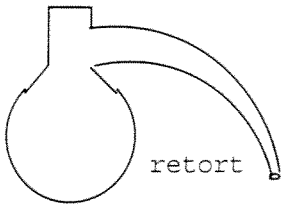
Simple and Fractional Distillation
Dr. Gergens - SD Mesa College

Distillations



1. most important and widely used process for purifying liquids
2. used to separate volatile components from nonvolatile (salts)
3. simple distillation for liquids of boiling point differences of 100°C or greater
4. fractional distillation for liquids of boiling point differences of 40°C or greater
5. smaller boiling point differences if the mixture is 90% pure with 10% crud
6. effective for rapid purification as a pre-purification step

Safety Considerations

1. never fill beyond two-thirds full
2. never distill in a closed system
3. never distill to dryness
4. never distill without a boiling chip—only one—or stir bar w/stir plate
5. never distill with an open flame
6. never use cracked glassware; check all glassware for cracks
7. secure all glassware with rubber bands or blue joint clip
8. run condenser water, but not with deionized water
9. cloth heating mantels are plugged into a transformer, not the wall outlet

Buzz Words

1. still - apparatus for distillation
2. still pot - appropriate size, start at 2/3 full
3. bumping - violent eruptions, use a stir bar or boiling chip
4. still head and thermometer placement
5. distillate - condensed liquid
6. receiving flask - clean and dry
7. forerun - discard lower boiling point fraction
8. takeoff - rate of entry 1-3 drops/second into the receiving flask
9. pot reside - don't distill to dryness
10. holdup - flask and head

Raoult's Law

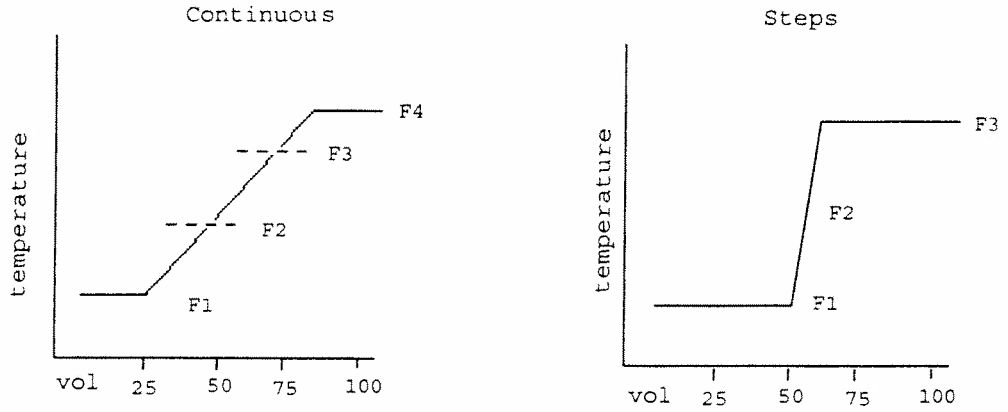
1. ideal solution - two miscible liquids that do not react with each other

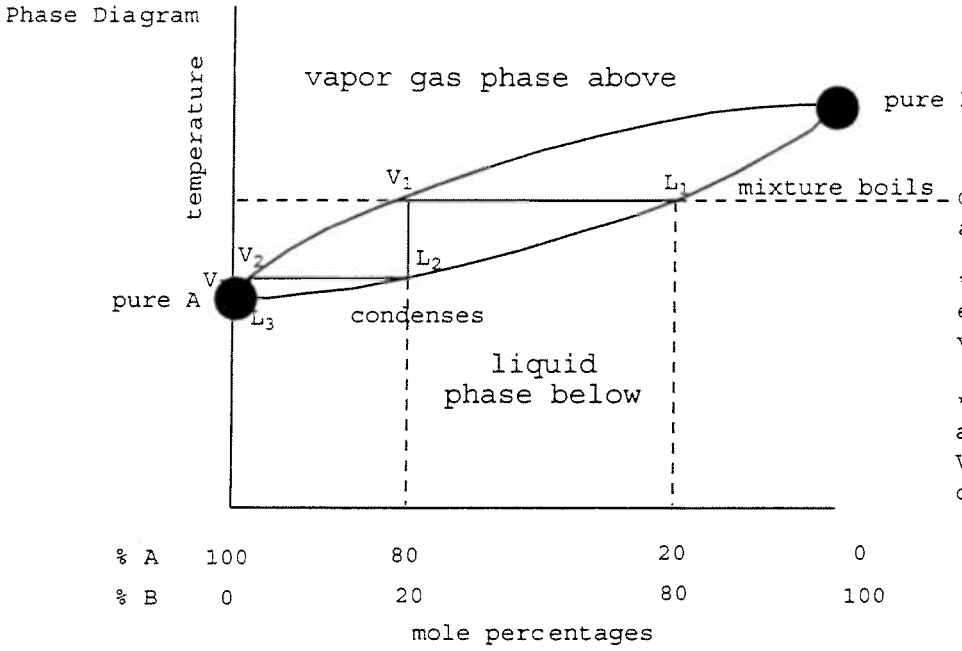
simple or fractional distillation

$$P_T = X_A P°_A + X_B P°_B$$

$$X_A = \frac{\text{moles A}}{\text{moles A + moles B}} \qquad X_B = \frac{\text{moles B}}{\text{moles A + moles B}}$$

Temperature versus Volume Graphs





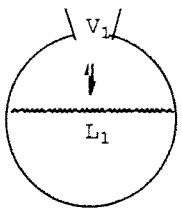
@ a constant temperature and composition

*the horizontal is L_1 to V_1 equilibrium phase @ cont T of variable compostion

*the vertical line is a condensation phase, where V_1 and L_2 would have the same composition

