

2021 – EXPERIMENT 1 LAB REPORT: DISTILLATION OF A LIQUID MIXTURE

DUE: week of 9/27, by noon on your lab day as a pdf file

Name of Unknown Mixture: Glenquidditch***Answer the following:***

1. Attach tables of the Temperature and Volume data for both the simple and fractional distillations.

Table 1. Simple Distillation of Glenquidditch temperature was recorded for every 1ml of distillate produced.

(Note: There was an experimental error where I was not able to obtain data from 10ml to 17ml)

Volume (ml)	Temperature (°C)
1	75
2	77
3	78
4	80
5	82
6	83
7	86
8	88
9	88
10	N/A
11	N/A
12	N/A
13	N/A
14	N/A

15	N/A
16	N/A
17	N/A
18	110
19	108

Table 2. Fractional Distillation of Glenquidditch. The Temperature was recorded at the start and the end of every 2 ml of distillate collected.

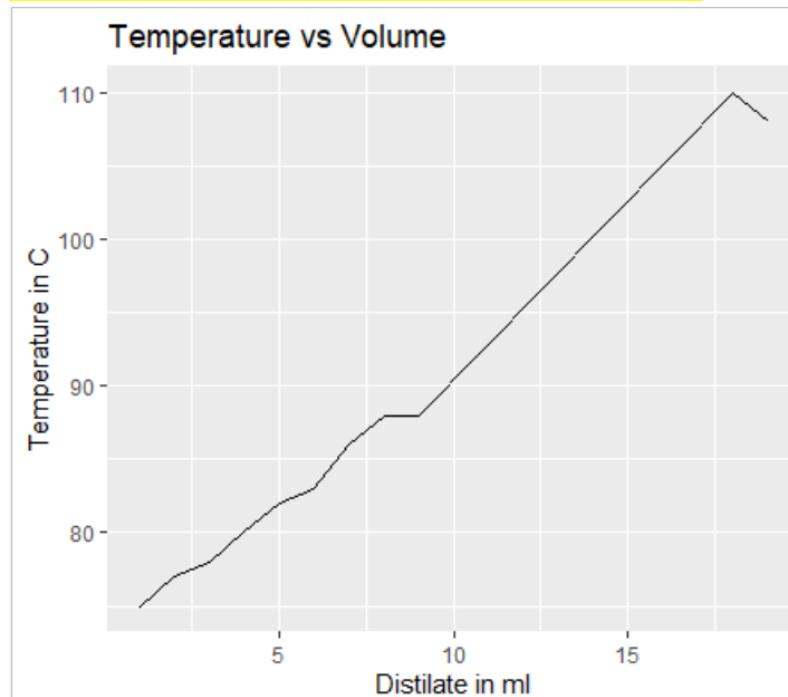
(Note: Our group had to stop after 12 tubes were collected due to a flooding risk)

Volume (ml)	Temperature (°C)	Start (S) End (E)
1	65	S
1	70	E
2	70	S
2	75	E
3	74	S
3	75	E
4	74	S
4	72	E
5	72	S
5	74	E
6	74	S
6	76	E
7	78	S
7	78	E

