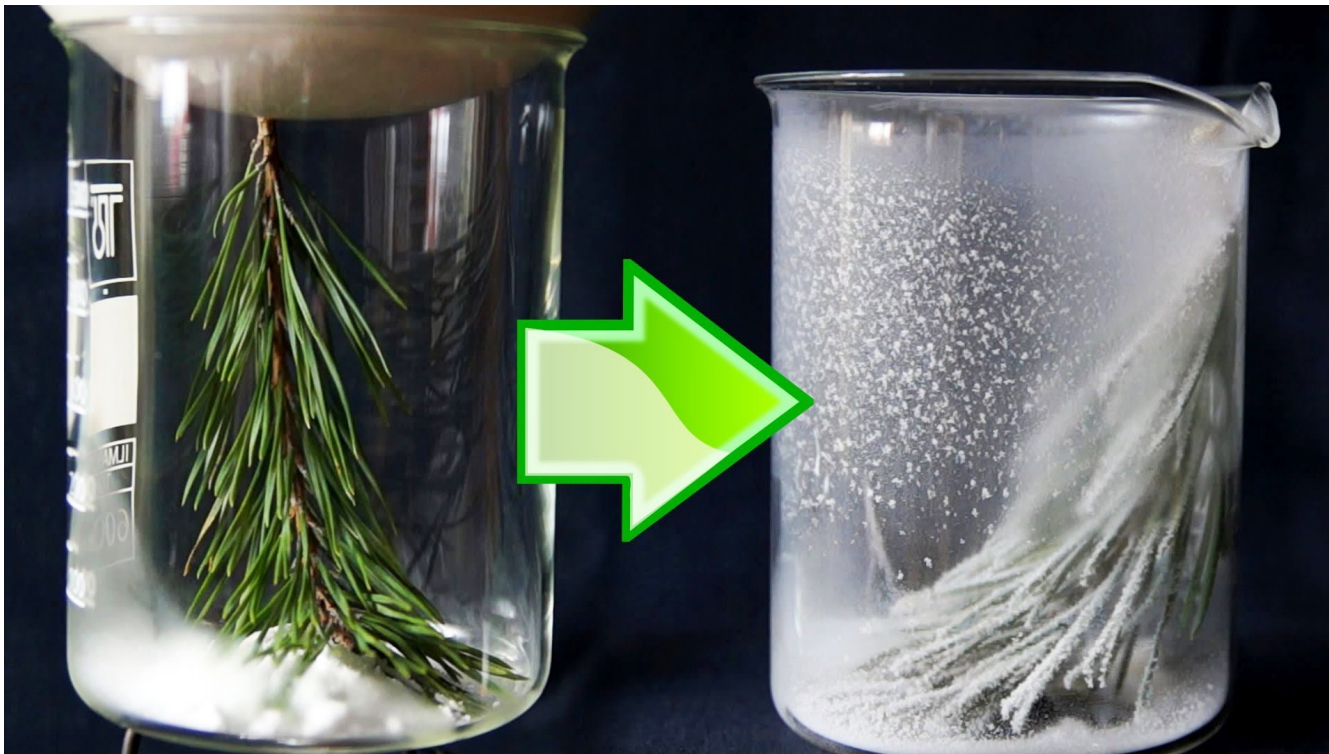
Sublimation

- ▶ Sublimation is molecules passing from the solid to the gas state without going through the liquid state. This happens all the time.
- ▶ If the rate at which they sublime just a question of at what rate it sublimates and how that rate compares to its rate of melting in the same conditions.
- ▶ Sublimation requires:
 - ▶ high heat
 - ▶ low pressure
- ▶ If pressure is too high, materials will melt before subliming.
- ▶ Not all materials significantly sublime,



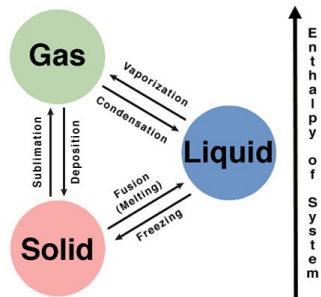
Sublimation

- ▶ Snow is created when water vapor—the gaseous state water—is cooled so much that it turns into solid ice crystals or snow.
- ▶ **Deposition** is when a substance goes directly from a gas to a solid. The molecular characteristics of water causes its solid state to be in regular crystals.
- ▶ The size and shape of these ice crystals is determined by the amount of water and the temperature at which snow is formed.

