Separation of Toluene and Hexane by Distillation and Gas Chromatography

Part 1, p. 133 : Simple distillation.(4th Ed. p. 129)
Part 2, p. 133: Fractional distillation (use same procedure as simple).
Part 3, p. 206: Separation by gas chromatography (4th Ed. p. 202)

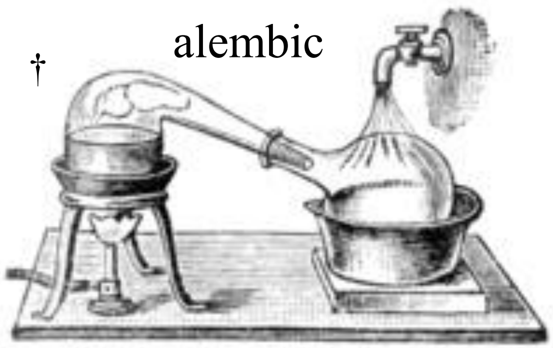
Important Concepts

- Theory of Distillation
- Distillation Setup/Equipment
- Dalton's Law & Vapor Pressure
- Raoult's Law
- Theoretical Plates
- Fractional vs. Simple Distillation
- Technique of Gas Chromatography

History of Distillation

Evidence of distilled spirits in Babylonia (ca 4000 BC)

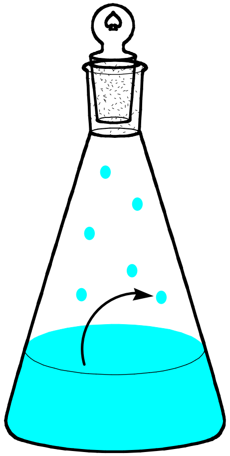
Jabir ibn Hayyan [aka Geber] (721-815) Born in Persia (Iranian). chemist, alchemist, astronomer, philosopher, physician.



Oil Refineries

Photo Source: † - Wikipidia

Distillation Theory



Vapor Pressure: a measure of the ease with which a molecule escapes from the liquid phase into the gas phase.

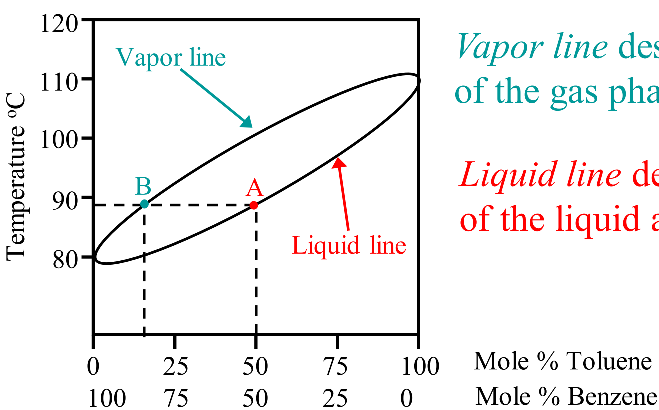
High vapor pressure → more molecules in the gas phase → Volatile compound (Low boiling point)

Note: Vapor pressure increases with temperature!

Dalton's Law: the total pressure is equal to the partial pressures of each component in the gas phase [$P_{tot} = P_x + P_y + \ldots$].

Raoult's Law: the vapor pressure (P_x) depends on the mole fraction (N_x) of each component [$P_x = P_x^o N_x$], where P_x^o is the vapor pressure of pure X at a given temperature.

$$N_x = \frac{nX}{nX + nY + nZ + \cdots}$$



Temperature composition diagram
For a mixture of benzene and toluene

Vapor line describes the composition of the gas phase.

Liquid line describes the composition of the liquid and the b.p.

At 89 °C the liquid in the stillpot is ~50:50 benzene: toluene, whereas the vapor phase is ~80:20 benzene: toluene. Thus, the vapor phase is enriched in the lower b.p. component.

Procedural Details

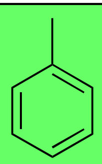
- Follow procedure on p. 133 (4th ed. p. 129) for both fractional and simple distillation, just use different apparatus. Work in pairs, one student does simple and the other does fractional.
- Use hexane instead of cyclohexane.
- Do not use a packed Hempel column for fractional distillation, instead use the apparatus described here.
- Collect three fractions for each distillation .