8	80	S
8	120	E
9	120	S
9	122	E
10	122	S
10	122	E
11	122	S
11	120	E
12	120	S

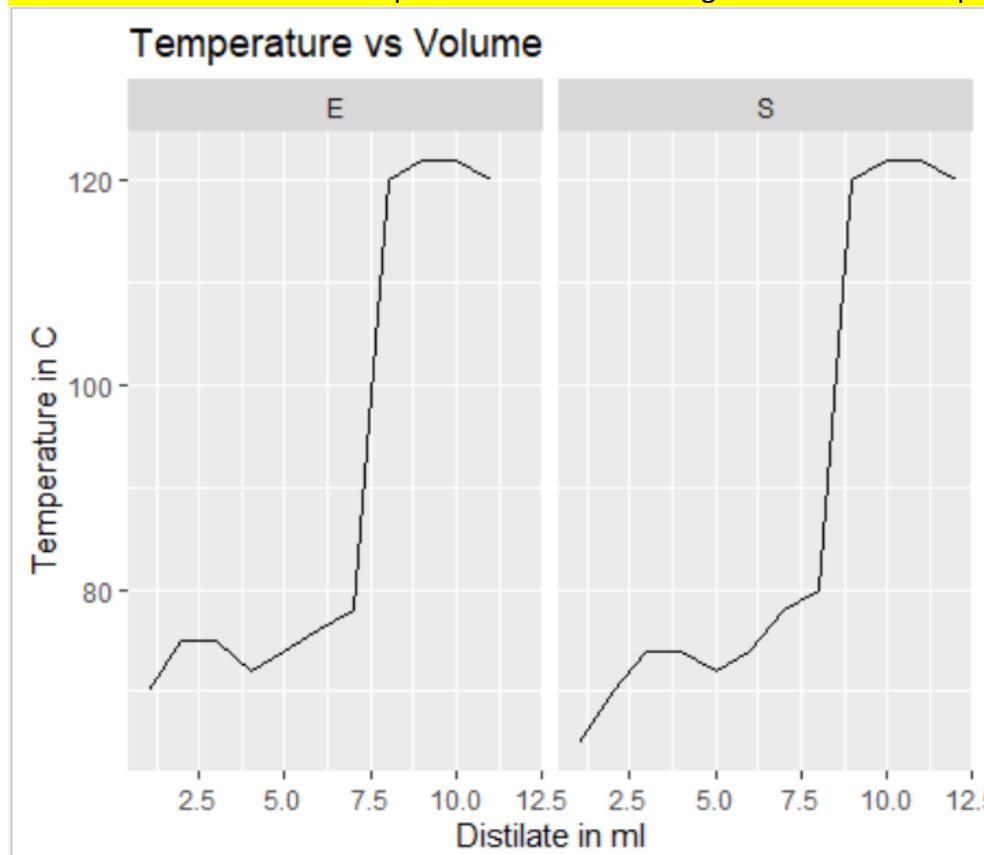
2. Attach a Temperature ($^{\circ}\text{C}$) versus Volume (milliliters) graph for **both** the simple and fractional distillations. Include both lines in the same graph.

(Note: I could not get R to put all lines in the same graph without having issues with the data frame not having the same number of columns).



Graph 1. Comparison of Distillate Volume and Temperature in C for the Simple Distillation.

(Note: I could not get the computer to only connect the first and second set of points. It tried to draw the line over all points and I could not figure out how to stop it).



Graph 2. Comparison of Distillate Volume and Temperature in C for the Fractional Distillation. S represents the temperature at the start of each tube and E represent the temperature at the end.

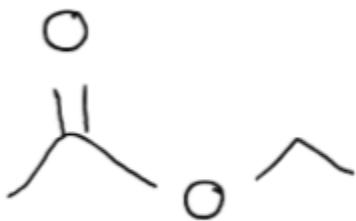
3. Comment on the effectiveness of each technique in the separation of the components of the mixture in three sentences or less. Support your statements by referencing your graph.

It seems that the simple distillation was more effective than the fractional distillation. The temperature in the simple seemed to level off while in the fractional seemed to increase throughout the duration of the low boiling component. However, since some of the data for the simple is missing this statement cannot be fully supported.

Provide the following information:

4. **Low boiling component**

- systematic name: ethyl acetate
- structural formula
 $\text{C}_4\text{H}_8\text{O}_2$



- degree of unsaturation (DU): 1 (1 double bond)
- scent description (two words or less): sour and rancid
- experimental bp ($^{\circ}\text{C}$): 76 from fractional
- literature bp ($^{\circ}\text{C}$): 77 Reference: <http://www.chemspider.com/Chemical-Structure.8525.html>