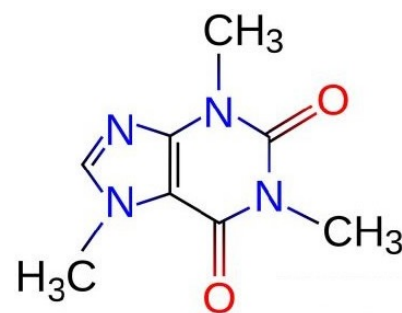
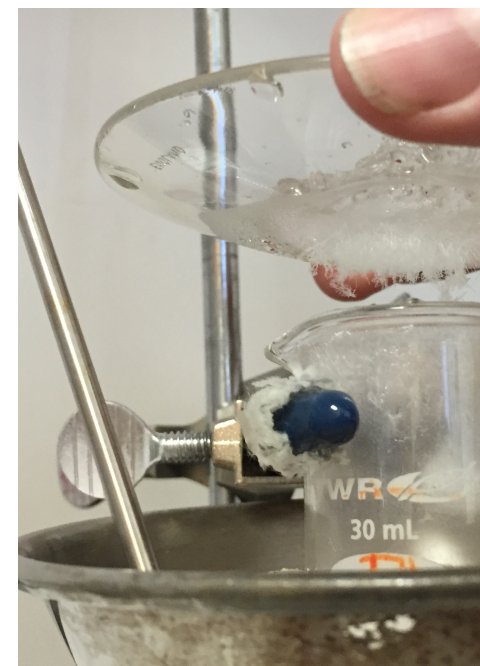
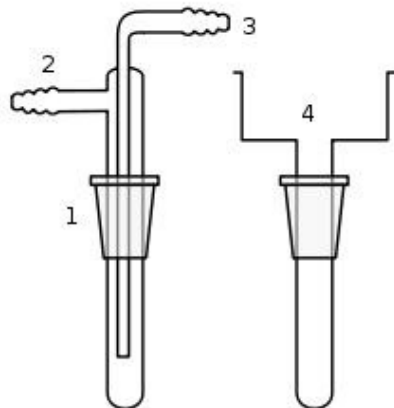


Sublimation

- ▶ Sublimation
 - ▶ Property
 - ▶ Process
 - ▶ Techniques

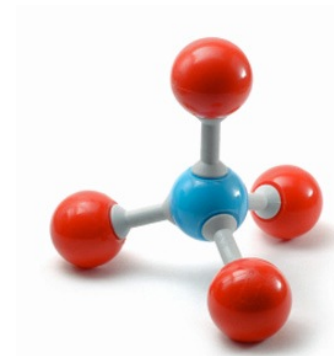


- ▶ The Experiment
 - ▶ Preparation
 - ▶ Sublimation
 - ▶ Analysis
- ▶ For Next Week



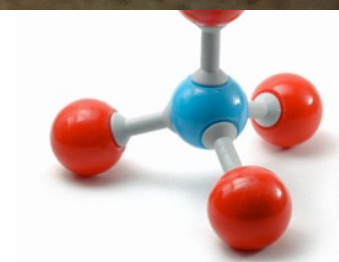
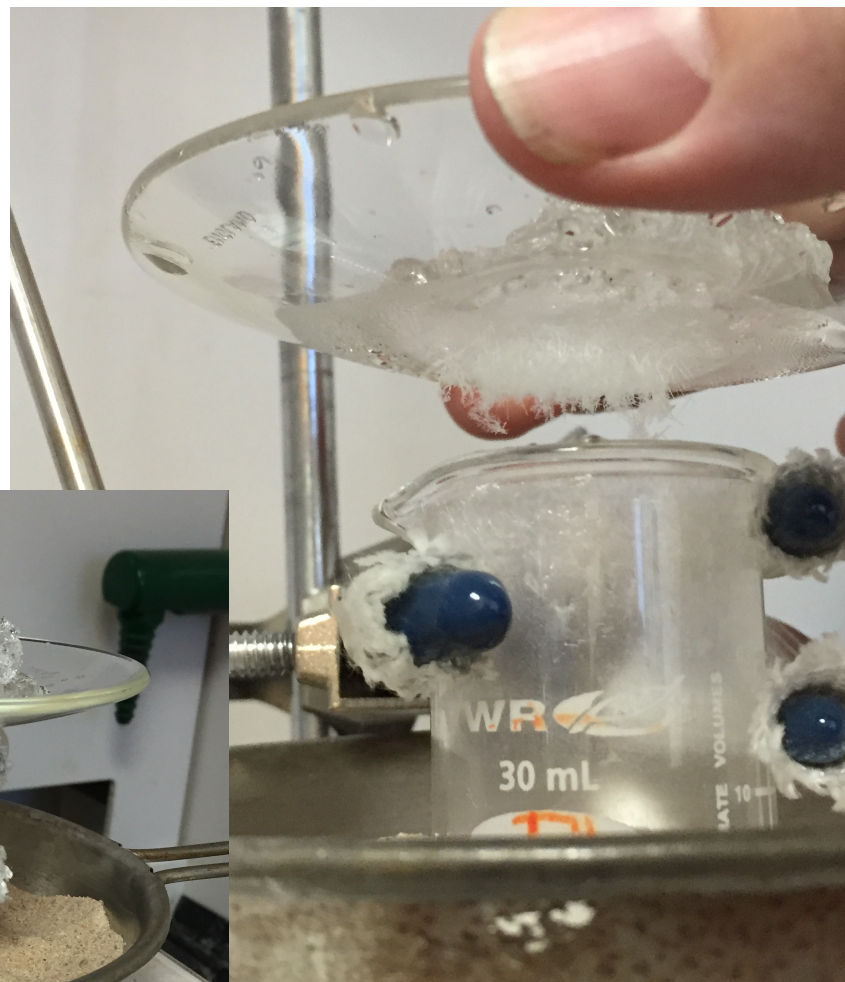
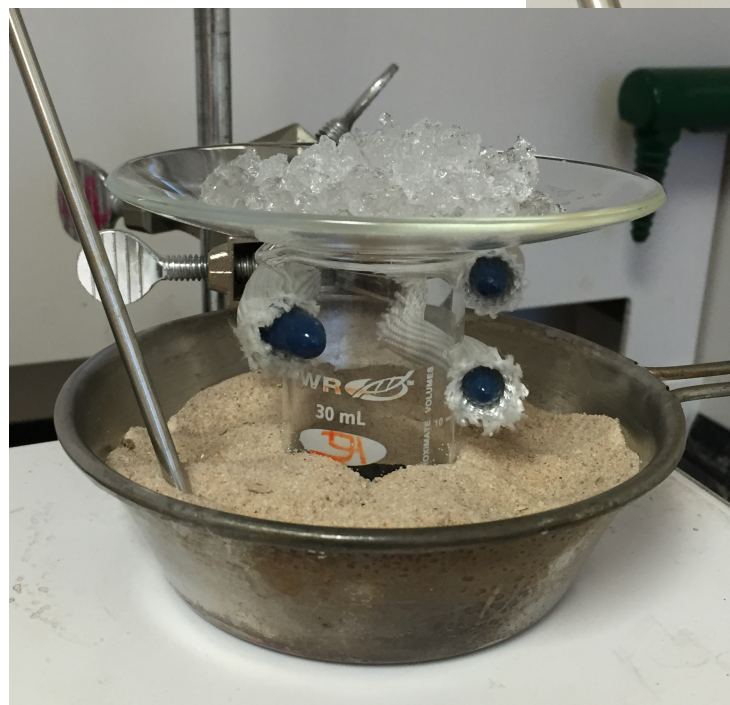
Sublimation