  Fraction 1: collect hexane until temperature drops; *note that the temp may not be accurate for the simple distillation.*

  Fraction 2: collect until temperature rapidly increases to ~100 ^{o}C; *note that the temp may never reach that for the simple distillation.*

  Fraction 3: collect after 100 ^{o}C until the still pot is nearly empty. Do not distill to dryness.

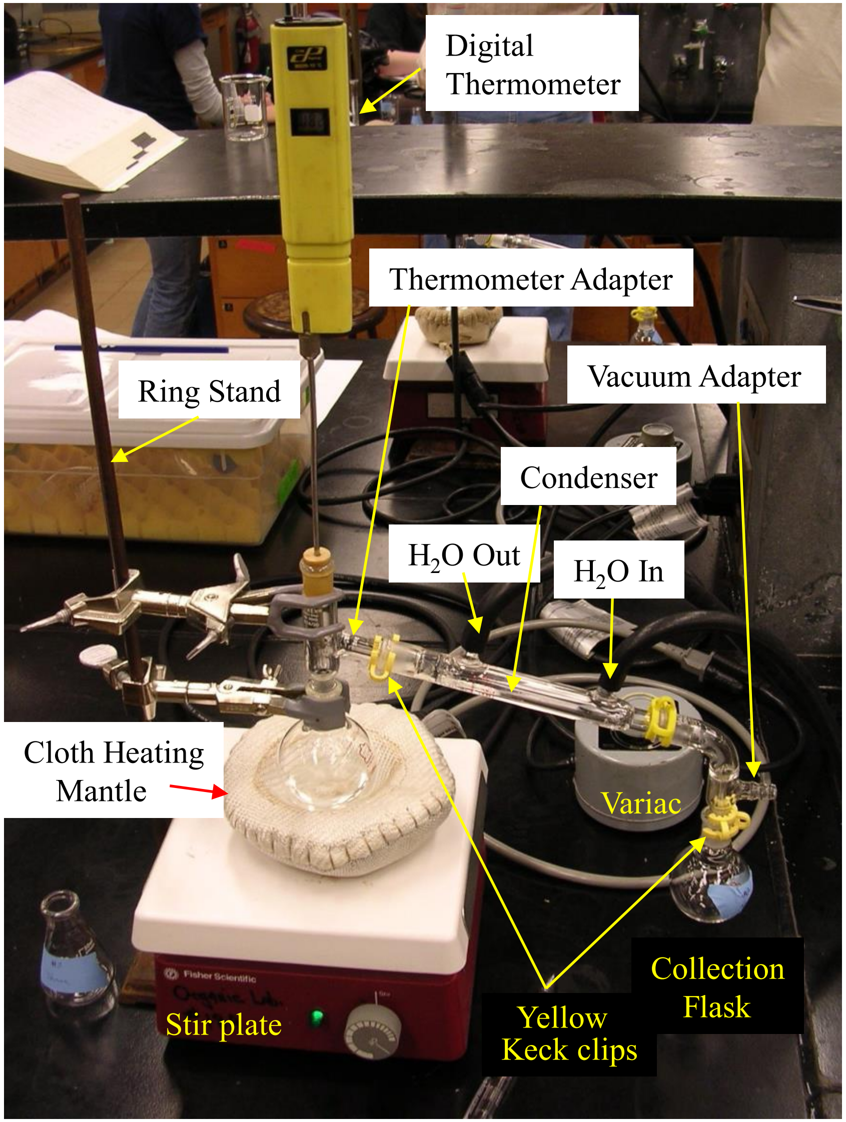
- Record b.p. range for each fraction.
- Perform GC on original mixture and all three fractions; get copies of your partners data for the worksheet.