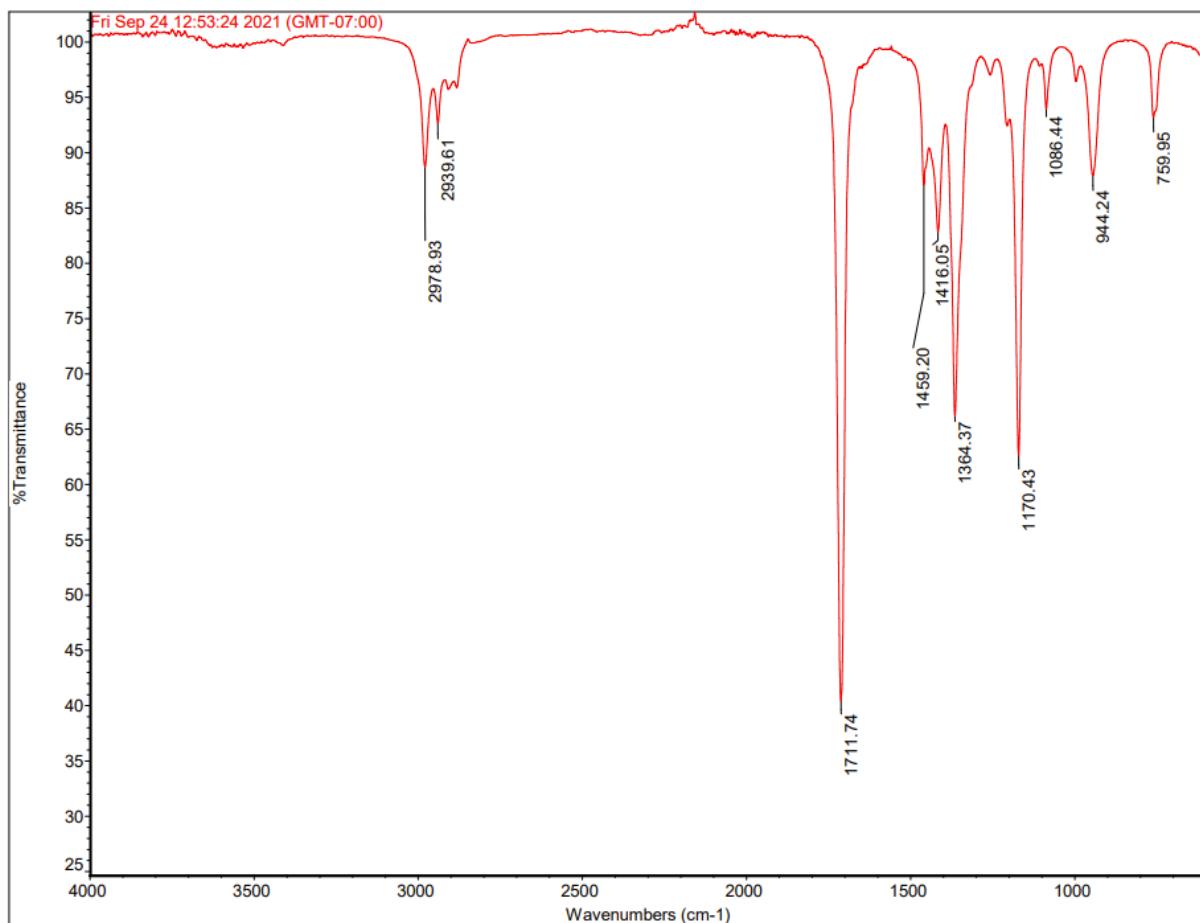


Figure 1. IR of the low boiling component of the Ethyl Acetate

Spartan'18 Predicted Absorption Maxima (cm ⁻¹)	Observed Absorption Maxima (cm ⁻¹)	Relative intensity (e.g. s, m, w)	Shape (e.g. sharp, broad, W- shaped)	Assignment (assign to a specific bond e.g. O-H)
1770	1712	s	Sharp	C=O
1439	1459	m	Sharp	(O=)C-O
3045	2979	w	Sharp	O-C(H ₂ CH ₃)
N/A	2939	w	Sharp	C-H

5. High boiling component

- systematic name: 3-methyl-1-butanol
- structural formula



- degree of unsaturation (DU): 0 (no double bonds or rings)
- scent description (two words or less): sweet and floral-like
- experimental bp (°C) 122 from fractional
- literature bp (°C) 130 Reference: <http://www.chemspider.com/Chemical-Structure.29000.html#:~:text=3%2DMethylbutan%2D1%2Dol%20%7C%20C5H12O%20%7C%20ChemSpider>