- ▶ If one component of a mixture can be readily sublimed chemists can use this property to separate the mixture.
- ▶ The process of sublimation is similar to that of crystallization.
 - ▶ It's done in the gas phase.
 - ▶ Our solvent is most often air.



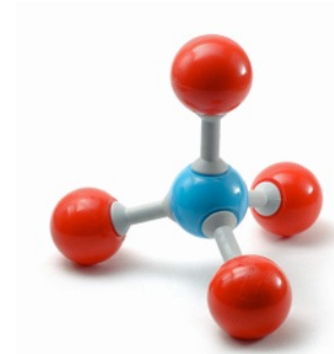
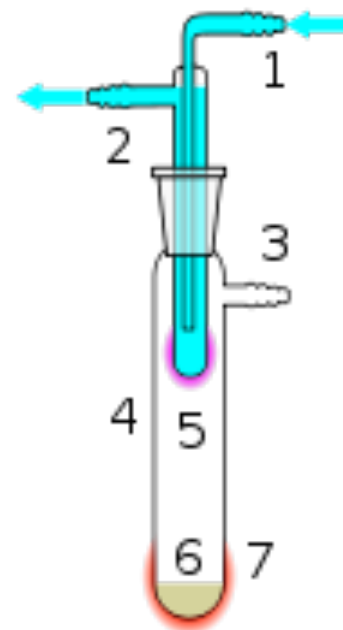
Sublimation

- ▶ Sublimation can be done in the steady state (material isolated as soon as it is created) by subliming the product directly from the reaction mixture.



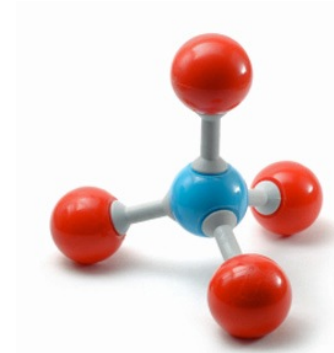
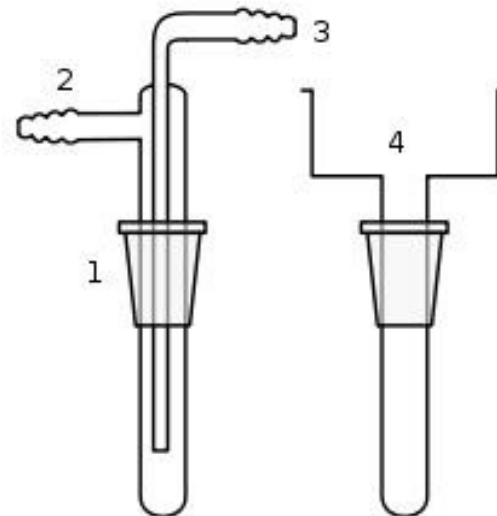
Sublimation

- ▶ Sublimation separations are accomplished by creating a temperature difference between the crude material and the collection site.
- ▶ Temperature differentials can be created by using cold water flow or ice reservoir at one point and a heat source at the other.
- ▶ Vacuum applied to the system reduces the vapor pressure of the system and increases the concentration of substance in the gas phase, accelerating the process.



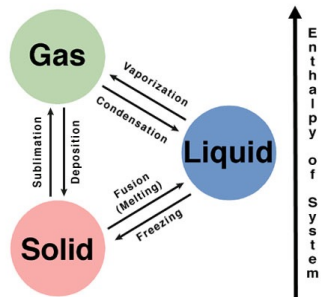
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Sublimation

