

Steam Distillation

Purpose:

Use steam distillation techniques to separate/isolate organic molecules such as essential oils

Materials

Blender
Boiling Stones
Condenser
Sep funnel
Thermometer/adaptor
10-ml syringe
Vacuum adaptor
Heating mantle
round bottom flask

Chemicals

| | | |
|--------------------|---|-----------|
| Anhydrous $MgSO_4$ | — | inorganic |
| Dichloromethane * | — | organic |
| Diethyl Ether * | — | " |
| 95% Ethanol * | — | " |
| Hexane * | — | " |
| Methanol *† | — | " |
| * Flammable | | |
| † toxic | | |

Hazards

- Hot Steam
- Dichloromethane — handle in hood
- Essential oils — irritant → wash cold water/soap

TS

Procedure (orange)

- 1) blend peels into slurry
- add to round bottom flask
- 2) Set up distillation
 - a) attach heating mantle to flask
 - b) attach R-B flask to stand and immerse into water
 - c) grease glass joints and attach 3-way side arm adapter
 - d) insert thermometer into adapter and place into top of side arm
- bulb should be just below side arm bend
 - e) attach condenser clamp (loosely)
 - f) connect water tubing to condenser (in bottom out top)
- 3) follow instructions for chilled water loop

~ Clave = 5.0 g.

Procedure

- 4) immerse Erlenmeyer flask in ice bath and place under receiving end
- 5) have TA check set up
↓
- 6) variac set to 80
- 7) wrap RB flask / distillation adapter with foil
- 8) adjust heat to allow 1-2 drops/sec
- 9) after ~25 mL distillate, add 25 mL DI water
- 10) need ~ 50 mL (60 min)
- 11) raise RB flask out of heat
↓
- 12) isolate oils
 - a) transfer distillate to sep funnel
 - b) shake / vent
 - c) separate layers (label)

$T_1 = 89^\circ\text{C}$ T_2

Distillate milky white

Procedure

d) Extract with 2-10 ml

Petroleum or Ether

e) Dry organic layers
with $MgSO_4$

f) gravity filter

13) Roto vap

14) IR

15) NMR

a) 1 mL $CDCl_3$ to
Sample

b) transfer to 5 cm
mark on NMR tube

c) Clean outside of
tube with acetone

A

NOTE

Clove: 1)

5 g ground clove with
150 mL water

9) if $V_{res} < 25$ mL
add 25 mL

Anise: 1) 8 g anise +
150 mL water


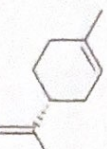
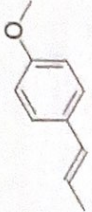
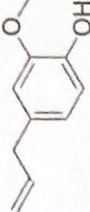
→ 9)

Funnel m = 42.490 g

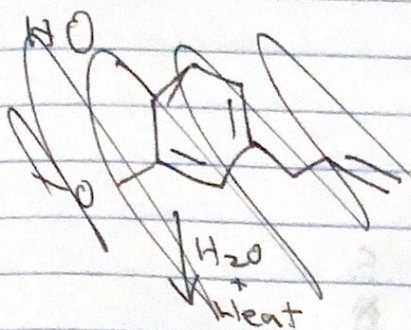
total m = 42.752

Sol'n turned yellow

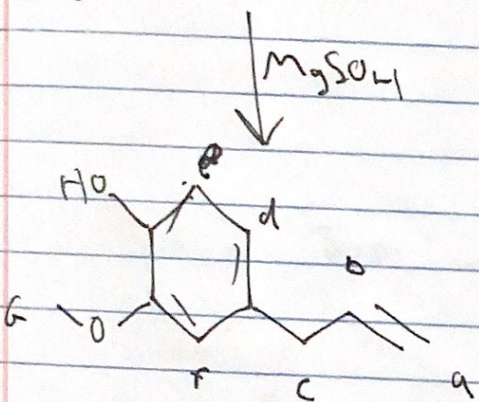
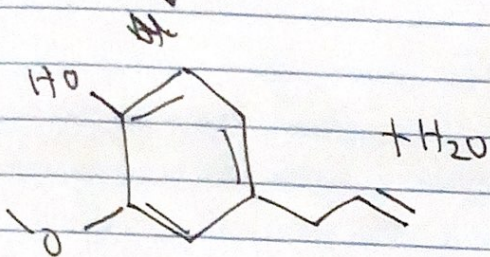
Added $CDCl_3$ and D₂O
separately to the NMR tube
- removed, mixed, re-added

| Molecule | Functional Group(s) | Key peaks in IR (cm ⁻¹ , bond) | Number of peaks expected in the ¹³ C NMR | ¹ H NMR chemical shifts of key peaks / type of hydrogen/functional group indicated/number of peaks |
|---|--------------------------------------|--|---|---|
|  | alkane, alkene | 3000-3100 sp ² C-H 1640-1680 C=C | 5 | 5.0-6.5 ppm/ sp ² C-H/alkene/5 peaks; ~2 ppm/sp ³ C-H/alkane/1 peak |
|  | Alkene, Alkane | 3000-3100 C-H 1640-1680 C=C | 8 | 5.9-2 / sp ² C-H / Alkene / 4 ~2 / alkene / 3 / sp ³ C-H |
|  | Aromatic ring, Ether, Alkene, Alkane | 1600-1585 1250-1050 3000-3100 1600-1680 | 8 | 6.5-8 / phenol-H / phenol / 4 0.9-2 / sp ³ C-H / Alkene / 6 2 / alkene / 1 |
|  | Alcohol, Ether, Alkene, Alkane | 2400-3000 " " | 10 | 6.5-8 / phenol / 3 ~2 / alkene / 3 1-5 / OH / 1 |