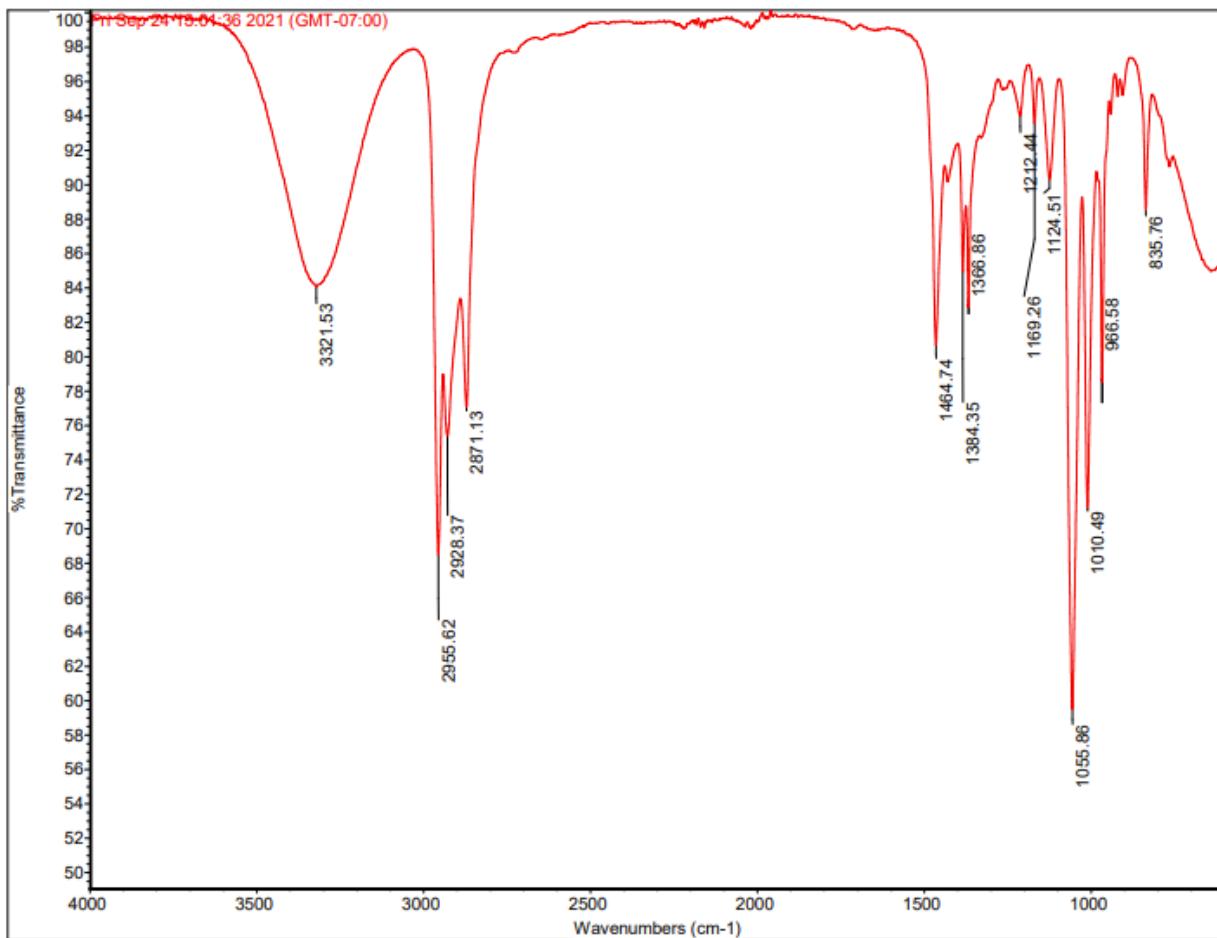


Figure 2. IR of the high boiling component of 3-methyl-1-butanol

- Complete the table using Figure 2:

Spartan'18 Predicted Absorption Maxima (cm^{-1})	Experimental Absorption Maxima (cm^{-1})	Relative intensity (e.g. s, m, w)	Shape (e.g. sharp, broad, W-shaped)	Assignment (assign to a specific bond e.g. O-H)
3608	3321.53	m	Broad	O-H
1066	1055.86	s	Sharp	C-O
2999	2955.62	s	Sharp	C-H
2930	2928.37	m	Sharp	C-H
2399	2871.13	w	Sharp	C-H
		m	W-shaped	C-H
		m	Sharp	C-H