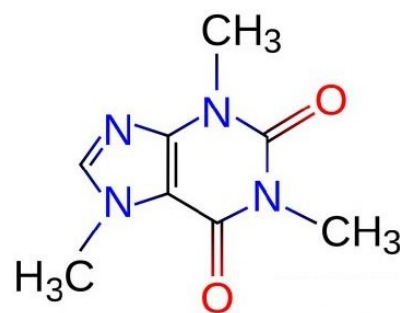
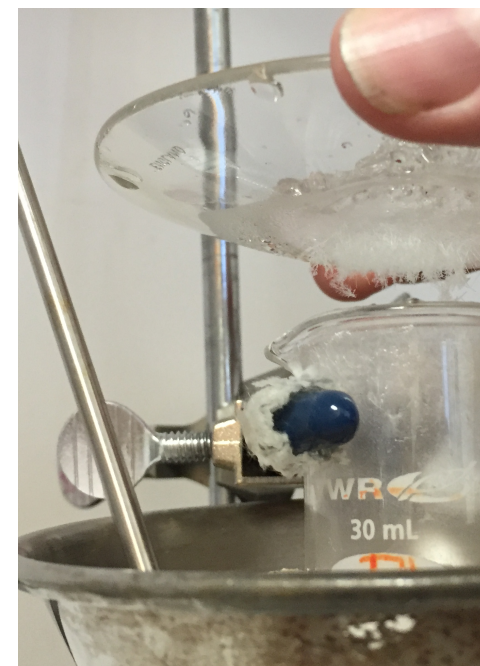
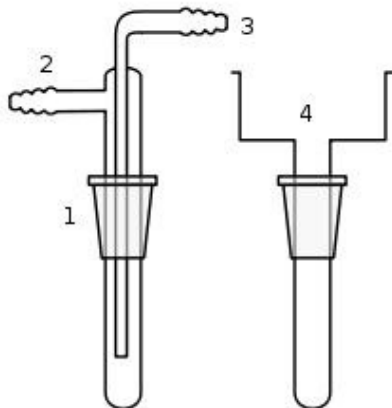
- ▶ Sublimation
 - ▶ Property
 - ▶ Process
 - ▶ Techniques



The Experiment

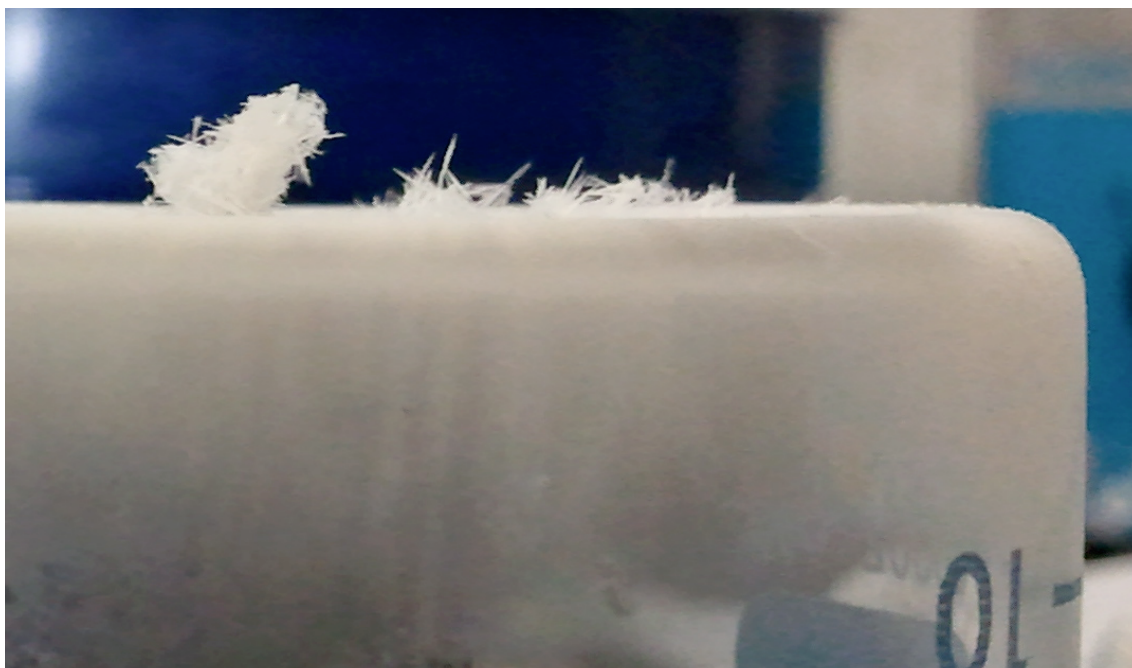
- ▶ Preparation
- ▶ Sublimation
- ▶ Analysis

For Next Week



Separation by Sublimation

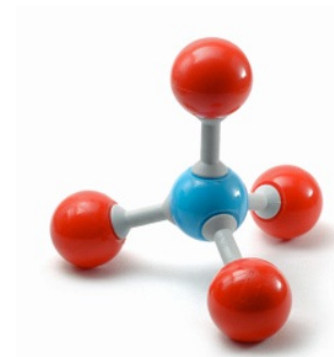
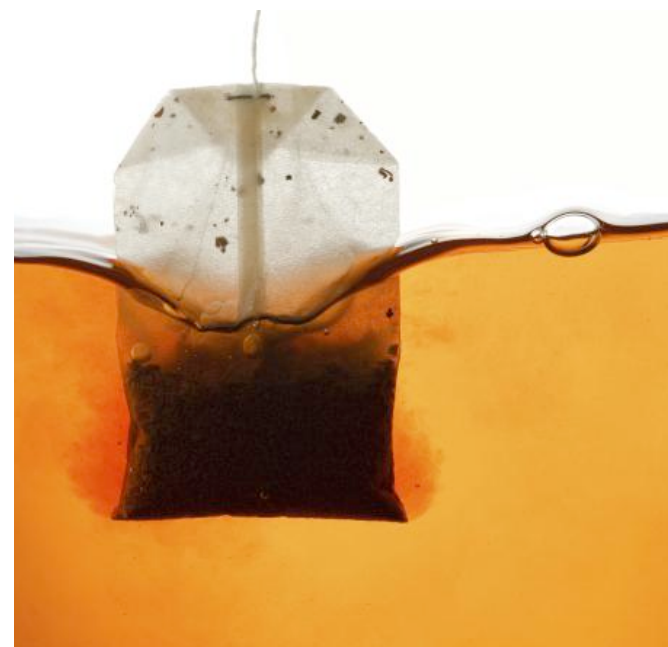
- ▶ **OBJECTIVE:** To isolate pure caffeine from tea by extraction and sublimation separations.
- ▶ **GOAL:** To practice your extraction techniques and melting point techniques while exploring sublimation as a new method of separating mixtures.



