CHEM 108M, Lecture 1

Syllabus & Course Overview

- Exp 1 - Separation of Limonene & Carvone from Spearmint or Caraway
  - Column Chromatography - Mohrig 18
  - Analysis via - Polarimetry, GC, & IR

Spearmint & Caraway both contain Limonene & Carvone

(-)-S-Lim
(-)-R-Carv.

(+)-R-Lim
(+)-S-Carv

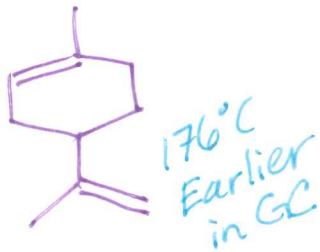
(-) = l, levorotatory

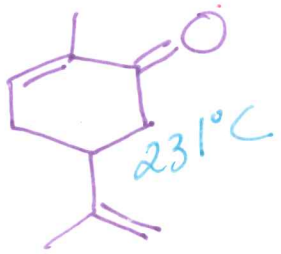
(+) = d, dextrorotatory

* no correlation b/w R/S & d/l

R/S: predict

d/l: can't predict, exp. obs only





+ other terpenes too, not investigated in this lab

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

3 GC Runs

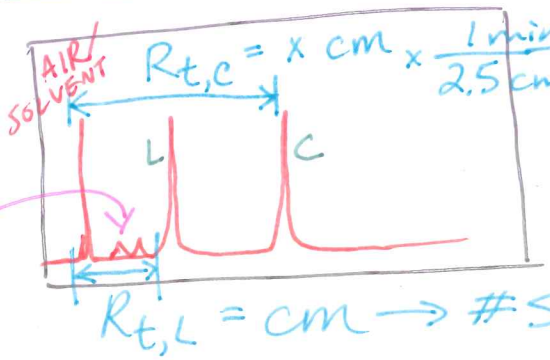
① Crude Oil ② Fraction 1 ③ Fraction 5

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

GC Reminders

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

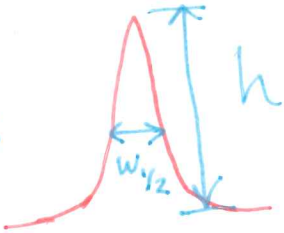
① Crude Oil



$$\text{x cm} \times \frac{1\text{ min}}{2.5\text{ cm}} \times \frac{60\text{ s}}{1\text{ min}} = \#\text{S}$$

calculate area of other components (no R_t for ID)

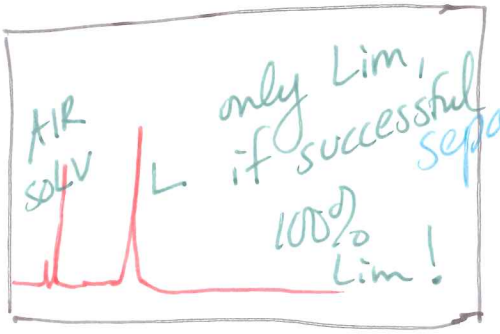
R_t from beginning of air to beg. of sample peak