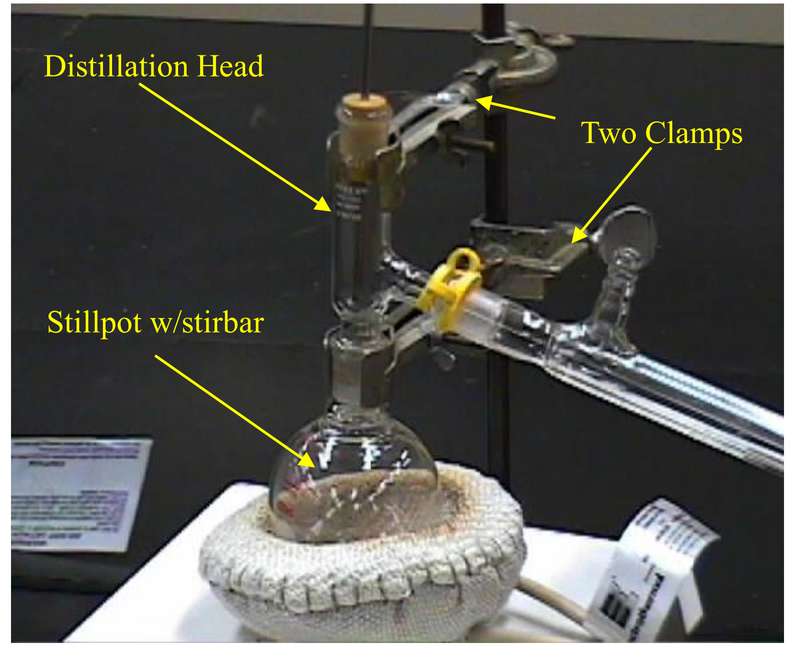


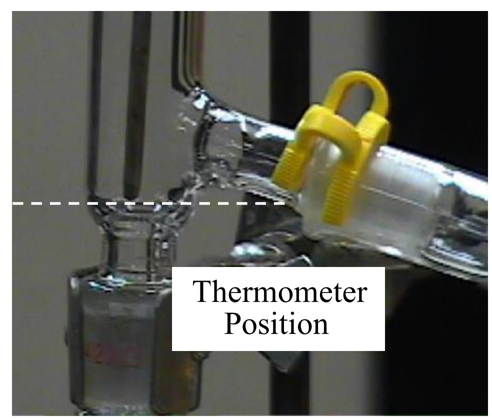
Toluene
b.p. ~ 110 ^{o}C

Hexane
b.p. ~ 69 ^{o}C

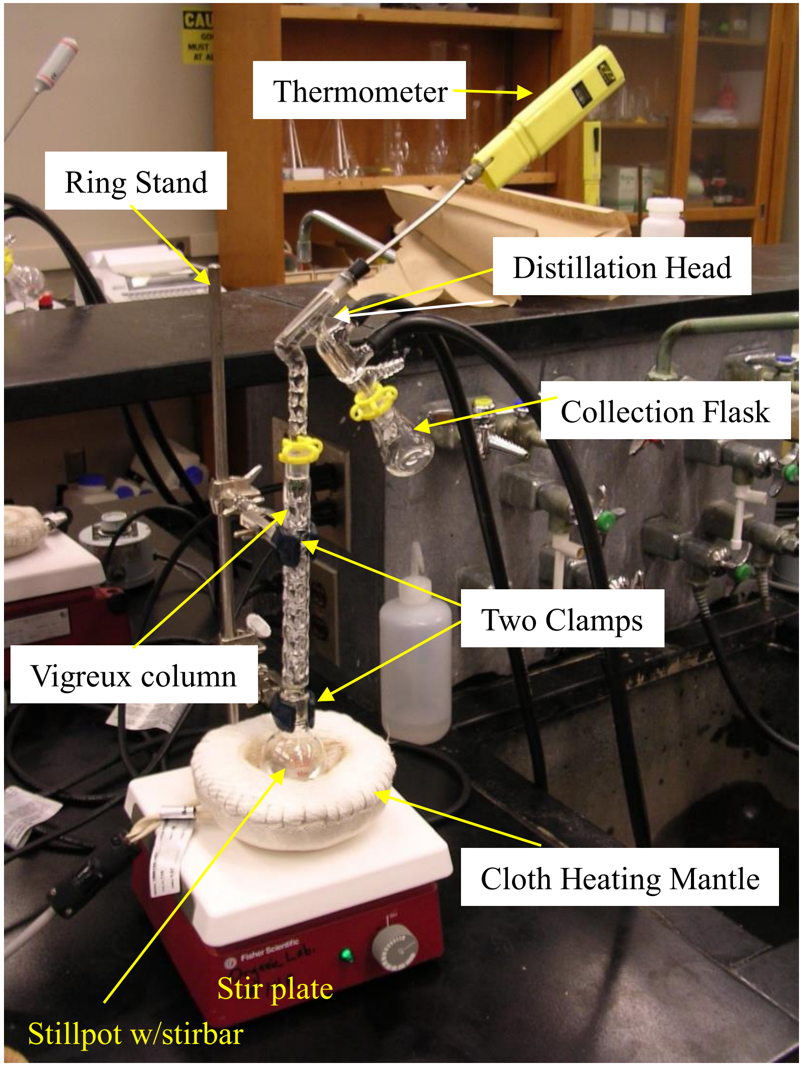
Simple Distillation Apparatus

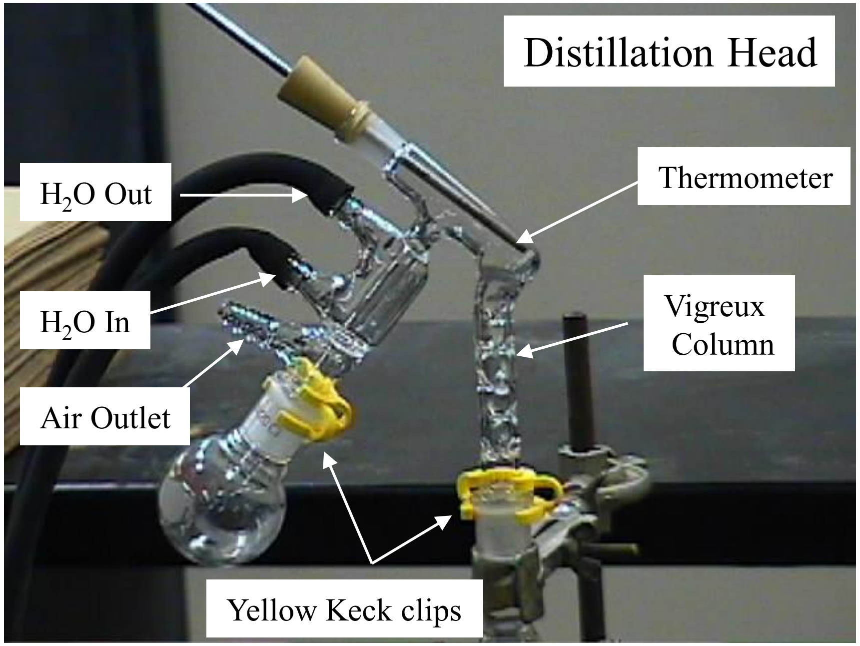






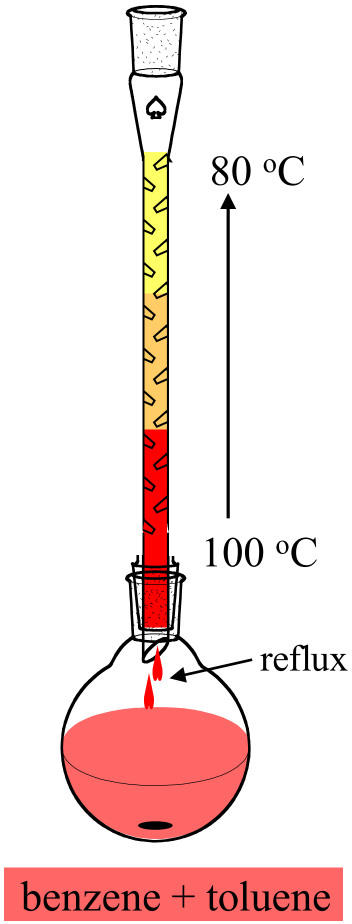
Fractional Distillation Apparatus





Why is fractional distillation more efficient?

The vigreux column provides more surface area for condensation to occur. At