Sublimation

▶ Preparing Solution

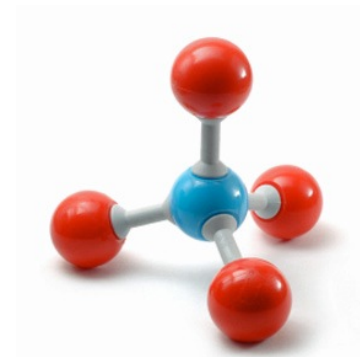
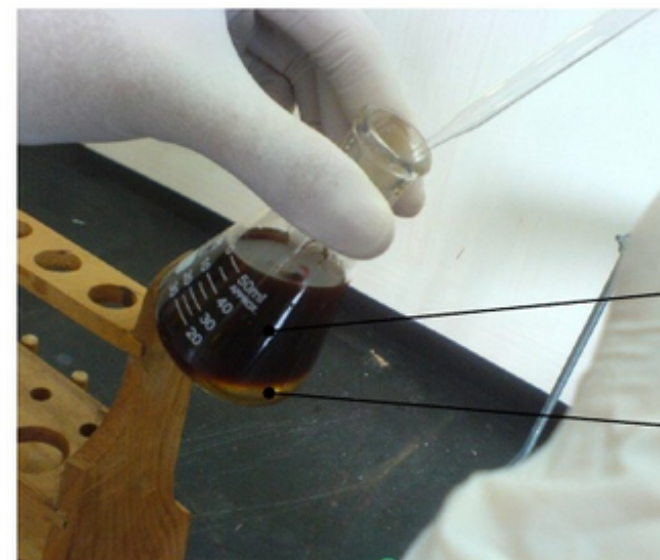
- ▶ Add 20 mL of d H₂O in a 50 mL beaker.
- ▶ Mark water level on beaker.
 - ▶ Heat to almost boiling
 - ▶ Cover beaker with a watch glass
- ▶ Add tea leaves to beaker.
- ▶ Heat for 15 minutes
 - ▶ add additional water to marked level as needed.
- ▶ Transfer half the solution to each of two capped centrifuge tubes.
 - ▶ Transfer by pipette, be careful not to bring across any leaves.
- ▶ Crush leaves with test tube to extract as much caffeine as possible when transferring.
- ▶ Add 0.50 grams of sodium carbonate (Na₂CO₃) to each test tube.
- ▶ Cap and shake to dissolve all solid.
- ▶ Let cool to room temperature.



Sublimation

▶ Extraction

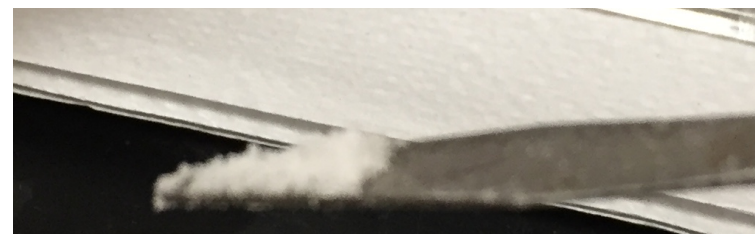
- ▶ Calibrate a pipette for 3 mL.
- ▶ Add 3 mL of methylene chloride (CH_2Cl_2) to each centrifuge tube.
- ▶ Shake for 3-5 seconds, vent tube to release pressure.
- ▶ Shake again for at least 30 seconds.
- ▶ Centrifuge tube for 5-10 minutes to break emulsion.
 - ▶ If two immiscible phases are not observed after centrifuging repeat until emulsion is gone.
- ▶ Pipette methylene chloride layer (lower layer) into a clean dry 25 mL Erlenmeyer flask.
 - ▶ Do not transfer any of the darker aqueous layer.
- ▶ Repeat extraction by adding 3 mL more of methylene chloride to each test tube.
- ▶ Combine all extracts in the 25 mL Erlenmeyer flask.
 - ▶ If any aqueous drops are visible, transfer off the organics to a new flask. Leave behind some organic if necessary to make sure not aqueous drops are transferred.



Sublimation

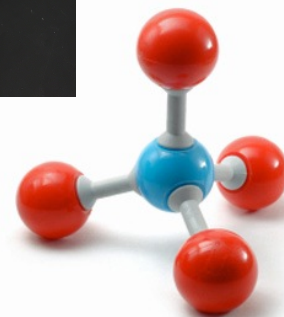
▶ Drying

- ▶ Add 1/3 of the tip of a spatula of gradual anhydrous sodium sulfate to your solution.
- ▶ Stir with a spatula.
 - ▶ If sulfate clumps up, add again.
- ▶ When some of the sodium sulfate last added moves freely, let mixture stand 10-15 minutes.
 - ▶ stir occasional (every 2-3 minutes) while standing
- ▶ Pipette the dried methylene chloride to a clean, dry pre-weighed 25 mL erlenmeyer flask.