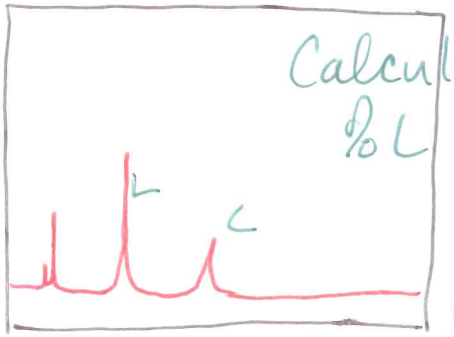
$A = h \times W_{1/2}$

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

② Fraction 1 - should be only limonene



OR

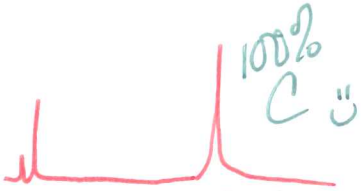


Calculate A for %L & %C

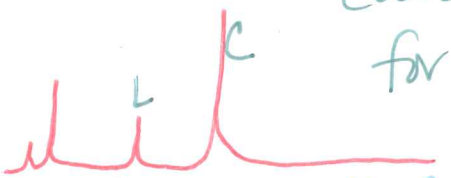
less successful separation

LI
PB

③ Fraction 5 - should be only carvone



successful separation of C from L



unsuccessful separation of C from L

calculate A's for %L & %C

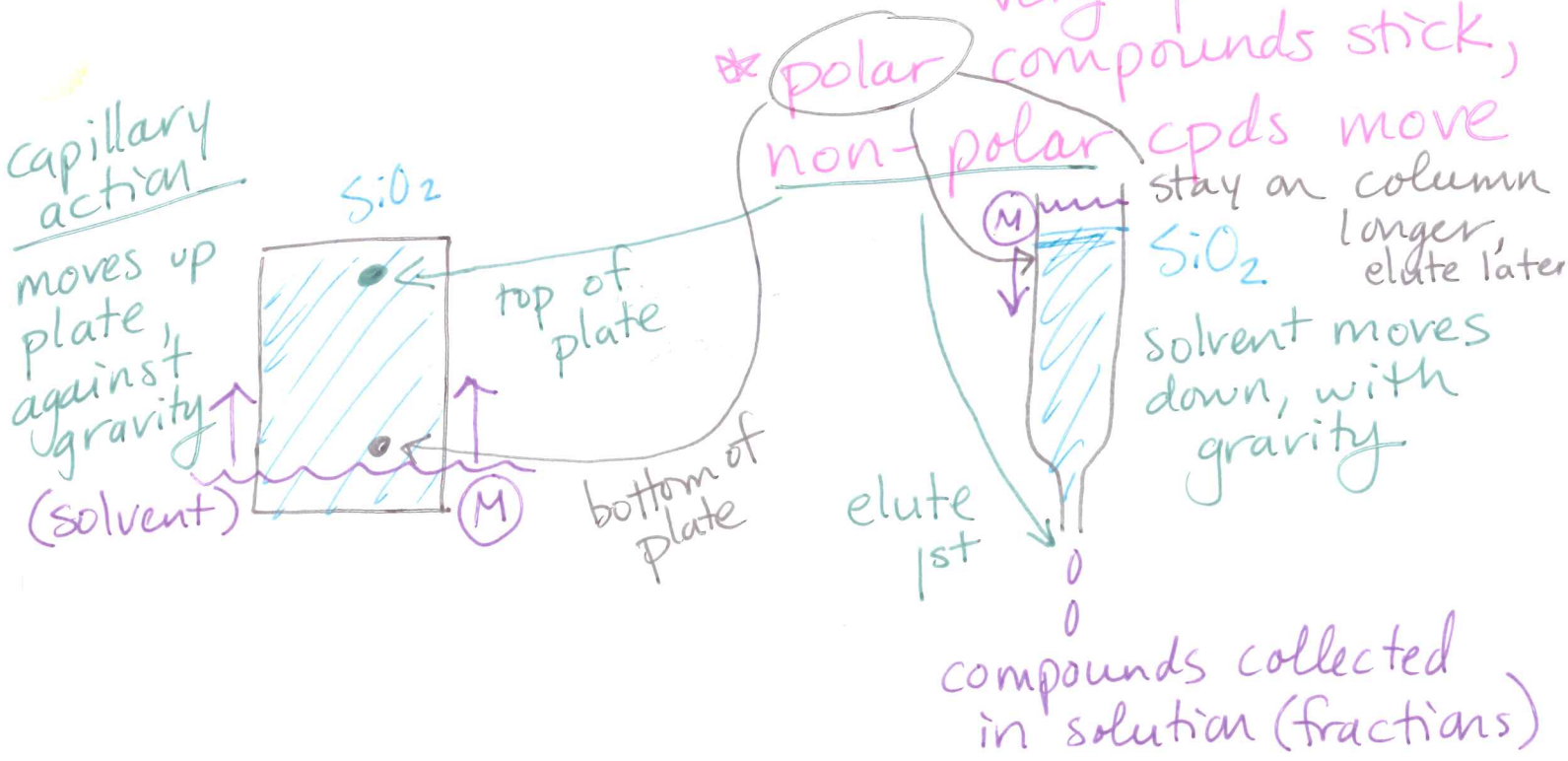
COLUMN CHROMATOGRAPHY

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

TLC (Thin-Layer) vs. Columns

Both absorption chromatography

(M) = mobile phase = solid SiO_2, silica



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
(5) SiO_2 is polar

