

CHEM 108M, Lecture 1

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pl

Syllabus & Course Overview

- Exp 1 - Separation of Limonene & Carvone from Spearmint or Caraway

Column Chromatography - Mohrig 18
Analysis via - Polarimetry, GC, & IR

Spearmint & Caraway
(-)-S-Lim (+)-R-Lim
(-)-R-Carv. (+)-S-Carv

(-) = l, levorotatory
(+) = d, dextrorotatory

* no correlation b/w

R/S & d/l
predict can't predict
exp. obs only

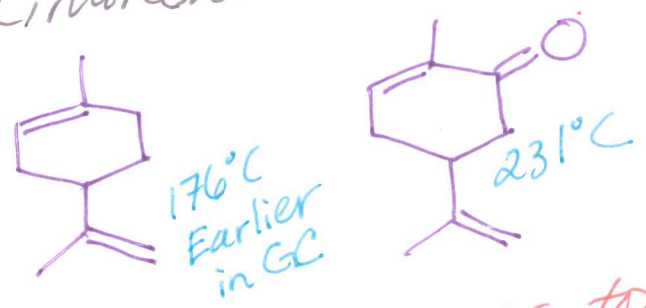
α_{obs} = observed rotation

$[\alpha]_D^{20}$ = specific rotation @ 20°C, "D line" of Na

$$[\alpha]_D^{20} = \frac{\alpha_{obs}}{c \times l}$$

given in lab → c × l ← 2 dm

both contain
Limonene & Carvone



+ other terpenes too,
not investigated in this lab

3 GC Runs

① Crude Oil

② Fraction 1

③ Fraction 5

beg. end of separation from column, more on that soon!

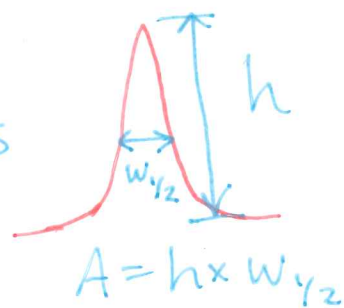
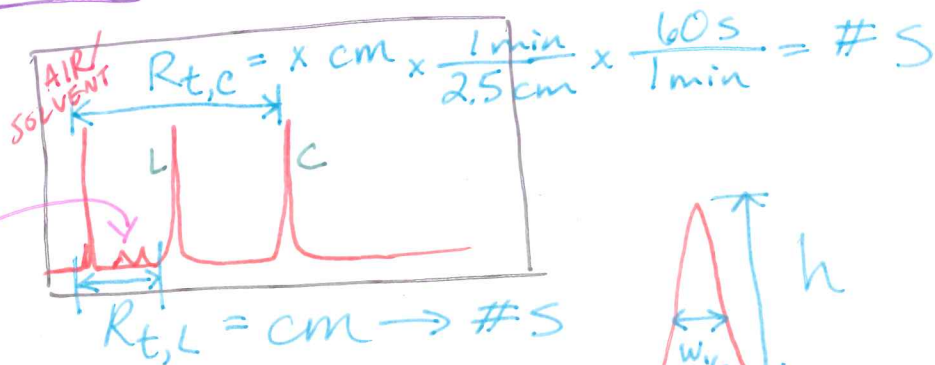
GC Reminders

- Ovens are hot!
- Inject no more than 0.2 μ L
- Needles are delicate
- Use acetone without salt (salted acetone = IR)

① Crude Oil

calculate area of other components (no R_t for ID)

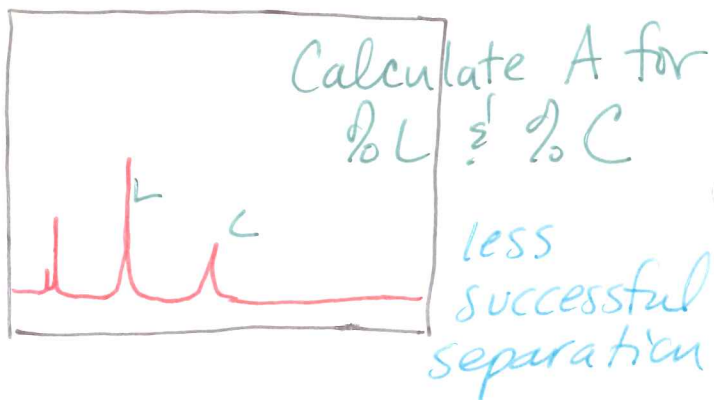
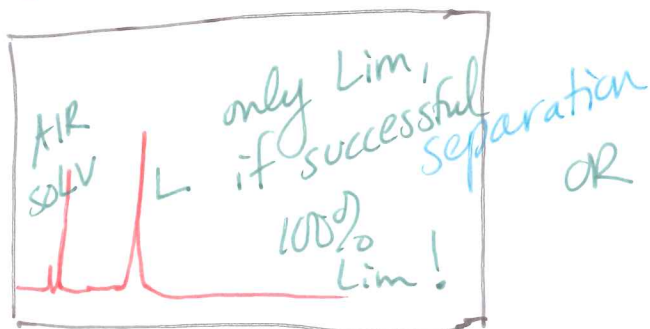
R_t from beginning of air to beg. of sample peak