▶ Evaporation

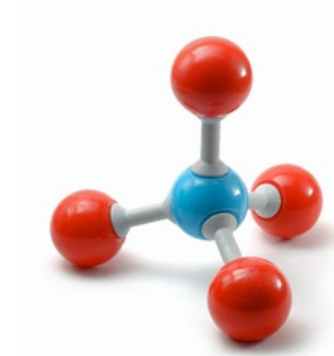
- ▶ Pre-heat a 50°C water bath in the hood.
- ▶ Secure your Erlenmeyer flask in the bath.
- ▶ Allow all the methylene chloride to evaporate (in the hood)
- ▶ Immediately remove the beaker from the bath when all liquid is gone and white power remains.
- ▶ Separate a small portion of the crude caffeine for melting point determination.



Sublimation

▶ Sublimation

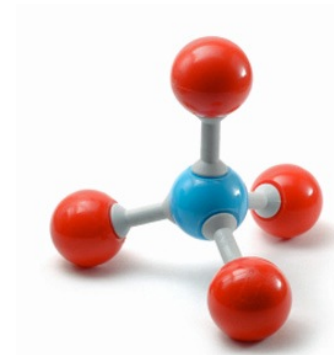
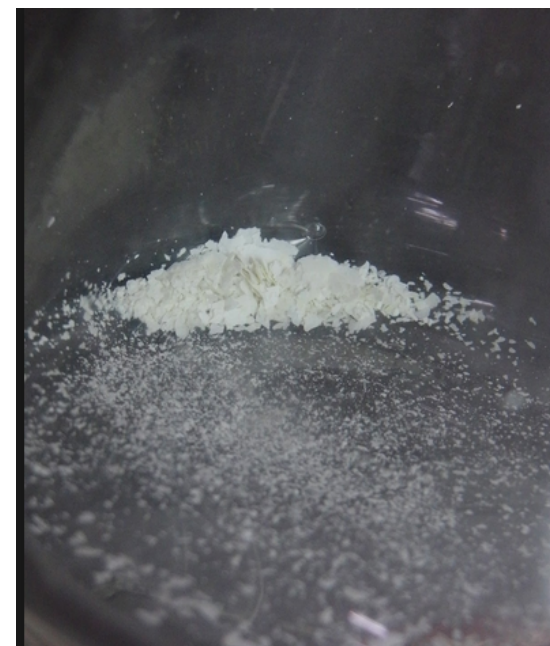
- ▶ Preheat a sand bath to 120°
- ▶ Collect ice in a 250 mL beaker, add enough tap water to create a slurry.
- ▶ Transfer the remaining crude caffeine to the sublimation apparatus.
 - ▶ Scrape the solid with a steel spatula.
 - ▶ Transfer the solid to the sublimation chamber
 - ▶ attach the cold finger and charge with ice slurry
 - ▶ turn on vacuum
- ▶ Monitor the sublimation closely, if the the vacuum is not sufficient it may be necessary to increase heat.
- ▶ If the impure material begins to melt or burn, reduce the heat.



Sublimation

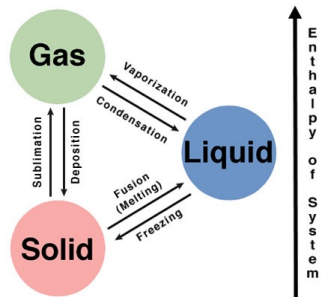
▶ Collection

- ▶ After cooling the sublimation chamber.
- ▶ Carefully remove the cold finger.
- ▶ Hold over a pre-weighted watch glass.
- ▶ With a steel spatula scrape the sublimed caffeine onto the watch glass.

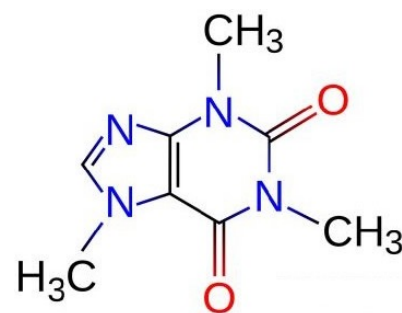
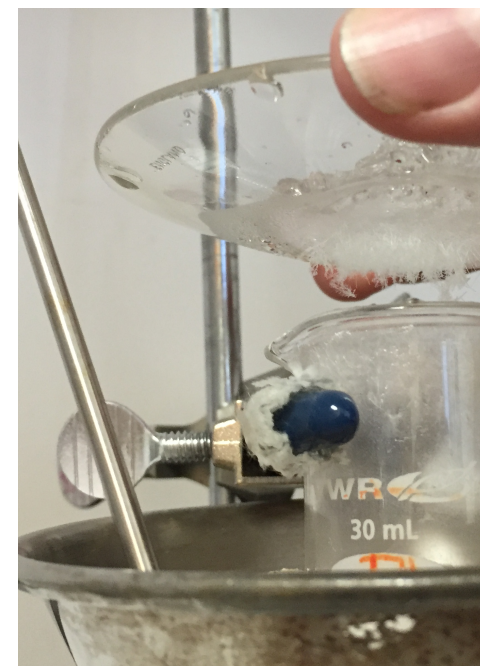
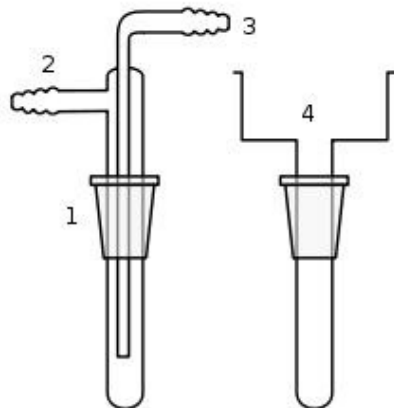