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

General Polarity Scale

ROH — POLAR

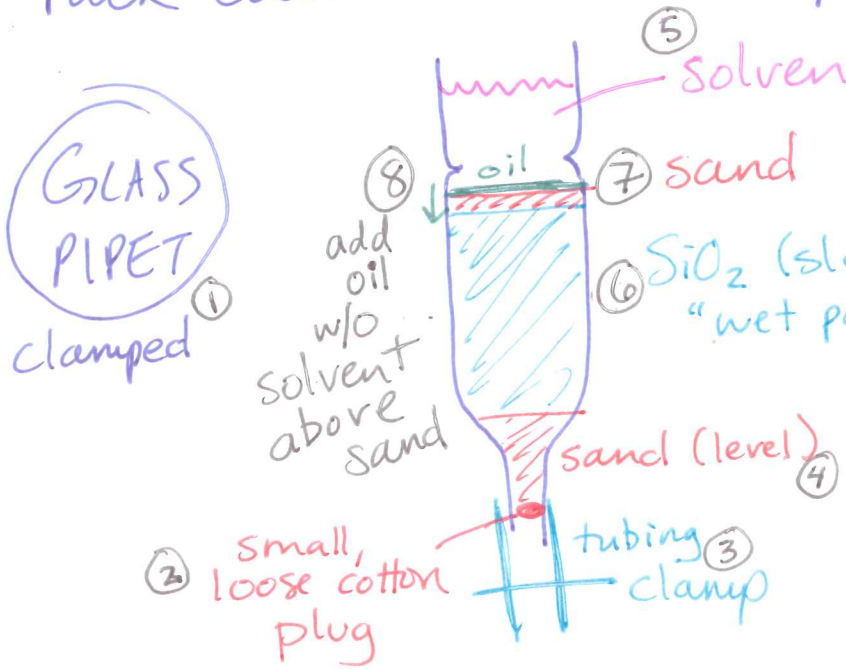
RNH_2 /  ex. Acetone

Hydrocarbons — NONPOLAR ex. Hexanes

(2) 3×2mL 10% Acetone in Hexanes, elutes C

(1) 3×2mL portions, quickly elutes L, C stays behind

"Pack Column & Load Sample"



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

Separation of Limonene and Carvone from Caraway or Spearmint Oil

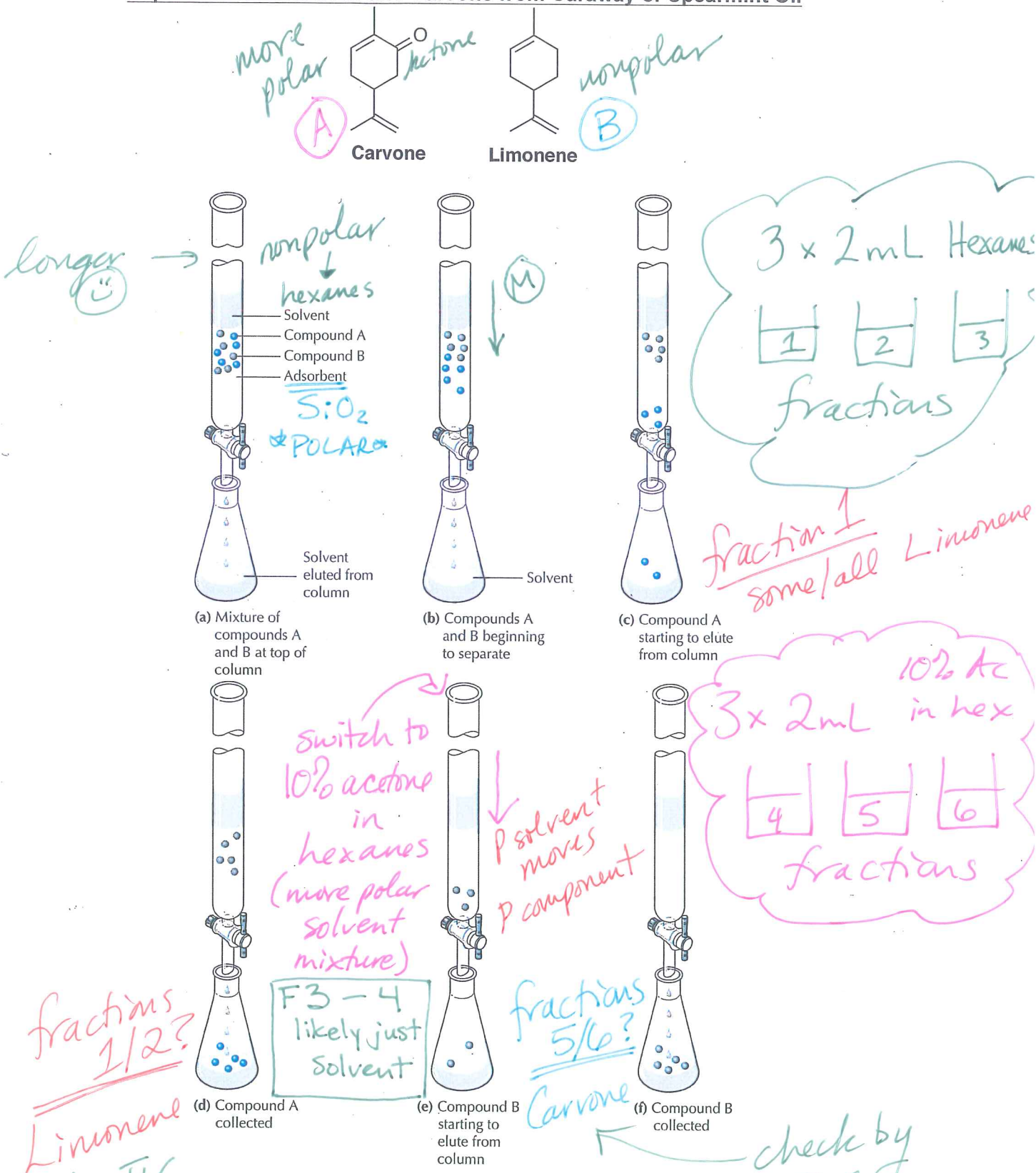


FIGURE 19.1 Stages in liquid chromatographic separation of a mixture containing two compounds. Compound A moves faster than compound B, which is more strongly adsorbed on the stationary phase.

Figure re-produced from Mohrig's *Laboratory Techniques in Organic Chemistry, 4th Edition.*