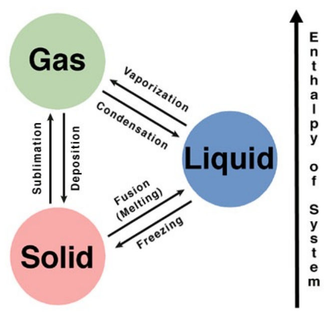
Ex15

Essence of Cloves

- Distillation
  - Vaporization
    - Boiling Point
  - Simple Distillation
  - Steam Distillation
    - Vapor Pressure
- Clove Essence
  - Background
    - Properties



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  - Distillation
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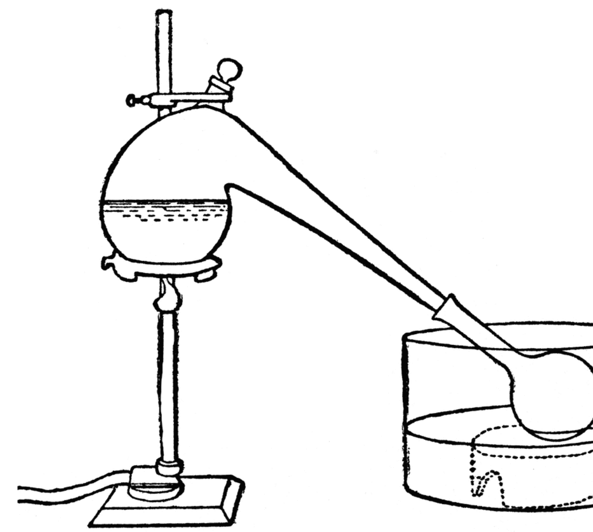
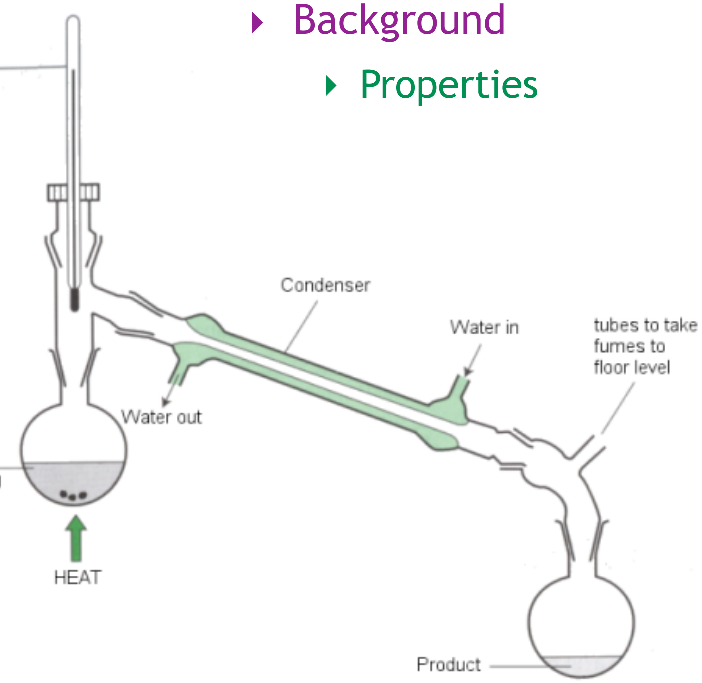
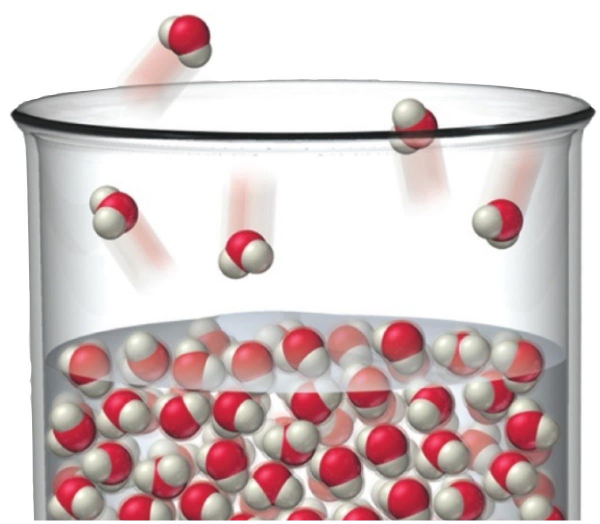
  - Analysis
- For Next Week

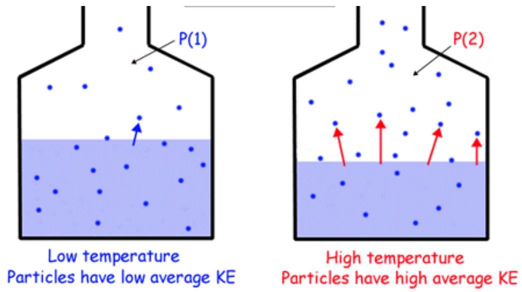


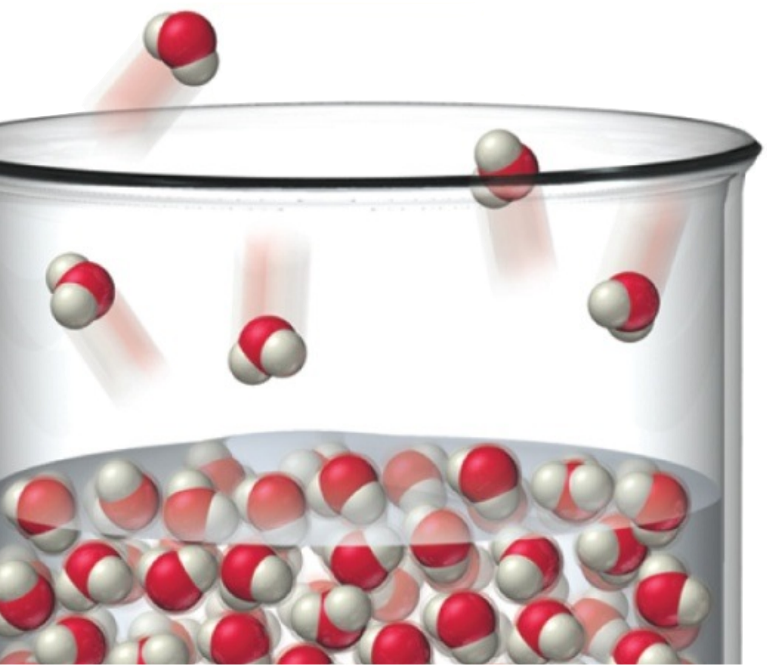


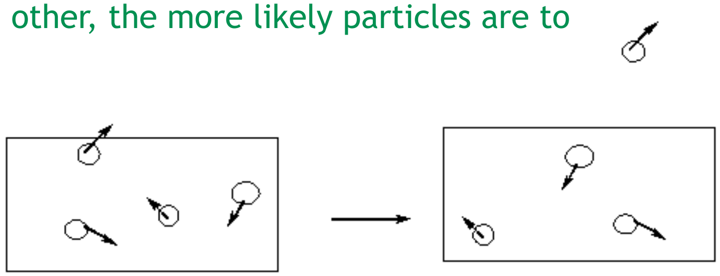


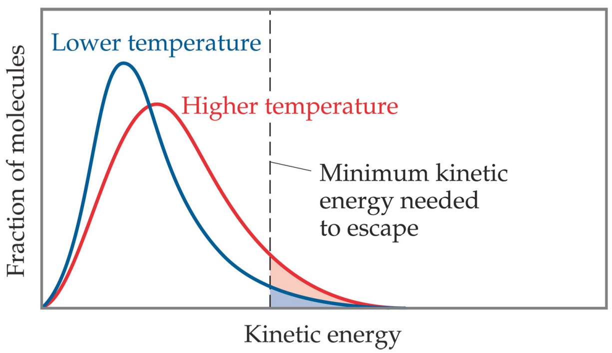
Vaporization

- Vaporization is the process where thermal energy (motion) overcomes intermolecular forces to change the state of matter from liquid to gas.
  - Vaporization is temperature dependent.
    - At any temperature some molecules in a liquid have enough energy to escape.
    - When those particles reach the surface, they overcome the cohesive forces of the liquid and fly free as gas molecules.
    - As the temperature rises, the fraction of molecules that have high enough energy to escape increases.
  - Vaporization depends on the intermolecular forces.
    - The weaker the particles hold each other, the more likely particles are to escape.