Separation Scheme



Clove + H₂O
Heat



Steam Distillation

The goal of this experiment was to distill clove oil from ground up cloves using steam distillation. Steam distillation is a process by which volatile components of a solution can be separated from the non-volatile components. This is done by mixing the starting material with water and heating it until the water starts to form steam. The volatile components are carried by the steam through a condenser which condenses the steam back into a liquid. This new liquid solution can then be dehydrated using an anhydrous solution, and the final product would be the volatile components, or for this experiment, the essential oil. Clove oil has traditionally been used to ease digestion issues and helping with respiratory conditions. Many essential oils also have a very pleasant aroma.

The percent of oil present in the cloves is given by $\frac{\text{mass of product}}{\text{mass of starting material}} * 100\%$ and was found to be about 5.24%

IR Spectra

Our IR spectra shows peaks that correspond to the functional groups in the expected product. Multiple peaks around 3000cm^{-1} which could be due to the C-H bonds, peaks at 1600cm^{-1} and 1765cm^{-1} indicating a phenol group and C=C. There is also a broad peak at 3434cm^{-1} due to the O-H group. There does not seem to be any peaks that do not correspond with the expected product suggesting that our product could be relatively pure.

H-NMR

Each proton that would produce a signal is labeled “a” through “F” on my drawing on the Eugenol molecule. The signals on the H-NMR spectra are similar to what was expected with peaks at 3.2ppm (F) and 3.6ppm (E) which correspond to the H on the alkanes with the 3.6ppm correlating to the H-C-O (E). Other peaks appear at about 5.0ppm (D) and 5.8ppm (C) which correlate to the H on the alkenes with 5.8ppm correlating to the H-C=R group. Finally, there were peaks at 6.6ppm (B) and 6.7ppm (A) which correlate to the H on the phenol group.

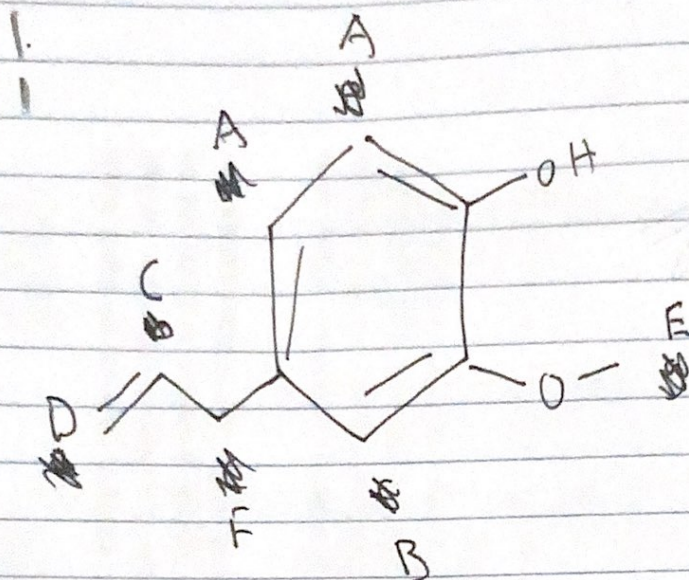
The pattern for each signal also suggests a relatively pure product. “F” is a doublet which means that there is one nonequivalent H neighboring that H splitting the signal. Looking at the molecule structure, there is one neighboring H (C). “E” has no neighboring H so the signal is a singlet. “C” is neighbored by 2 H on one side and another 2 H on the other so the signal would be expected to be some kind of sextuplet which is again what we see on the spectra. “B” is not neighbored by any H so we see a singlet but “A” and “D” do not follow the trend as closely since “A” has no nonequivalent neighbors yet a doublet is observed and “D” only has 1 neighbor so only a doublet would be expected yet a more complex pattern is observed. I don’t think that this is due to impurities because if our solution was impure then we would have seen more signals on the H-NMR and on the IR spectra but each main signal can be correlated to a part of the expected molecule. We did commit multiple small errors while preparing our sample for the H-NMR run and it is possible that by not following the instructions closely, we caused the spectra to be less exact.