each condensation event the vapor is enriched in the low b.p. component and the liquid is enriched in the high b.p. component. We call these surfaces where condensation occurs *theoretical plates*, so the more theoretical plates, the more efficient your separation.



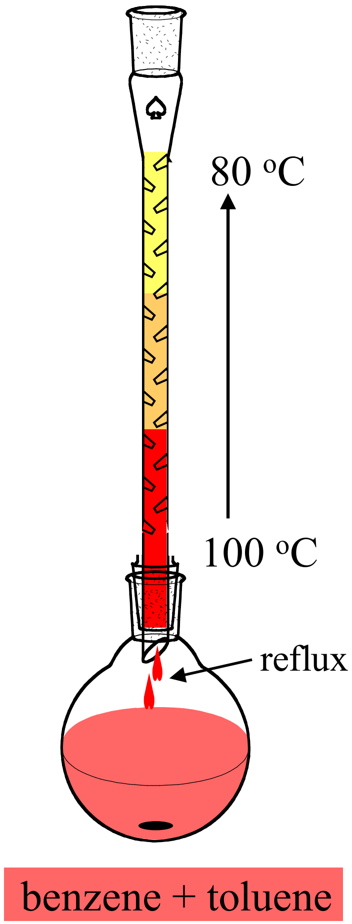
In order to get a good separation (i.e. high efficiency) we must distill at a moderate rate. Therefore we cannot add too much energy (i.e. heat).