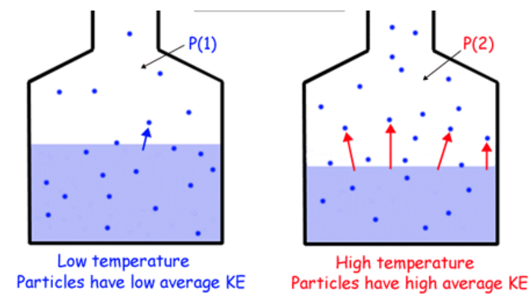


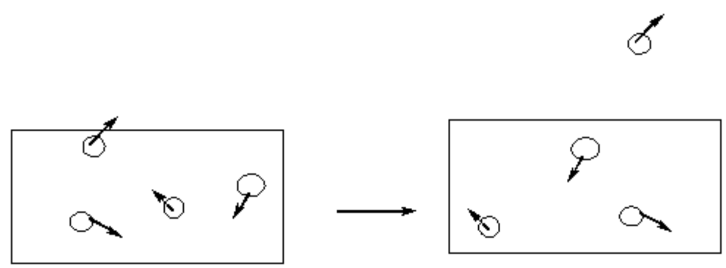


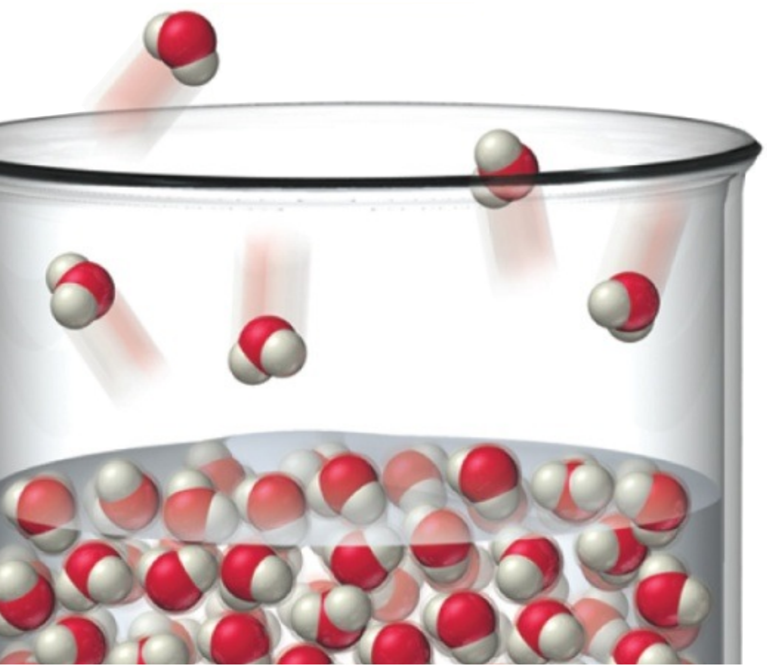


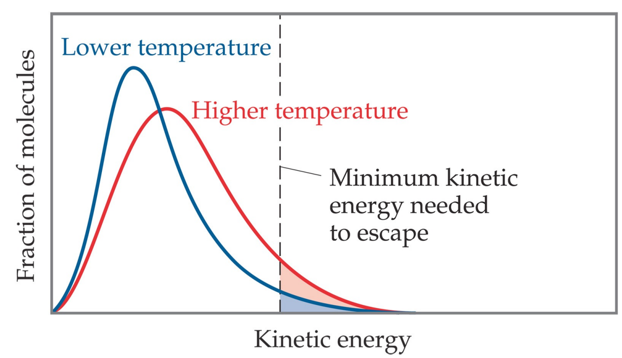
Vaporization

- Condensation is the process of a gas succumbing to intermolecular forces and becoming a liquid.
  - Condensation and Vaporization can both happen at the same temperature.
    - At any temperature some molecules in the gas have less thermal energy that the cohesive forces of the liquid.
    - When those particles strike the surface of the liquid, the cohesive forces cause the molecule to stick to the liquid.
    - As the temperature decreases, the fraction of molecule that have low enough energy to get trapped increases.



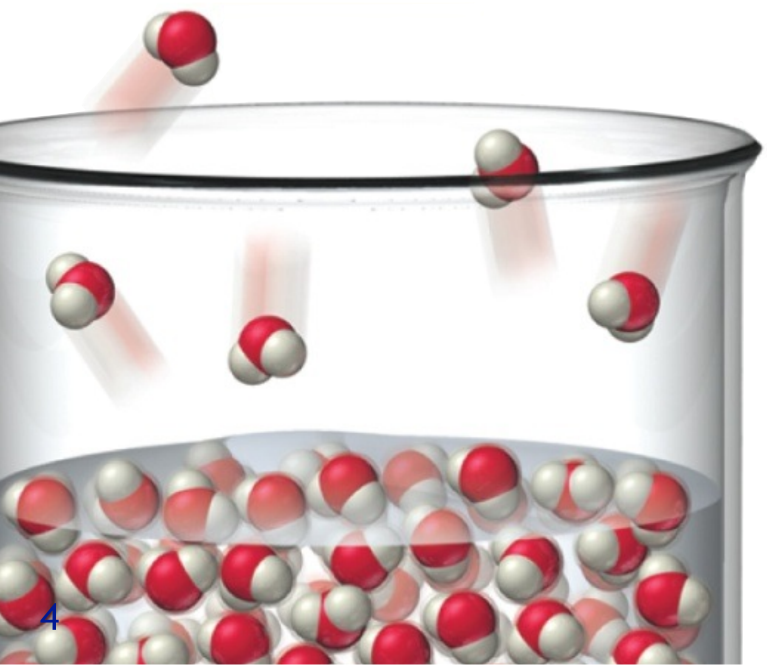


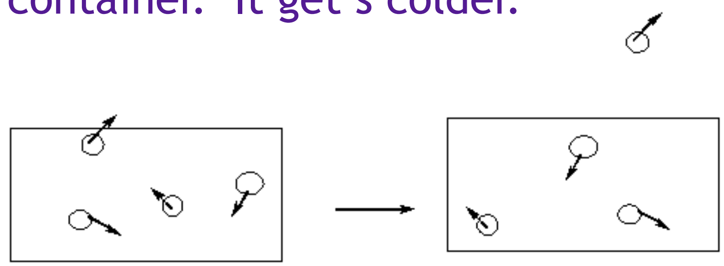


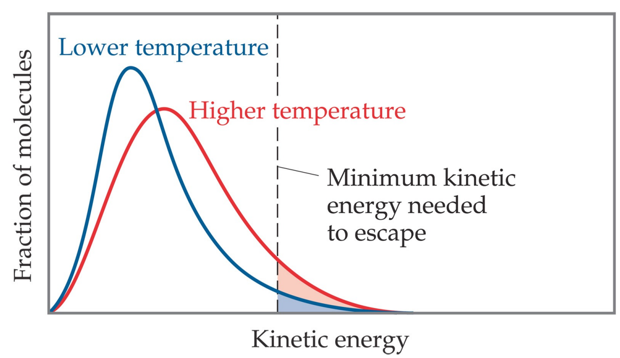


Heat of Vaporization

- Evaporation is an endothermic process.
- Thermal energy is motion of particles.
  - Temperature is a measure of the average speed of particles in a system.
  - As the faster moving (hotter) particles escape the system
    - … the average speed (temperature) of the remaining particles decreases.
- When particles evaporate from a container, the net result is lower the average energy of that container. It get’s colder.

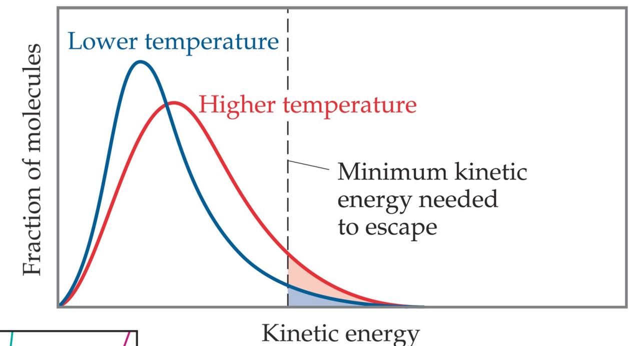


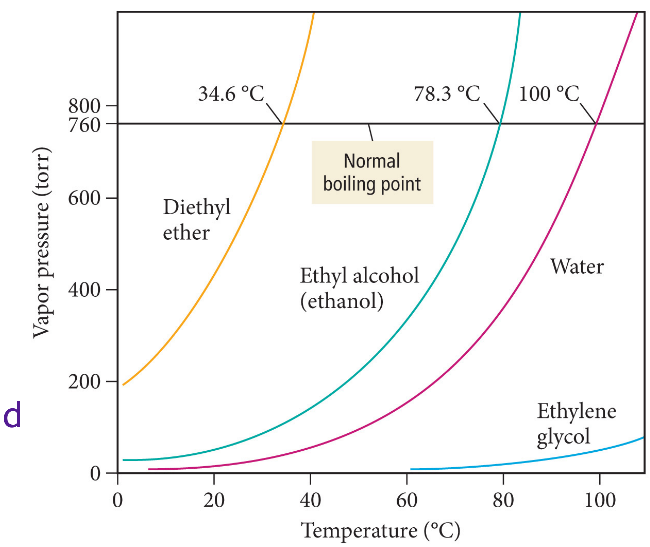




Boiling Point

- Boiling point is the temperature at which the Vapor pressure of the liquid equals the pressure of the world around it.
- It's a threshold.
- It's when a substance has absorbed as much heat as it possibly can in that state.
- It's the point where the heat absorbed is equal to the intermolecular forces holding that substance together.
- Every joule of heat that goes into that substance after it's reached it's boiling point defeats the intermolecular forces of some portion of that substance.
- It vaporizes that portion.
- It explodes it.
- And any portion still a liquid, stays at the boiling point.
- So water boils at 100˚C.
- As you add more heat, you vaporize more of that water.
- But the temperature stays a dependable, unwavering 100˚C until there is absolutely no liquid water left.