Sublimation

- ▶ Sublimation
 - ▶ Property
 - ▶ Process
 - ▶ Techniques

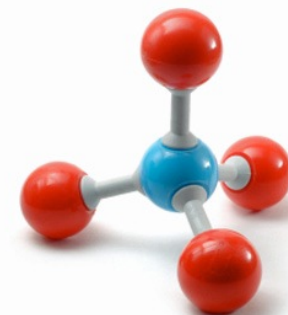
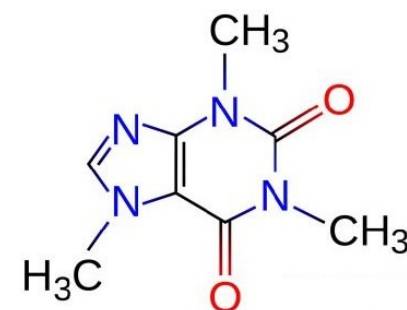


- ▶ The Experiment
 - ▶ Preparation
 - ▶ Sublimation
 - ▶ Analysis
- ▶ For Next Week

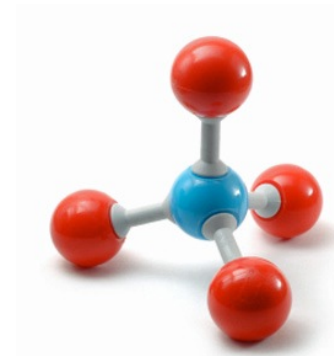
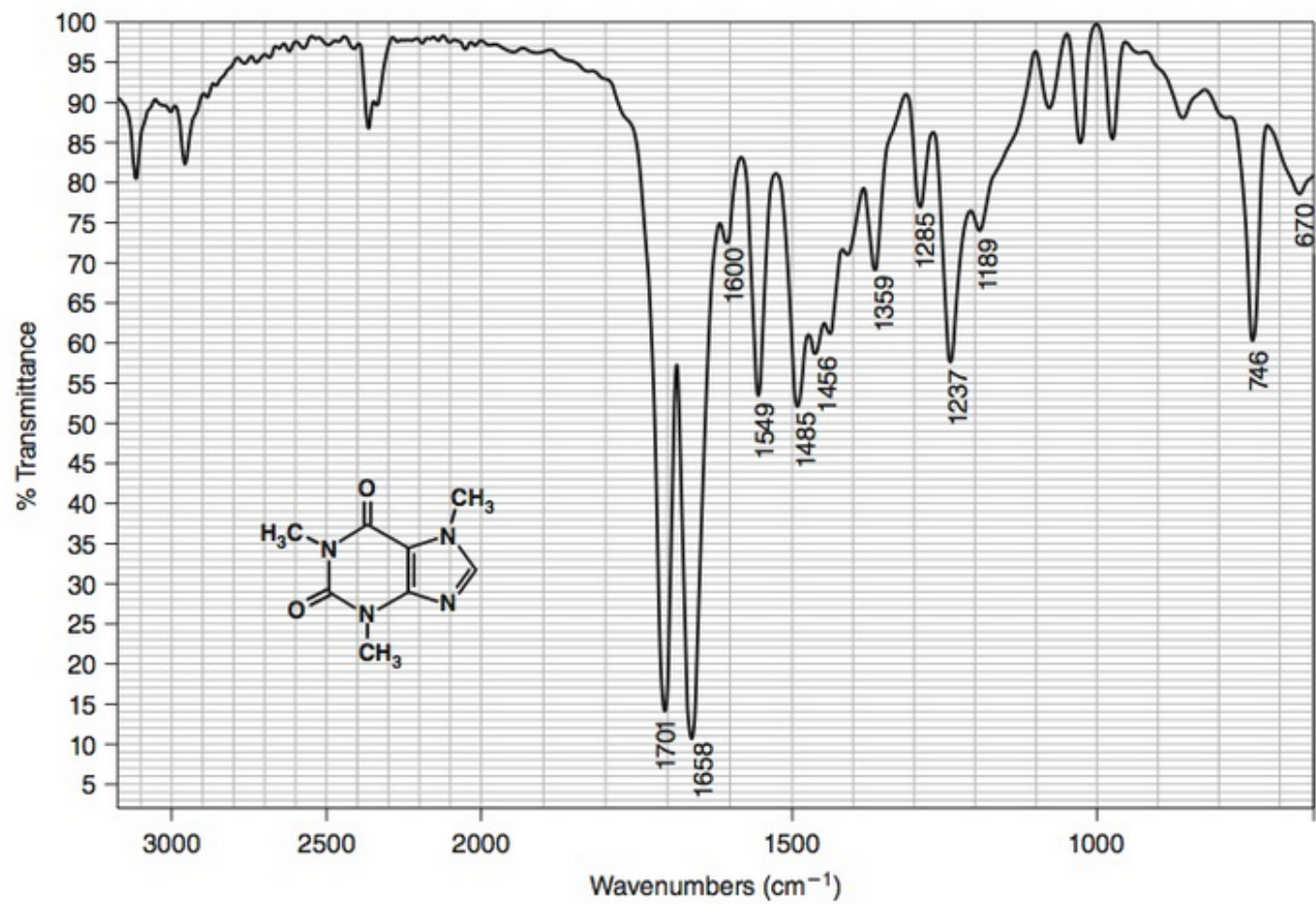