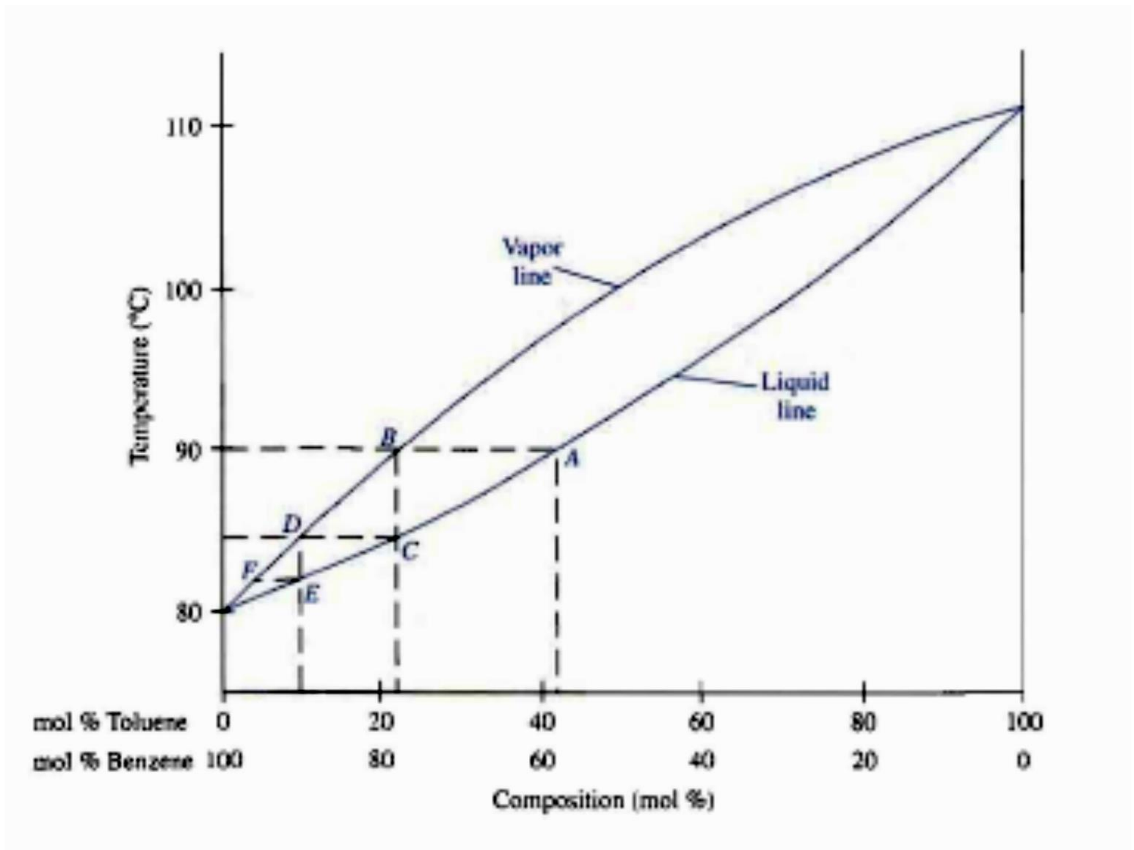
Imagine if 100 people were in a 2 kilometer race. If everyone were to get into a car and drive, then they would all arrive very close to each other. However, if they all ran instead, then their would be a good distribution of runners over the course of the race, and the fastest ones would arrive well ahead of the slower runners (i.e. they would be separated).

A simple distillation has fewer theoretical plates, so the temperature gradient is not as dispersed and the separation is poorer.