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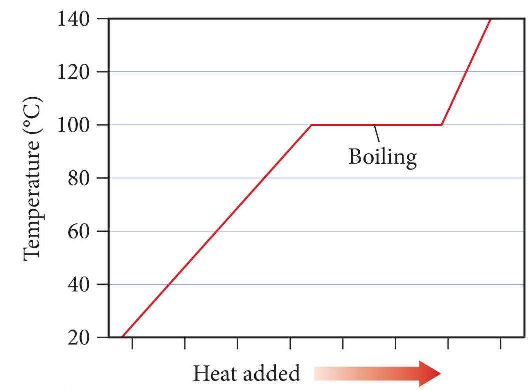

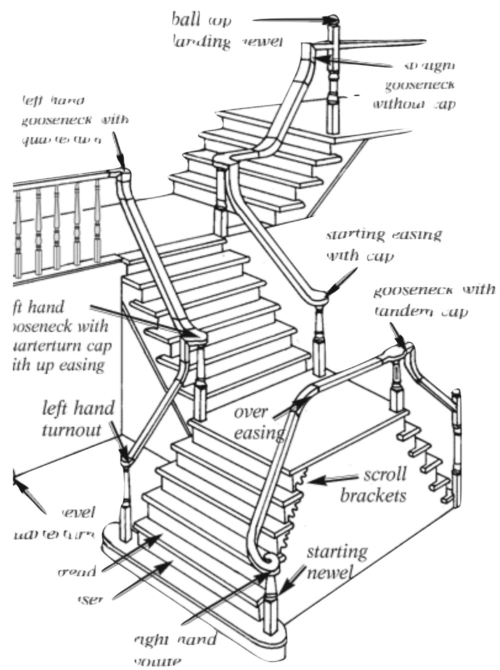

Ex15

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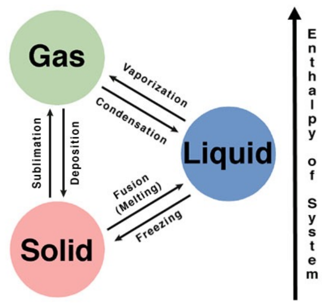

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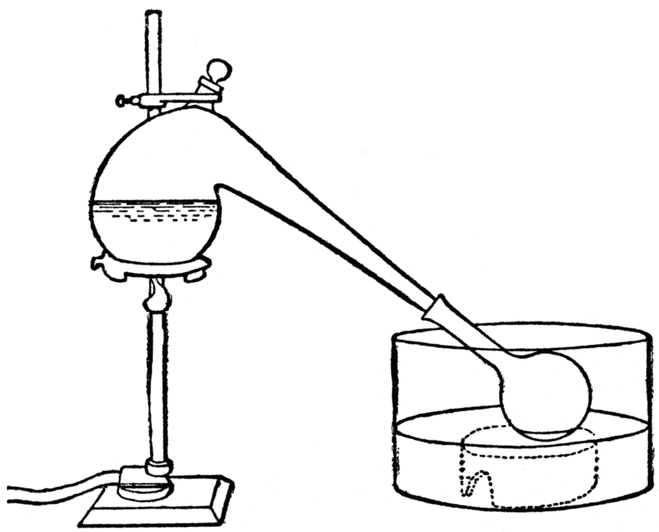
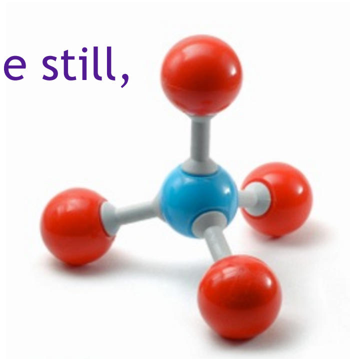


Essence

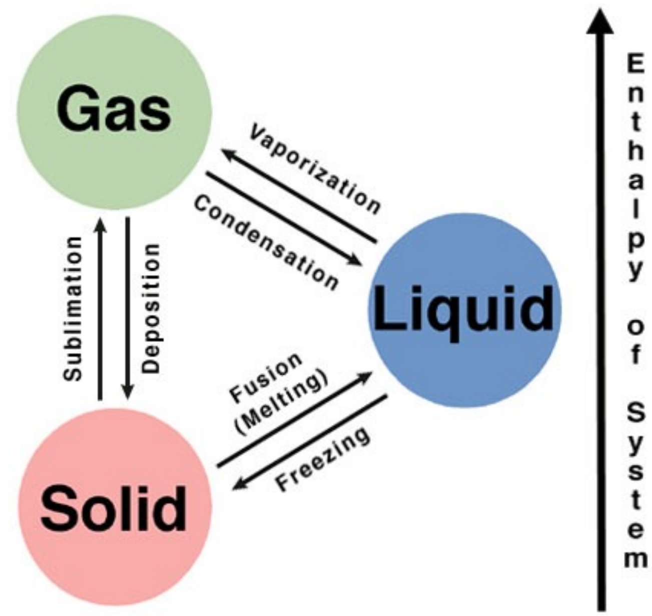
Painting by Stradamus 1550
"The Alchemists"

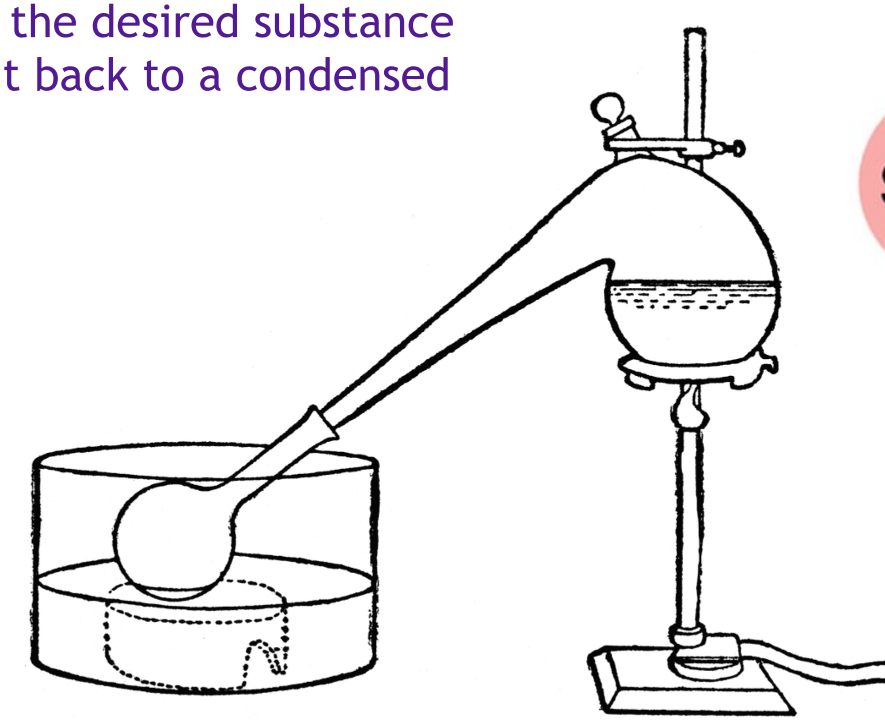
- The goal of alchemy is to find the essential substances in the matter around us.
- We didn't understand atoms or molecules in the time of alchemy, but we new there was something in fruit, grain, mud, or wine that gave it the essential properties we sought.
- These components were called essences and were most often extracted as viscous liquids (oils).
- The most common way essential oils were isolated was by distillation.
- The apparatus we used for distillation, the still, became an iconic symbol of alchemy.

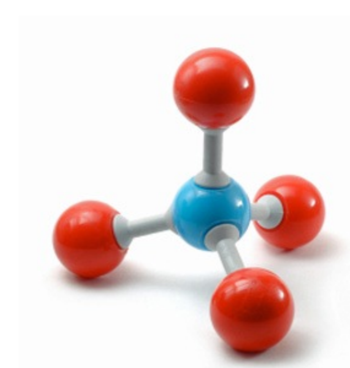


Distillation

- Distillation is isolating a substance based on differences in boiling point.
- Like sublimation, the process involves achieving a temperature at which the desired substance is freed into a mobile phase (gas) and transferred to another container.
- Then capturing the desired substance by condensing it back to a condensed phase (liquid).