Simple Distillation

L--->V at constant temperature Liquid Vapor Liquid (LVL) cycle for simple distillation affords one theoretical plate

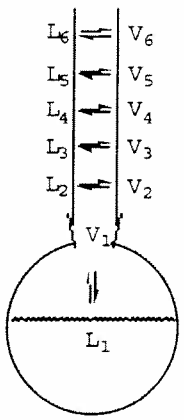


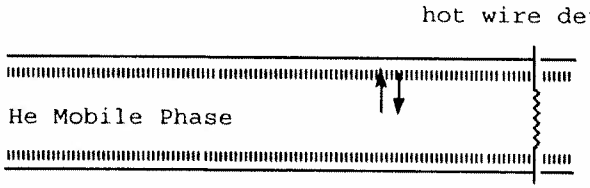
To separate a mixture of boiling point difference of

Δ difference in boiling point	Theoretical Plates
108	1
36	5
20	10
10	20
2	100

Fractional Distillation

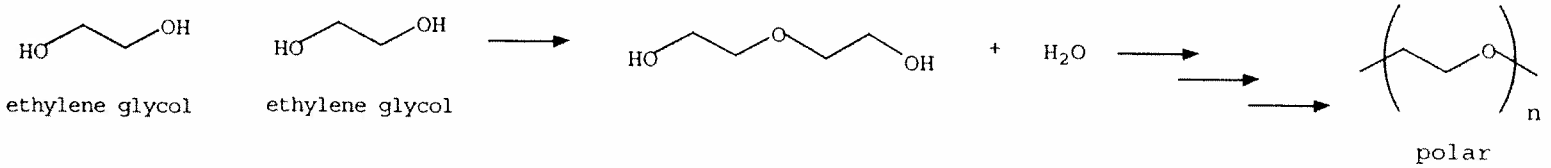
There are several LVL cycles that occurring within a fractionating column. Each cycle produces a mixture enrich with the lower boiling component.





Liquid Stationary Phase (several types with varying polarity)

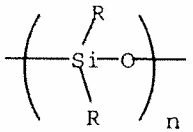
A. Carbowax (polyethylene glycol, PEG) which is a polyether



ethylene glycol ethylene glycol

polar

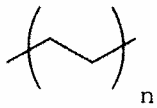
B. Silicone Wax



semi-polar

R = CH_3

C. Apiezon Grease (hydrocarbon mixtures) and Nujol (mineral oil)

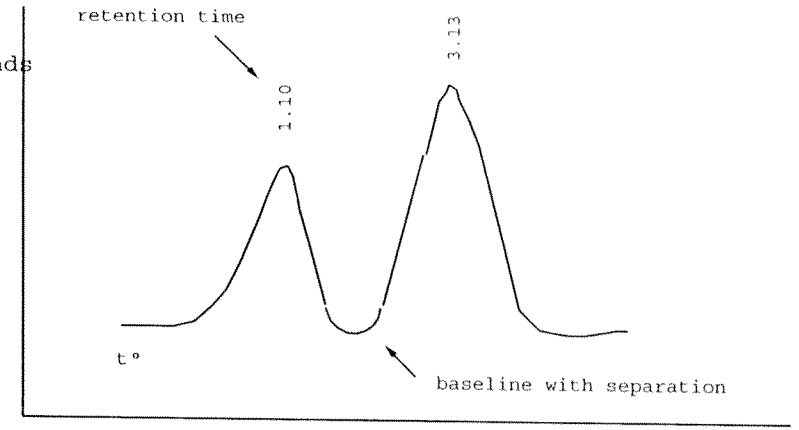


nonpolar

Separating Power					
Polarity	carbowax	>	silcone wax	>	Apienzon & Nujol
typically use to separate	alcohols ethers		aldehydes ketones halocarbons		hydrocarbons steroids pesticides

Retention Times (time spent on the column). Affecting factors:

1. low boiling point compounds versus high boiling point compounds
2. carrier gas flow
3. length of column
4. bore size
5. column packing material
6. oven temperature



Advantages of this technique for separation and analysis

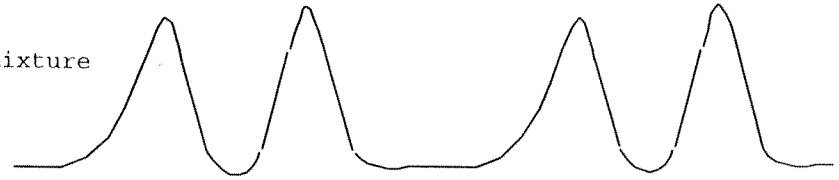
1. small sample size 0.2 - 1.0 microliter (μL)
2. quantity estimated quantitatively

After the Analysis, record the following data on the chromatogram: your intials, date, sample size, column material, oven, detector, & injection port temperature.

Detector Response Factor is used to make minor corrections (normalization) in peak area.

1. Determination of response factors requires one to prepare an equal molar mixture of compounds being analyzed.
2. An equal molar mixture of compounds should give peaks with equal peak areas, but this is usually not the case since every compound interacts differently with the detector.
3. Determine a response factor for each compound (peak) analyzed. Measure peak areas and divide by the smallest area.

analysis of equal molar mixture



Areas	$100cm^2$	$99cm^2$	$98cm^2$	$105cm^2$
dividing through by the smallest area affords the	$98cm^2$	$98cm^2$	$98cm^2$	$98cm^2$
corresponding response factors	1.02	1.01	1.00	1.07

4. For future analyses of mixtures containing the same compounds, use the calculated response factor to normalize each compound peak area by dividing measured peak area by its corresponding response factor.