Sublimation

- ▶ Analysis:
 - ▶ Determine and report the melting point of...
 - ▶ Sample A (the crude caffeine)
 - ▶ Sample B (the sublimed caffeine)
 - ▶ In your report, comment on the purity of each.
 - ▶ Report the percent yield of pure caffeine.

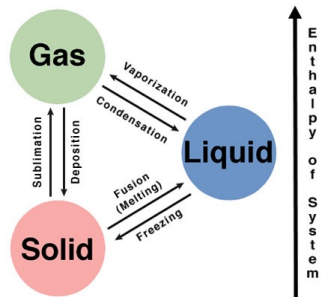


Sublimation



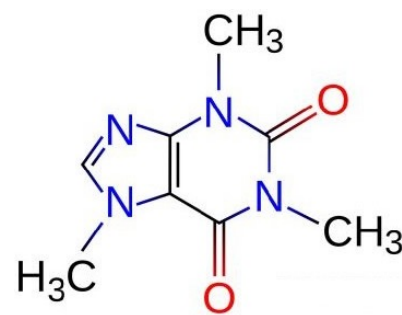
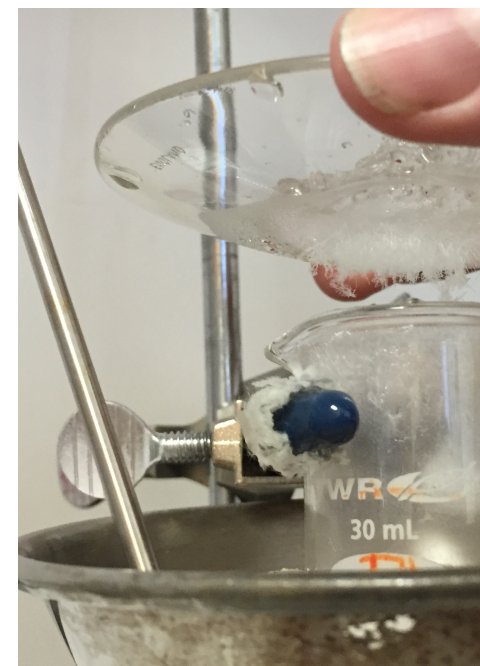
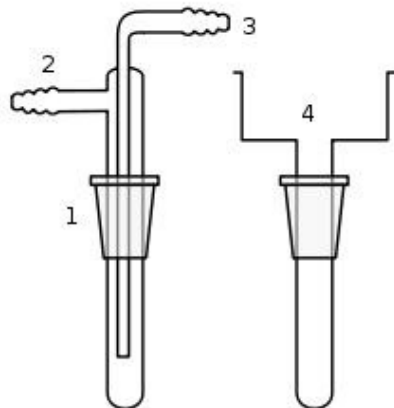
Sublimation

- ▶ Sublimation
 - ▶ Property
 - ▶ Process
 - ▶ Techniques



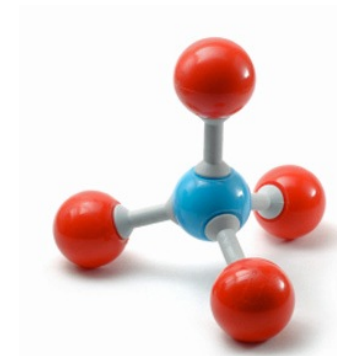
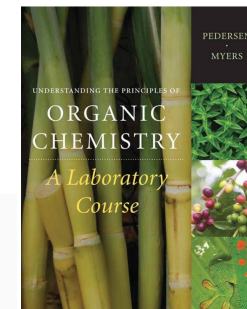
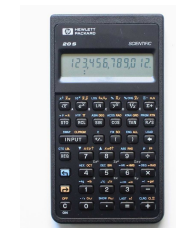
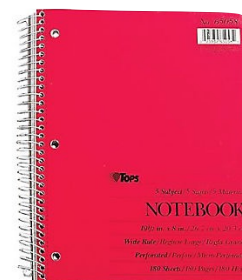
- ▶ The Experiment
 - ▶ Preparation
 - ▶ Sublimation
 - ▶ Analysis

➔ For Next Week



Next Meeting

- ▶ For next Meeting:
 - ▶ Bring to class:
 - ▶ Notebook
 - ▶ You will not be turning in notebooks, but this permanent record of your preparations, observations and notes will be essential to success in this class.
 - ▶ Textbook, calculator, pencils (yes, you can use pen)
 - ▶ Safety Glasses (you cannot participate without them)
 - ▶ Read through and take notes on:
 - ▶ Experiment 15 (p100)
 - ▶ Essay Caffeine (p96)
 - ▶ Sublimation Technique (p779)
 - ▶ Produce and bring to class:
 - ▶ Your pre-lab for exp 15 (p100)
 - ▶ Your procedure summary for exp 13



Questions?