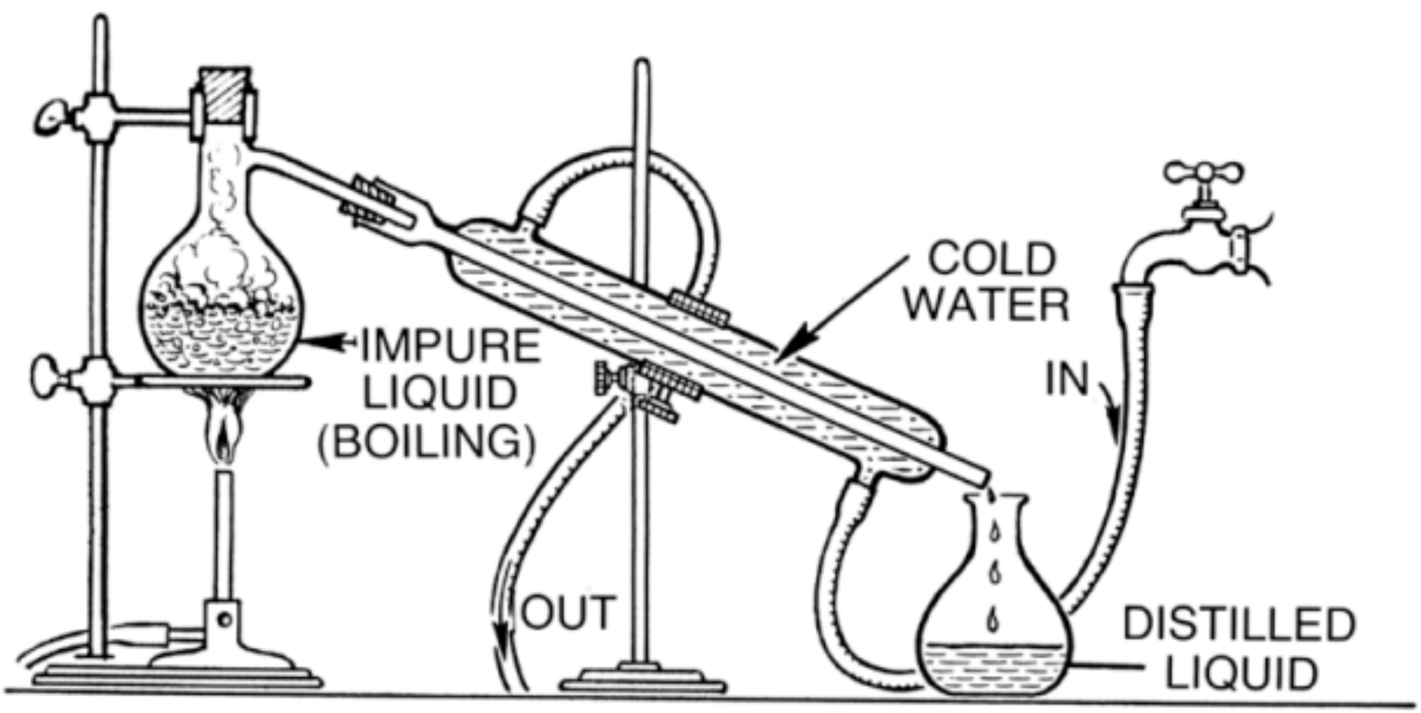


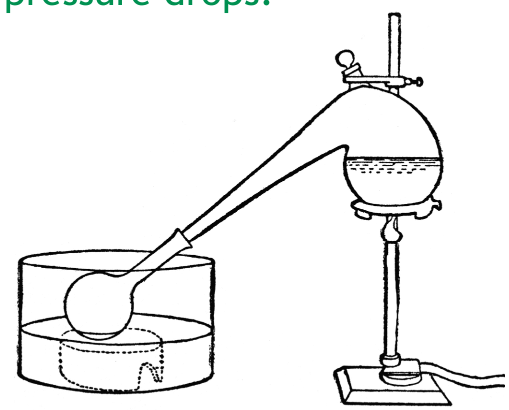


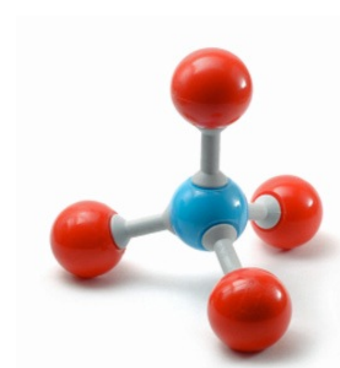


Distillation

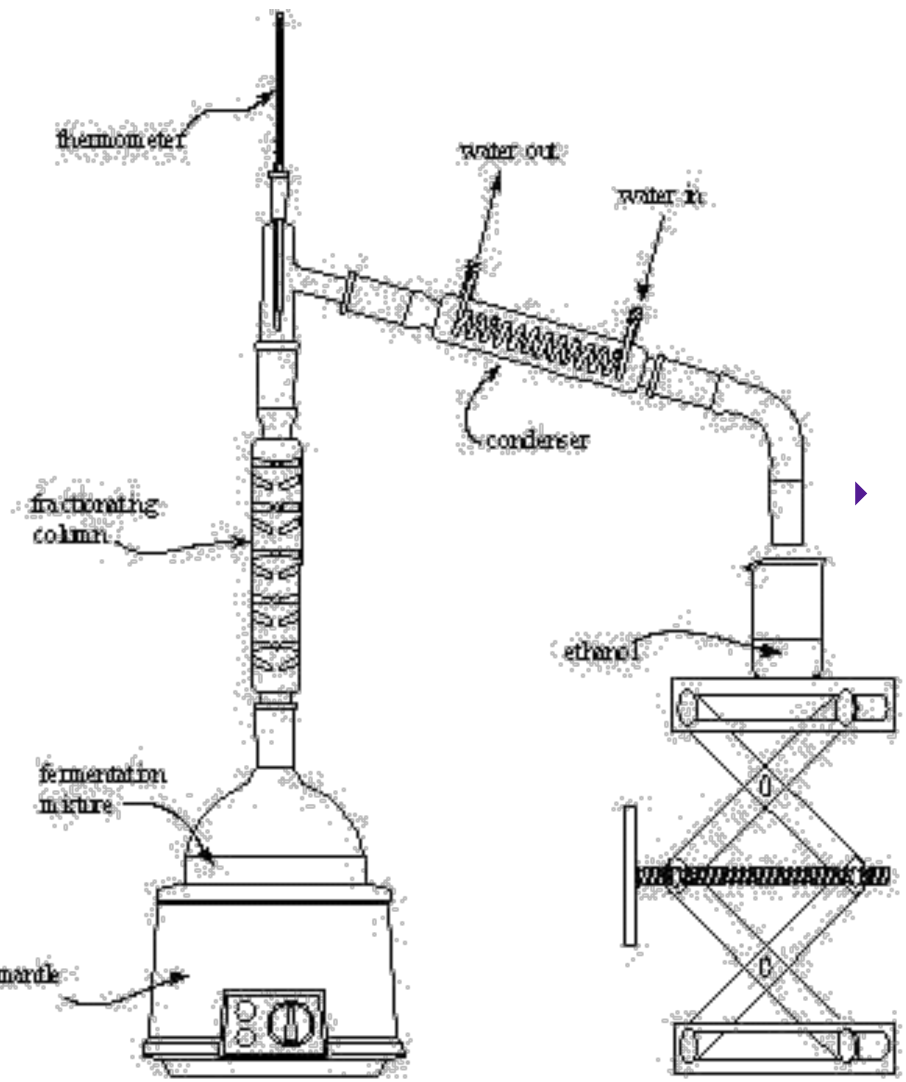
- Condensing the liberated gas can be accomplished with a variety of techniques.
- In the lab, we usually use a water condenser.
- The water condenser has an inner tube surrounded by an outer jacket.
- To cool the inner tube, we flow cool water through the outer jacket.
  - Always run the water up hill, in through the lower connector out through the higher.
  - If you reverse the flow of water your risk your jacket running dry if pressure drops.



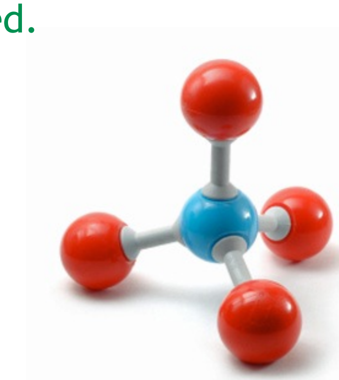




Distillation



- Laboratory distillation can separate substances with even very slight differences in boiling point.
  - Using a fractionating column and adjusting the heat source we can capture multiple fractions and separate different fractions.
    - Capture something that boils at 40˚C in one flask.
    - Then something that boils at 60˚ C in another.
    - Then something that boils at 80˚C in a third.
- We know what substance we are capturing by looking at the temperature of the gas being produced.
    - Your distillation head thermometer should always be well above the level of the liquid.
    - Position it at the start of the condenser, so you know what temperature gas is being condensed.