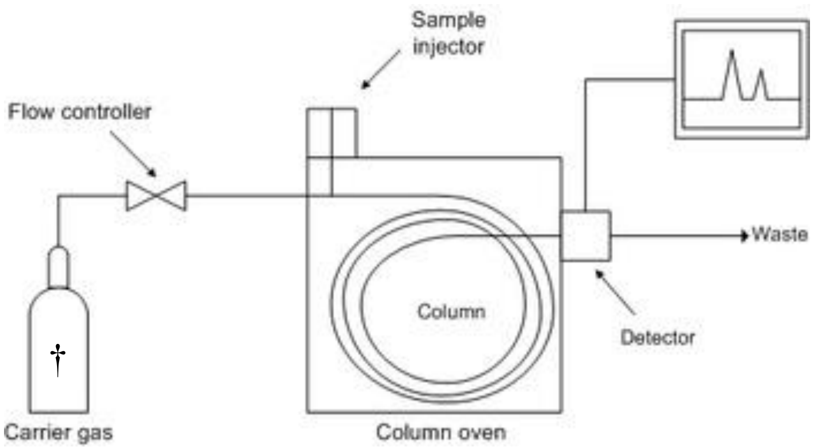
Only use the simple distillation for compounds that have a large difference in b.p. (e.g. purifying sea water).

Why is fractional distillation more efficient?





Gas Chromatography



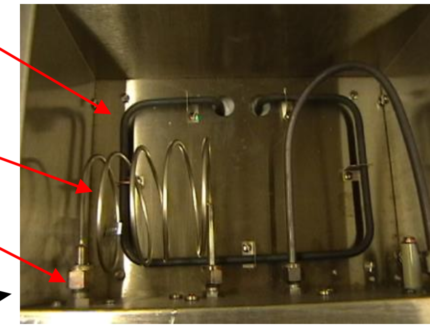
The column is packed with tiny beads that are coated with a viscous liquid. The vaporized compound travels down the column and adsorbs onto the column packing. Normally the carrier gas (or eluant) is helium.

Top of oven

Heating Element

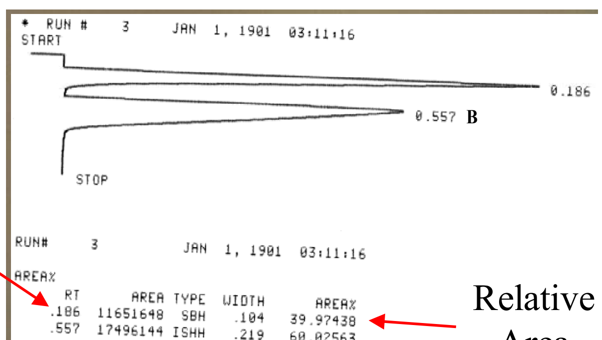
GC column

Injector port



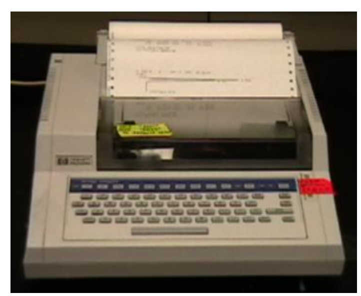
Use injector port A

Retention Time

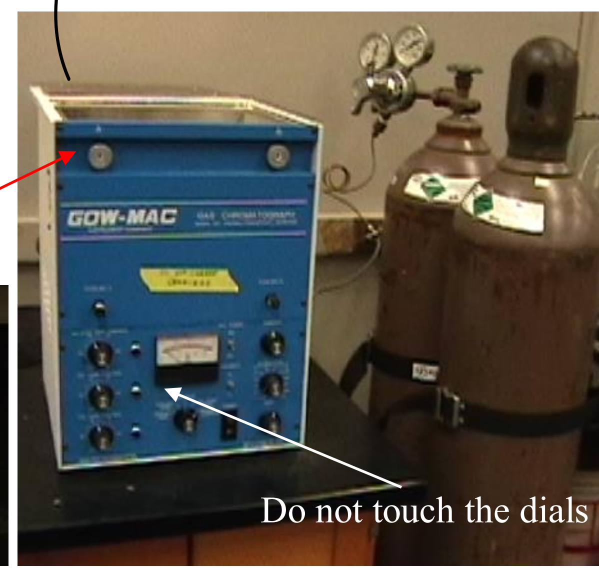


Relative Area

GC chromatogram



Printer



GC with He tank

Which peak is due to toluene?

Photo Source: † - Wikipidia

Safety Issues

- hexane and toluene are highly flammable.

- Never heat a closed distillation system. Always make sure there is a vacuum or air outlet.

- the glassware will get very hot.

- do NOT distill to dryness as this may leave solid residues, which could explode if there were peroxides present.

- the GC needles are very sharp and delicate. Inject carefully so as not to bend the needle