C-NMR

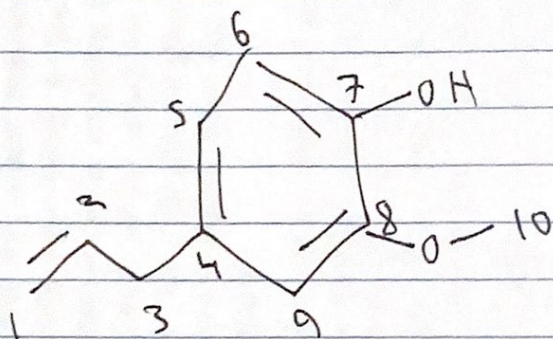
We did not have the opportunity to run a C-NMR on our sample but based on the structure of eugenol it can be predicted that there would be 10 different signals because the molecule has 10 different carbons. Referring to my drawing of eugenol, the following table shows the signal that each carbon can be expected to produce.

| Carbon | Functional Group | Signal (ppm) |
|--------|------------------|--------------|
| 1 | Alkene | 110 |
| 2 | Alkene | 140 |
| 3 | Alkane | 40 |
| 4 | Phenol | 130 |
| 5 | Phenol | 120 |
| 6 | Phenol | 115 |
| 7 | Phenol | 145 |
| 8 | Phenol | 147 |
| 9 | Phenol | 115 |
| 10 | Ether | 56 |

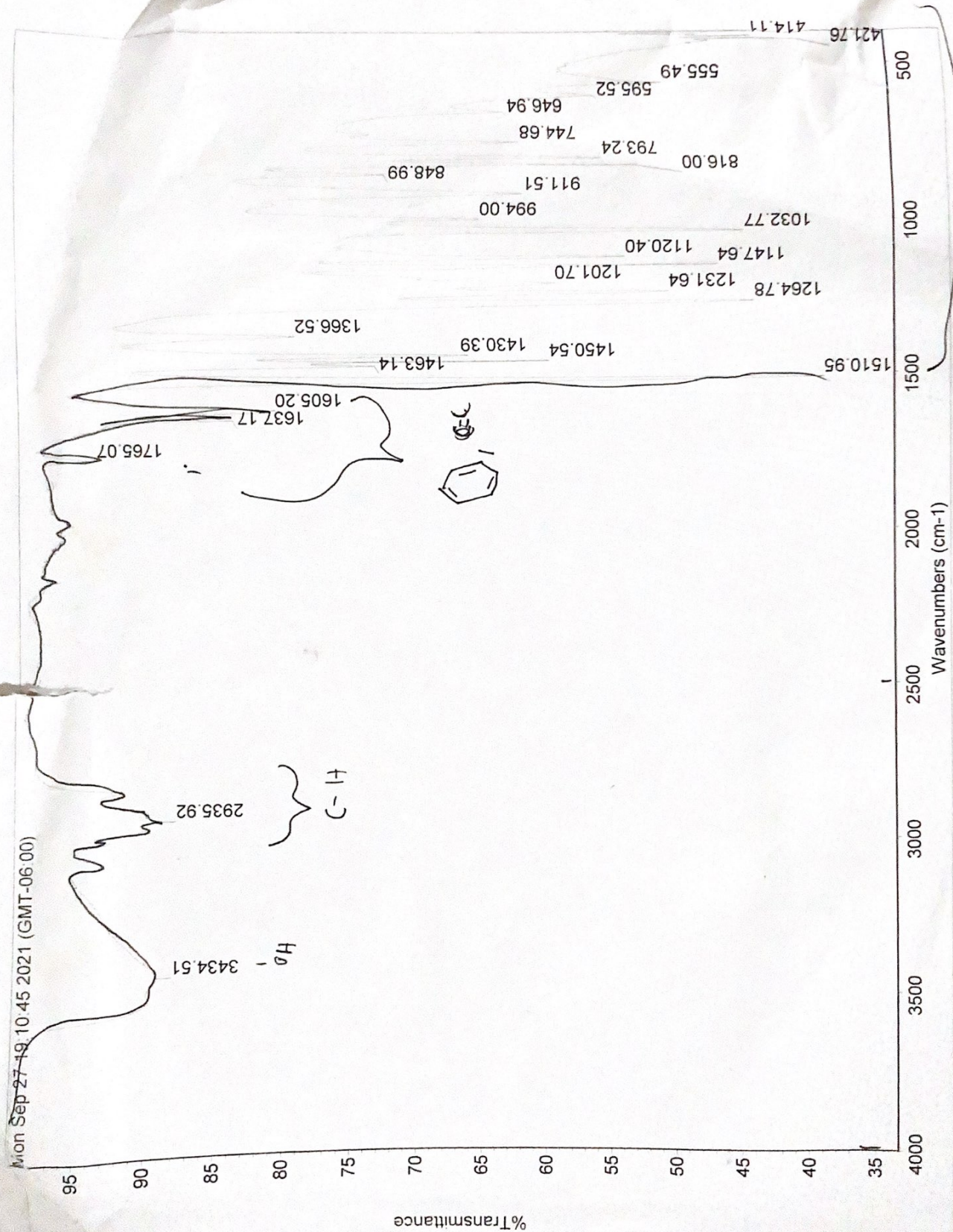
H-NMR



C NMR



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UU_proton CDCl₃ / opt / topspin 4.0.8 mjaustin-300