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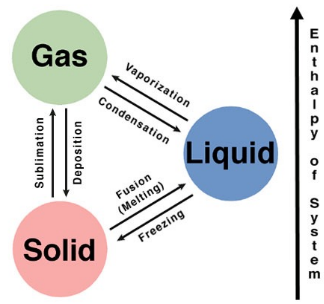

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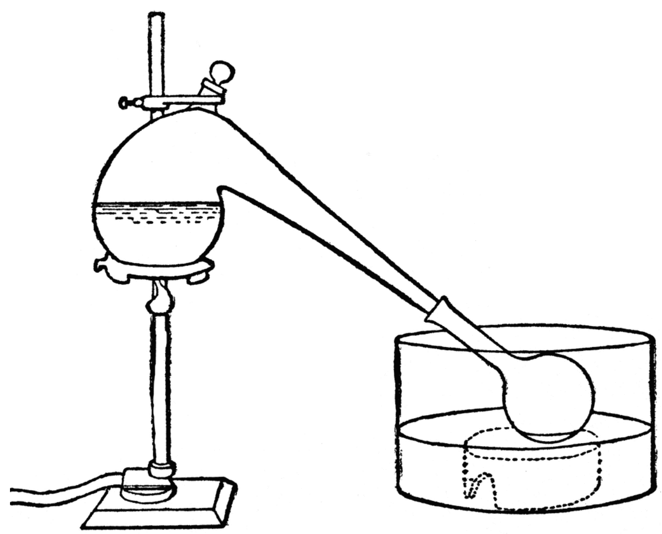
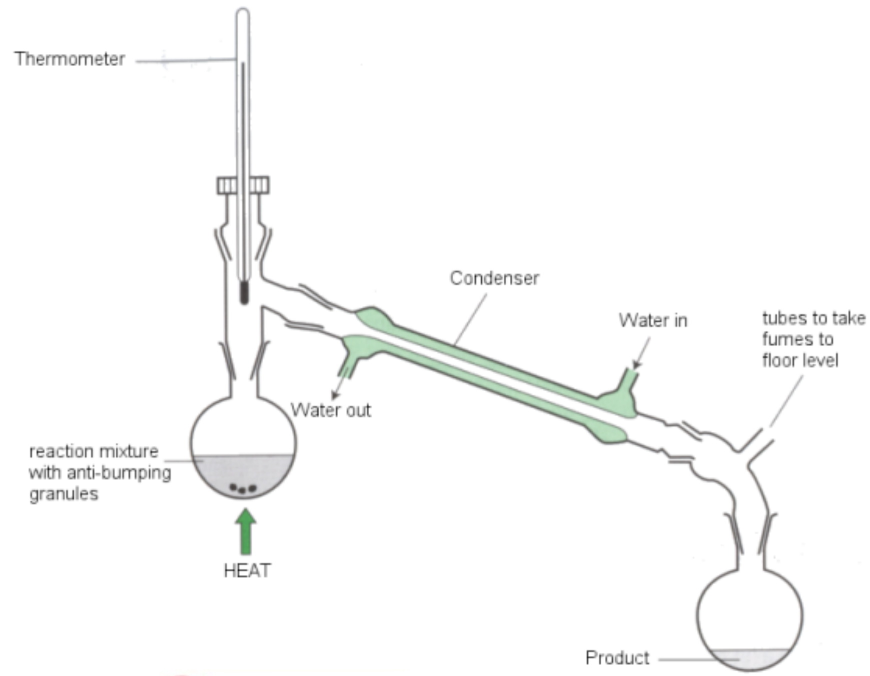
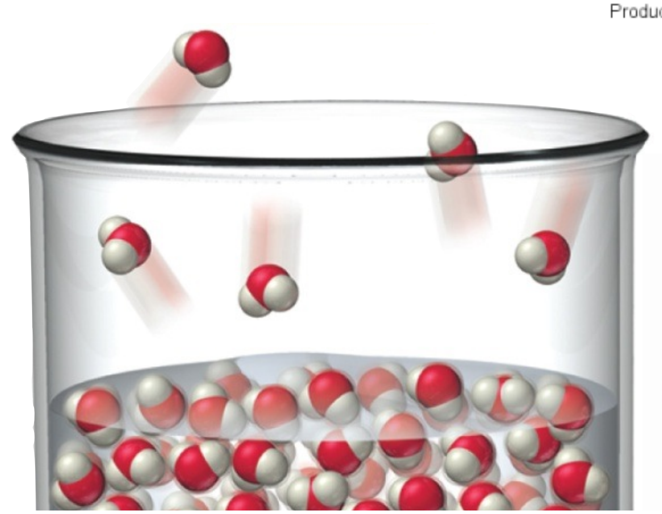
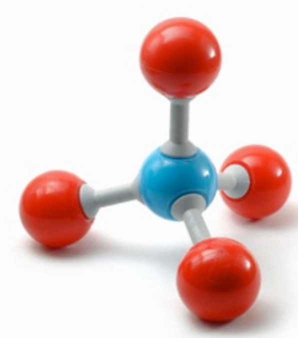


Distillation

- Steam distillation is distilling a water mixture with a volatile substance dissolved in water.
- The water vapor will sweep the small amount of substance that is volatile below it's boiling point along with the water.
  - This allows us to condense and isolate a sample that is not pure, but is rich in the more volatile components of the mixture.
  - The advantage of steam distillation is it allows us to produce these rich fractions at much lower temperatures than we might need to use for fractional distillation.







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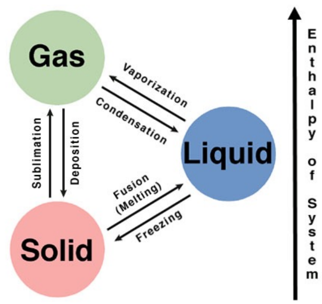

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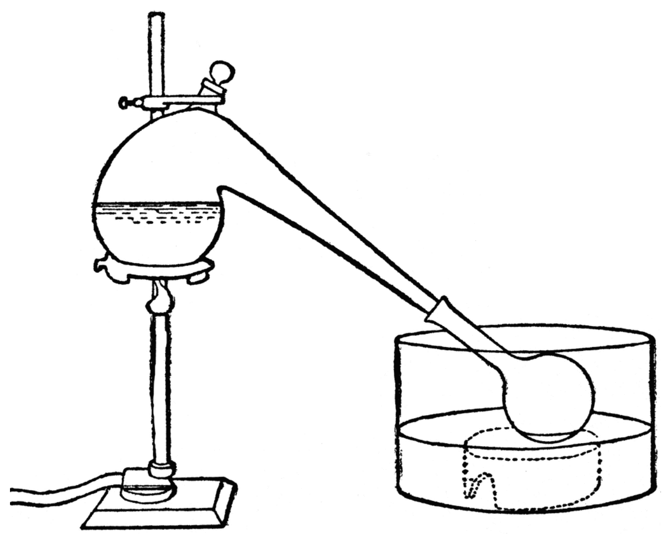
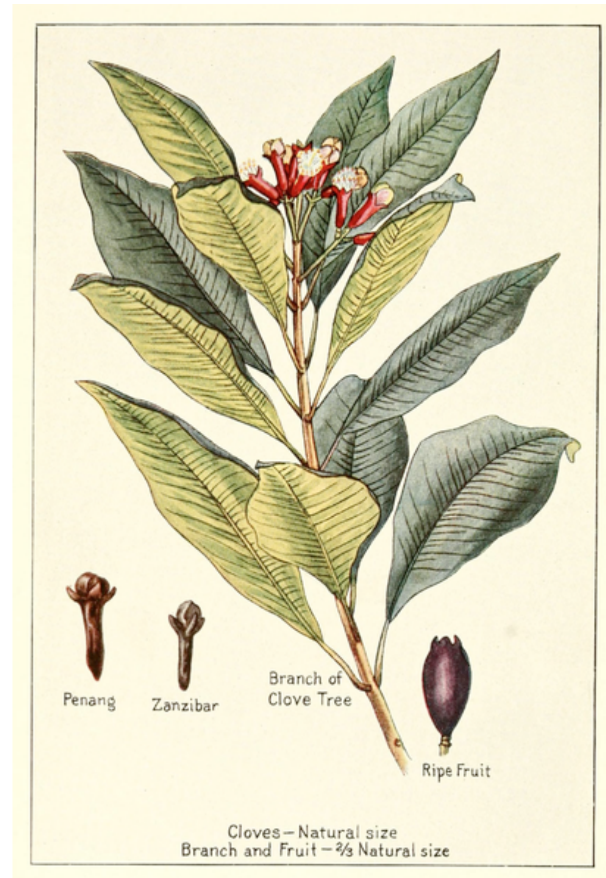
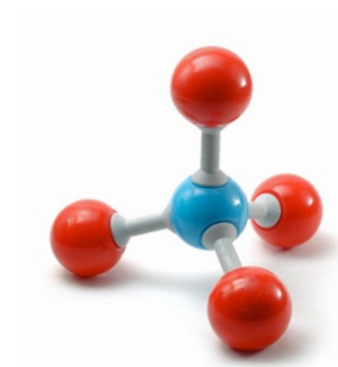


Cloves

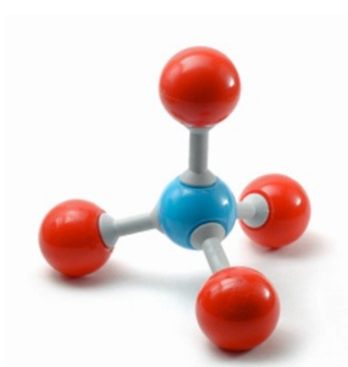
- Cloves are flower buds from an evergreen tree native to Indonesia.
- Evidence has been found that clove trees have been harvested for clove since at least 1721 BC.
- Originally cloves were only available from the Moluccas islands (also called the spice islands), but were valued and traded around the world.
- Today cloves are commercially harvested primarily in Bangladesh, Indonesia, India, Madagascar, Zanzibar, Pakistan, Sri Lanka, and Tanzania.