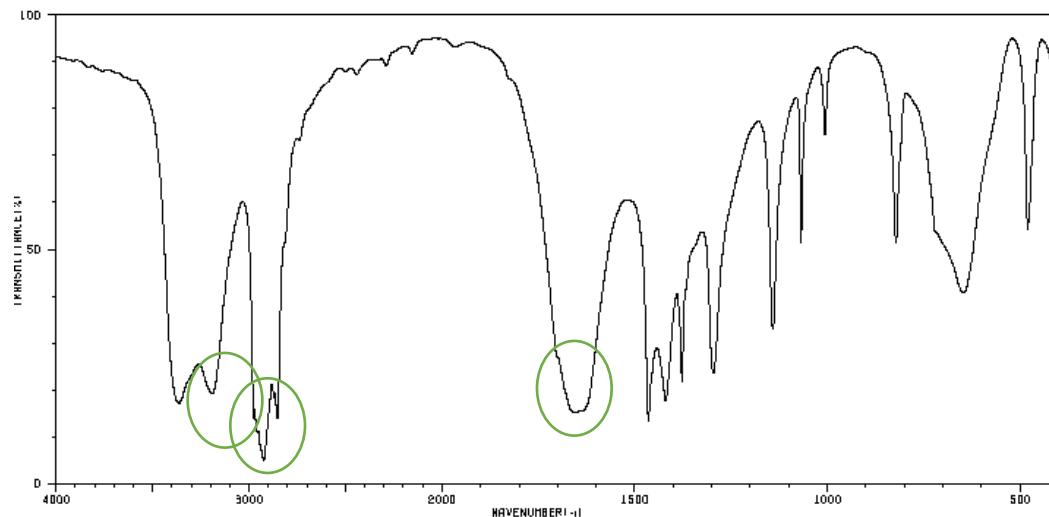
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6. Where there any significant sources of error in the simple or fractional distillation procedures that you performed? If so, did they affect the data analysis?

Yes. In the simple distillation I forgot to record the temperature from samples 10 through 18 which means that data on the higher boiling compound may not be accurate for that distillation since I only have 2 measurements. In the fractional distillation we had to stop after tube 12 due to flooding occurring in our apparatus. Thus, the temperature for the higher boiling compound may not be as accurate since we were not able to carry out our distillation all the way.

7. Given the molecular formula (C_3H_7NO) and the following IR, deduce the structural formula of the compound by answering the following questions.

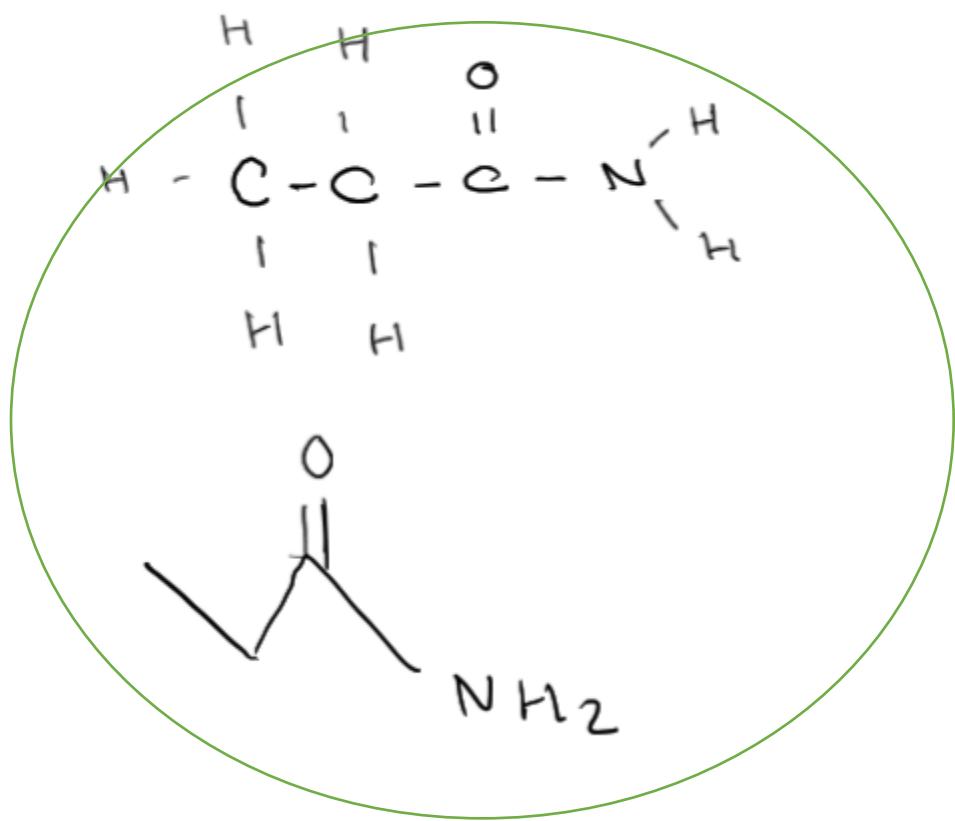
Helpful IR Tables: Sorrell 14.3, Padis pp. 66-76