$$\% \text{ comp. L} = \frac{A_L}{A_{TOT}}$$

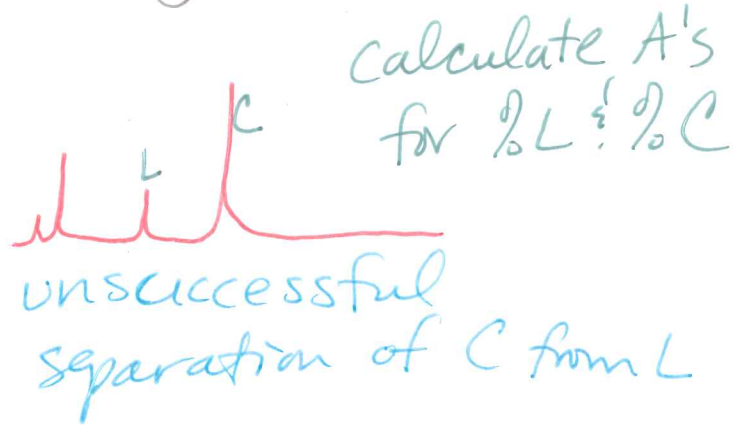
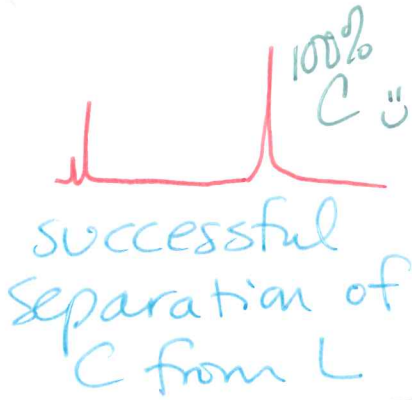
$$\% \text{ comp. C} = \frac{A_C}{A_{TOT}}$$

② Fraction 1 - should be only limonene



③ Fraction 5 - should be only carvone

LI
PB



COLUMN CHROMATOGRAPHY

very similar to TLC, except it's upside-down!

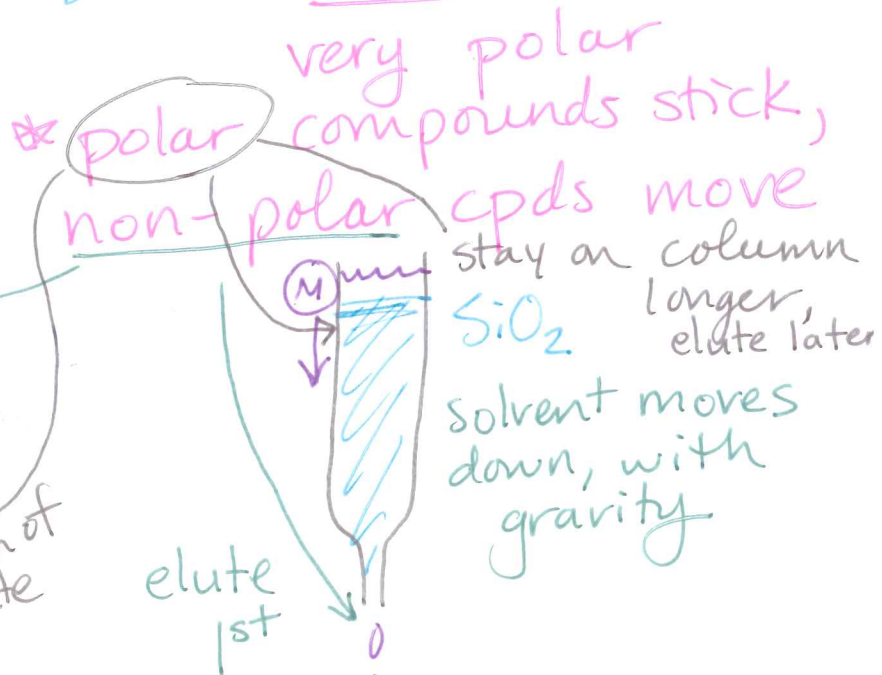
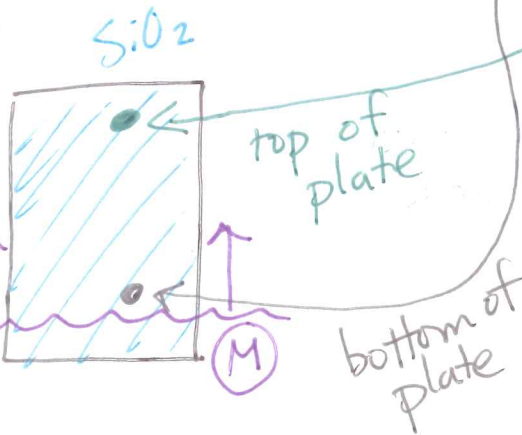
TLC
(Thin-Layer)