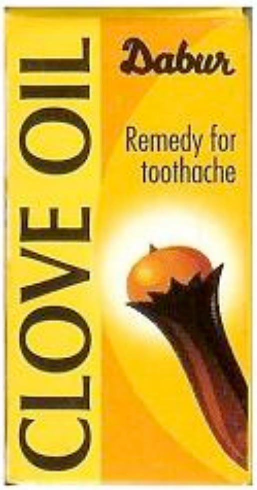


Cloves

- Cloves are used in the cuisine of Asian, African, and the Near and Middle East countries, lending flavor to meats, curries, and marinades, as well as fruit such as apples, pears or rhubarb.
- Cloves may be used to give aromatic and flavor qualities to hot beverages, often combined with other ingredients such as lemon and sugar. They are a common element in spice blends such as pumpkin pie spice.



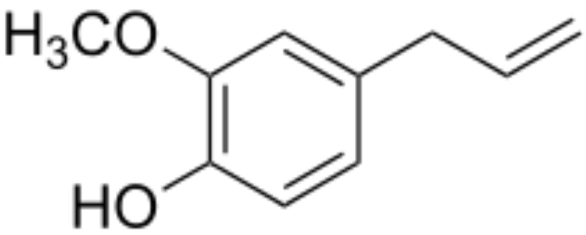
Clove Oil



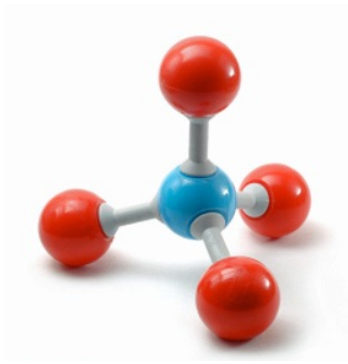
- Essence of cloves has antimicrobial, antifungal, antiseptic, antiviral, and stimulant properties.
- The oil is used for treating a variety of health disorders.
  - toothaches
  - indigestion
  - cough
  - asthma
  - headache
  - stress
- The most common use of clove oil is dental care. Several toothpastes, mouth wash and oral care medications contain clove oil as a primary ingredient.

Eugenol

- Eugenol comprises 72–90% of the essential oil extracted from cloves and is the compound most responsible for clove aroma.
- Eugenol is primarily responsible for the properties, flavor and scent associated with clove essence.
- Eugenol is toxic in relatively small quantities; for example, a dose of 5–10 ml has been reported as a near fatal dose for a two-year-old child.



- Physical properties:
  - Aromaticity: strong
  - Boiling Point: 259 ˚C
  - Water solubility: very soluble



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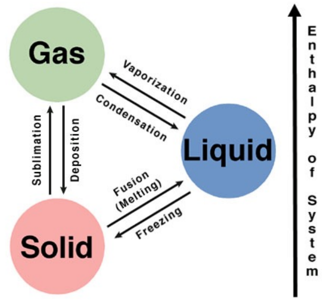

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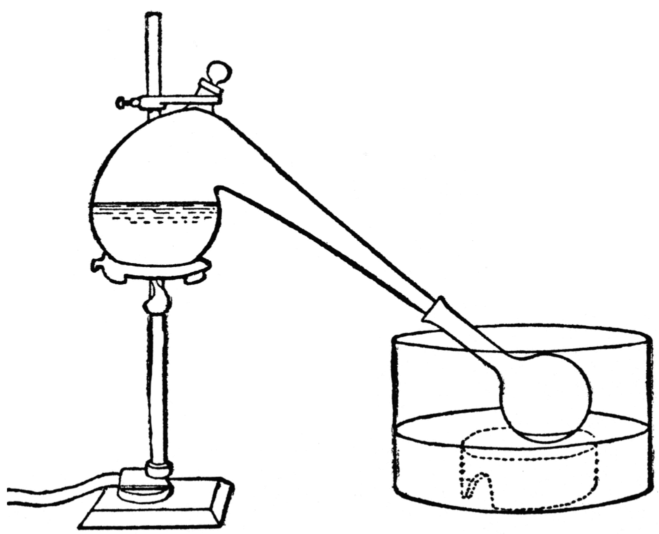
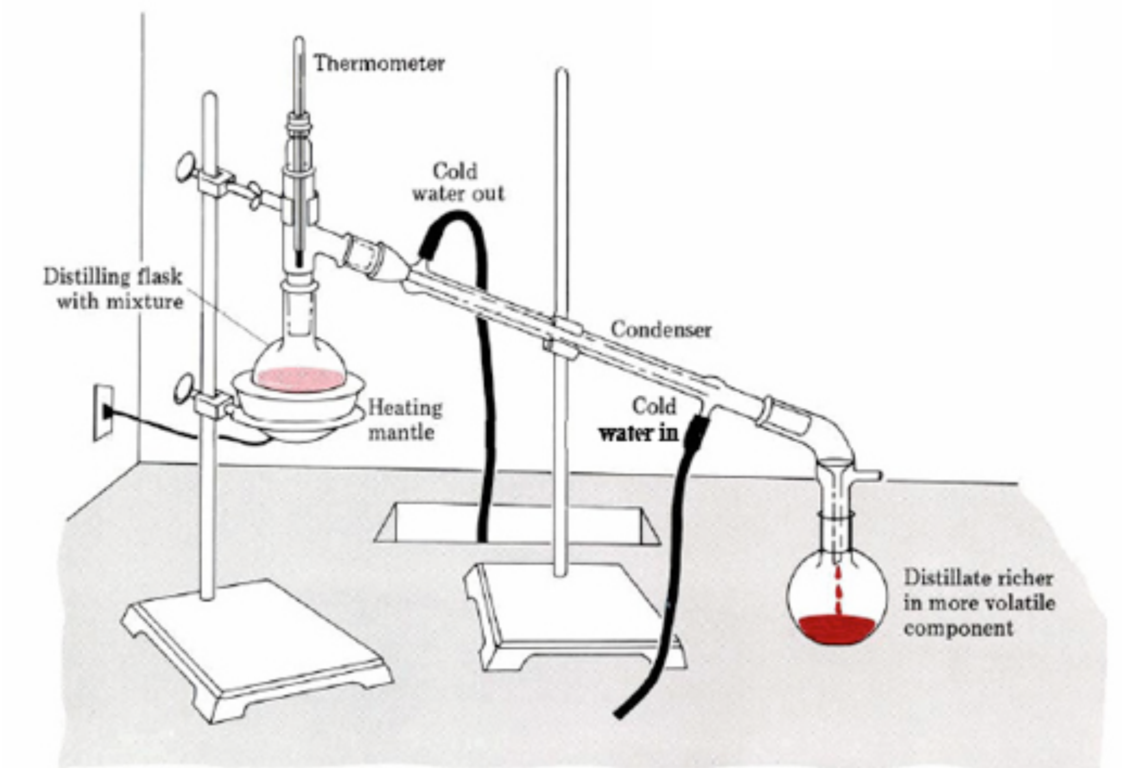
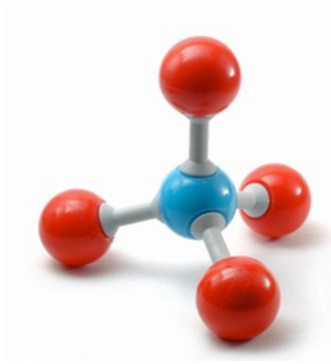


Essence of Cloves

- OBJECTIVE: To isolate eugenol from cloves by distillation and extraction.

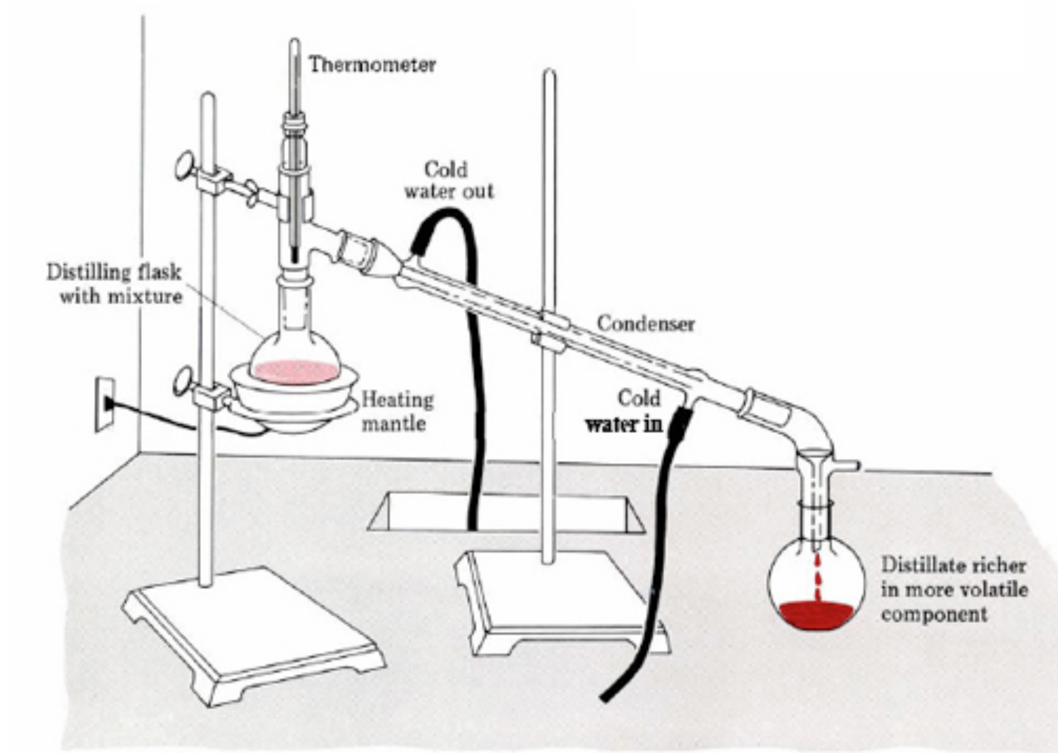
- GOAL: To explore distillation as a separation technique and get hands on experience with operating a chemical still.



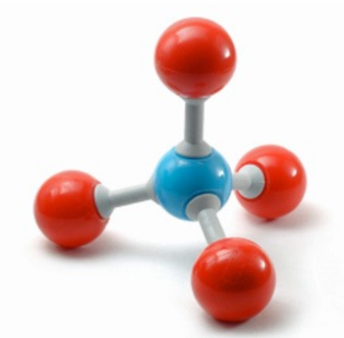


Essence of Cloves

- Setup
  - In the hood, assemble the:
    - distillation flask
      - on a sand bath
    - distillation head
      - secure with clamp
    - condenser
      - secure with clamp
    - collection flask
      - secure with clamp

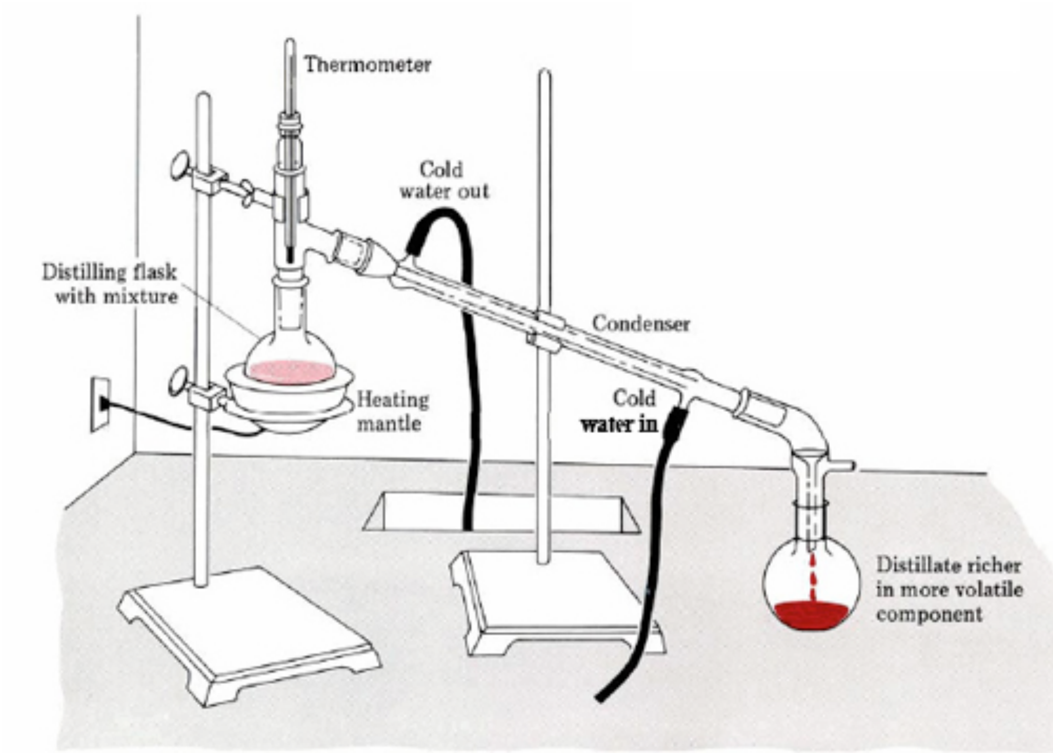


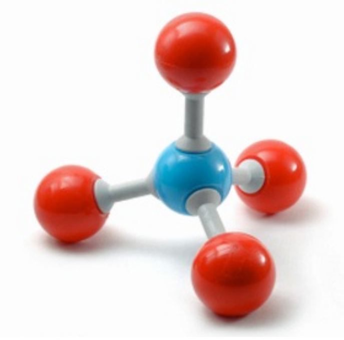
  - Add to the distillation flask:
    - 15 mL deionized water
    - mag stir bar
    - With stirring slowly add to this:
      - 1.0 grams ground cloves



Essence of Cloves

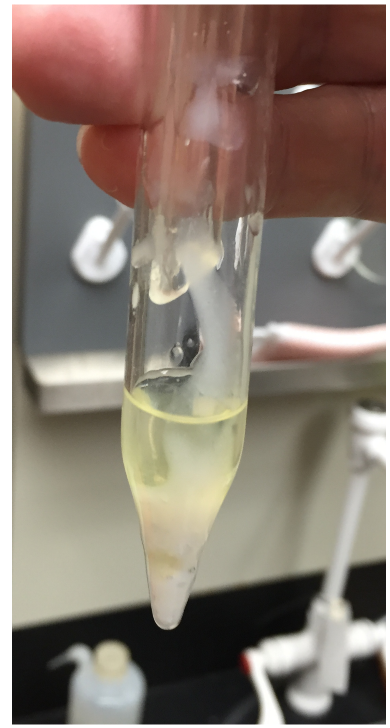
- Distillation
  - Add a second (electronic) thermometer to the sand bath.
  - Begin heating to a sand temperature of 130 ˚C.
  - Monitor and record the temperature of the distilled gas and collect 5-8 mL of the essence of cloves.

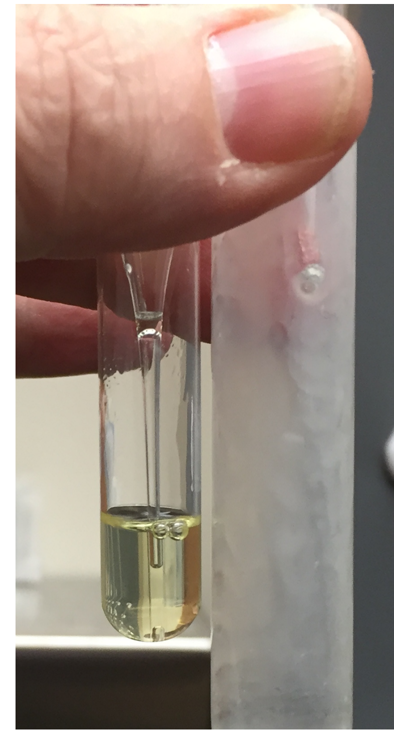


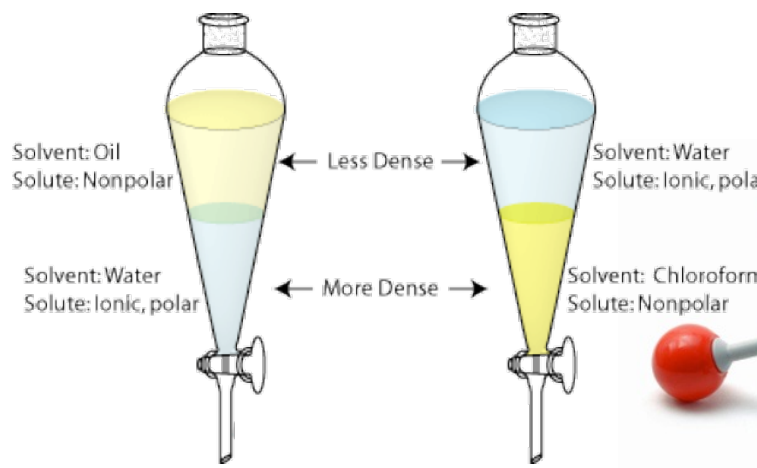


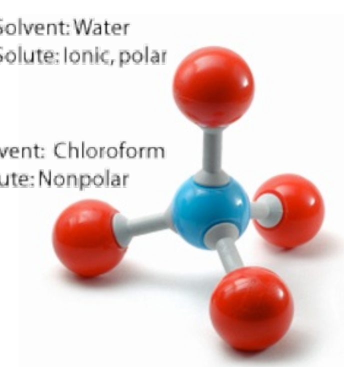
Essence of Cloves

- Extraction
  - Transfer the condensed liquid to a sep funnel or screw cap vial.
  - Add 2.0 mL of methylene chloride
  - Shake lightly
    - vent
  - Shake vigorously
  - Transfer the methylene chloride to a clean dry centrifuge tube
  - Repeat with two 1.0 mL portions of methylene chloride.
  - Collect methylene chloride fractions into a clean dry centrifuge tube.