vs. Columns

Both absorption chromatography

(M) = mobile phase = solid SiO₂, silica

capillary action
moves up plate, against gravity
(solvent)



MOBILE PHASE

↳ controls speed, success, & degree of separation

Polar vs. Non-polar

faster movement,
generally less separation
* elutes polar compounds,
but non-polar compounds
move even faster b/c

slower movement,
more separation
* use this 1st to elute
NP cpds, then
switch to polar
solvent

⑤ SiO₂ is polar

General Polarity Scale

ROH
POLAR

RNH₂ / CC(=O)C
ex. Acetone

Hydrocarbons

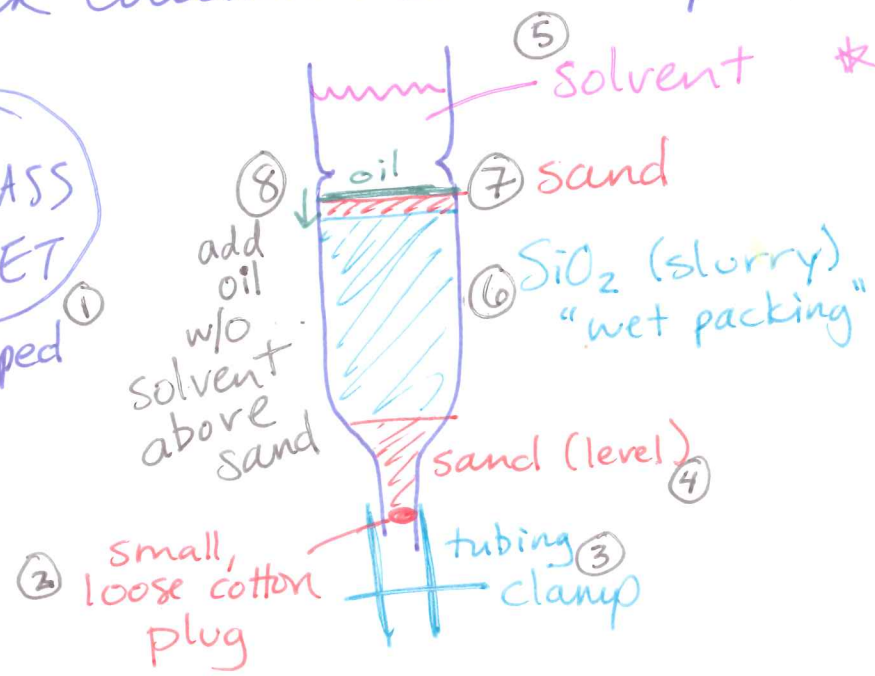
NONPOLAR
ex. Hexanes

② 3x2mL
10% Acetone in Hexanes,
elutes C

① 3x2mL portions,
quickly elutes L,
C stays behind

"Pack Column & Load Sample"

GLASS
PIPET
① clamped



* NEVER LET
COLUMN RUN DRY
(past sand on top)

"wet packing" with hexanes

Separation of Limonene and Carvone from Caraway or Spearmint Oil

more polar **A** Carvone CC1=C(C)C(=O)C=C1 ketone

nonpolar **B** Limonene CC1=CC=C(C)C1

longer 😊 → nonpolar hexanes

Solvent
Compound A
Compound B
Adsorbent
5:0₂
POLAR

Solvent eluted from column

(a) Mixture of compounds A and B at top of column

(b) Compounds A and B beginning to separate

(c) Compound A starting to elute from column

3 x 2 mL Hexanes
1 2 3
fractions

Fraction 1
some/all Limonene

switch to 10% acetone in hexanes (more polar solvent mixture)

solvent moves P component

10% Ac
3 x 2 mL in hex
4 5 6
fractions

F3-4 likely just solvent

fractions 5/6? Carvone

(d) Compound A collected

(e) Compound B starting to elute from column

(f) Compound B collected

fractions 1/2? Limonene
check by TLC

check by TLC

FIGURE 19.1 Stages in liquid chromatographic separation of a mixture containing two compounds. Compound A moves faster than compound B, which is more strongly adsorbed on the stationary phase.