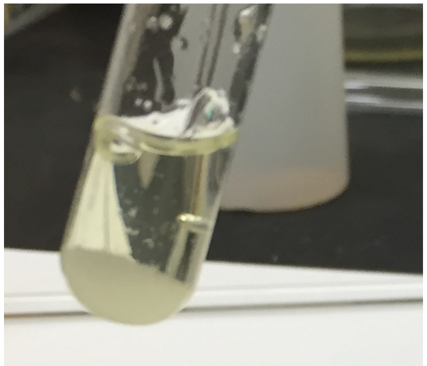


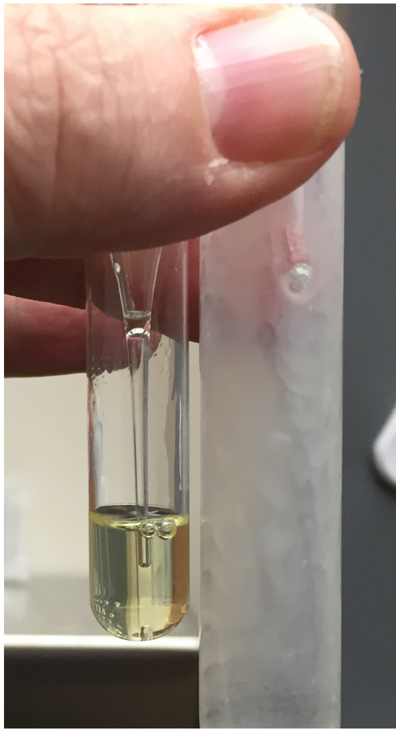


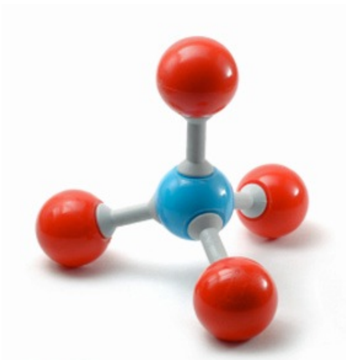


Essence of Cloves

- Drying:
  - Using the small end of the spatula, add one measure of sodium sulfate (Na_2SO_4) to your extracts.
    - Look for clumping of sodium sulfate (swirl tube or stir with clean dry micro spatula).
  - Continue adding small quantities of Na_2SO_4 until clumping occurs.
  - You will probably need 2-5 small measures of the drying agent.
    - If you use too much, you may have difficult removing the dried liquid and will get a poor yield.
  - Let solution dry at least 15 minutes.
    - Stir slightly every 2-3 minutes to increase the exposure to the drying agent.
  - Remove the dried solution by pipet to a try pre-weighted 5 mL conical flask.







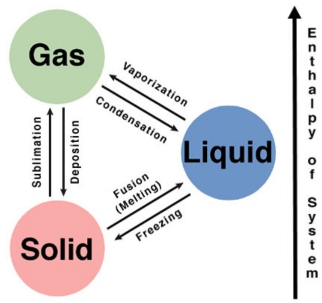
Essence of Cloves

- Concentration:
  - Evaporate the solvent using a stead stream of air.
    - DO THIS IN THE HOOD
  - Methylene chloride is much more volatile than you oil, it will evaporate quickly.
  - When the volume change seems to stop, end the evaporation. Your oil has some volatility, even at room temperature.
- Report the recovered yield for

Ex15

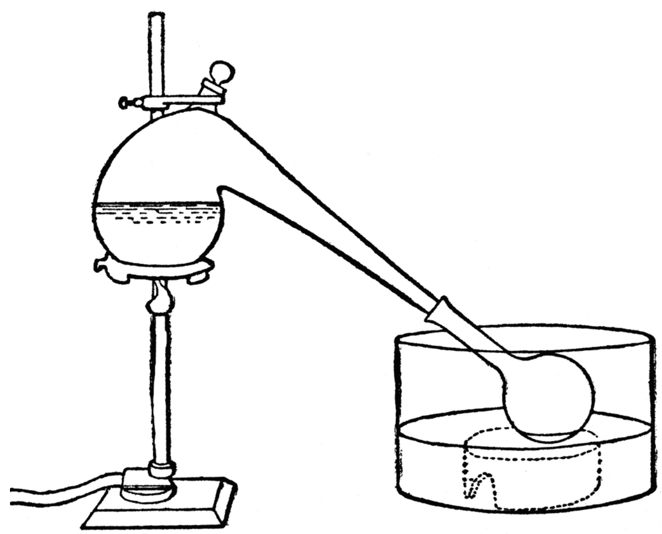
Essence of Cloves

- Distillation
  - Vaporization
    - Boiling Point
  - Simple Distillation
  - Steam Distillation
    - Vapor Pressure
- Clove Essence
  - Background
    - Properties



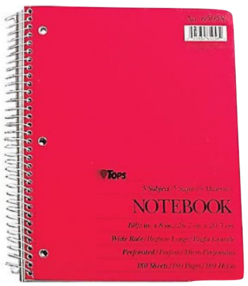
- The Experiment
  - Preparation
  - Distillation
  - Condensation
  - Analysis

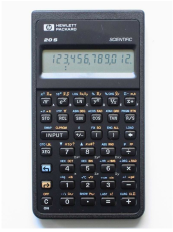
For Next Week

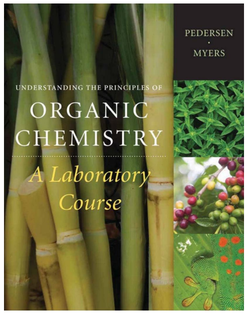


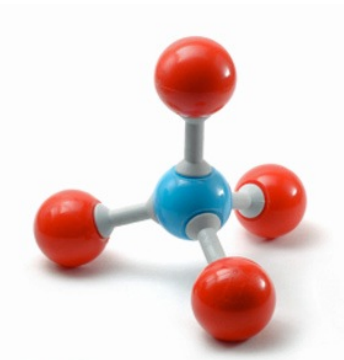
Next Meeting

- 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 23: Prep of t-Pentyl Chloride
    - Alkyl Halides in your lecture
  - Produce and bring to class:
    - Your pre-lab for exp 23 (p100)
    - Your procedure summary for exp 23
  - This prelab should include the chemical scheme including the structure and quantities of all reactants used (example on next slide).



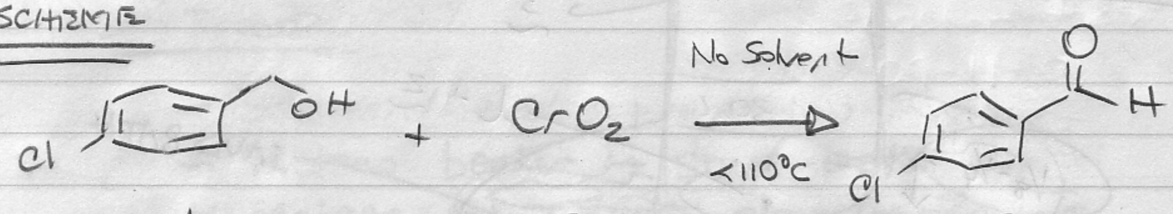






Exp 12: Magtrieve Oxidation
(p 268)

SCHEME



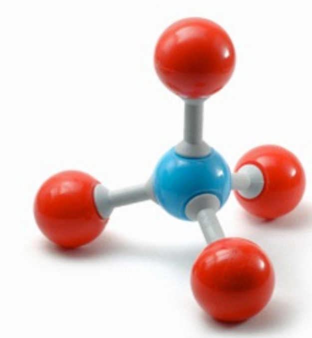
1	2	3
4-chlorobenzyl alcohol	Magtrieve™	4-chlorobenzaldehyde
C_7H_7ClO	CrO_2	C_7H_5ClO
142.58 g/mol	84.00 g/mol	140.56 g/mol

Theory {

1.0 mol	8.3 mmol	1.0 mmol
143 mg	700 mg	141 mg
1 :	8.3 :	1

White powder, flake crystaline	Black Tetehedral xtal	
mp 67–70°C bp 234°C	mp 375°C	mp 46 bp 60°C
Not significanly toxic	Insoluble in Water	skin irritant, wash w/ H_2O if exposed.
Pungent Odor		
Light sensitive	Can be regenerated by heating to 350°C.	Acute toxicity if swallowed.

Note: Get extra watch glasses and spotting dishes.
Condensation starts ~ 80°C.



Questions?