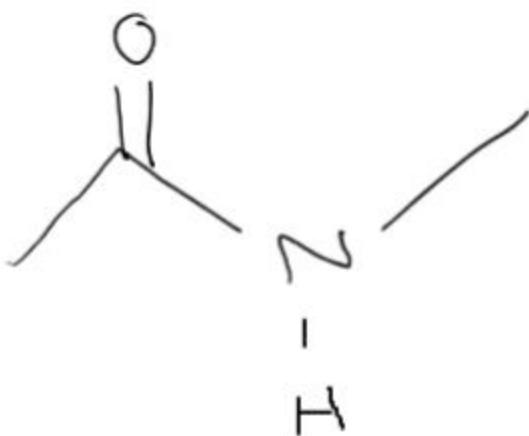
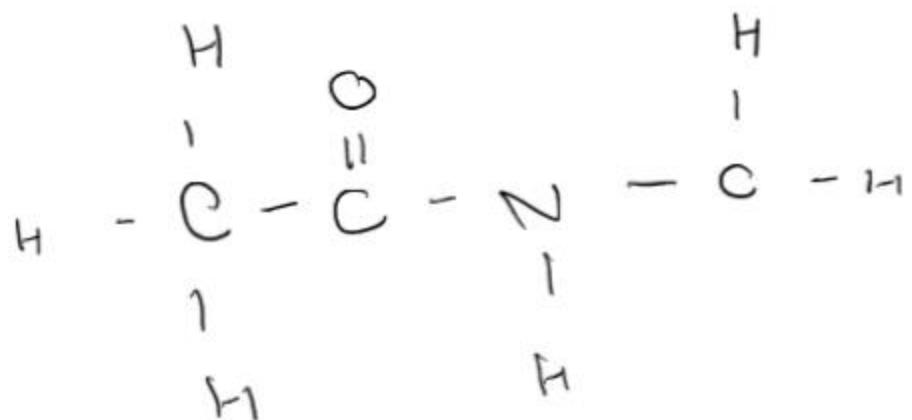


- (a) Circle the major IR bond stretches you see from 4000-1500 cm⁻¹ ONLY in the IR provided. Your answers should be consistent with the molecular formula. Do not include bonds that would appear below 1500 cm⁻¹.



- (b) Draw structural formulas of constitutional isomers that fit part (a) and the molecular formula. **At least two structures are required.** Circle the one you think is most likely correct based on the IR data provided.

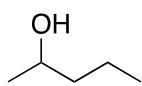
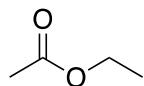
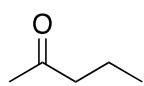




- (c) If a literature IR spectrum of your choice in part (b) was provided, what part of the IR spectrum above would you use to compare with the literature spectrum to identify the compound?

I would compare the fingerprint regions since those are unique for every compound. Not the functional group region because there may be similar functional groups in different molecules.

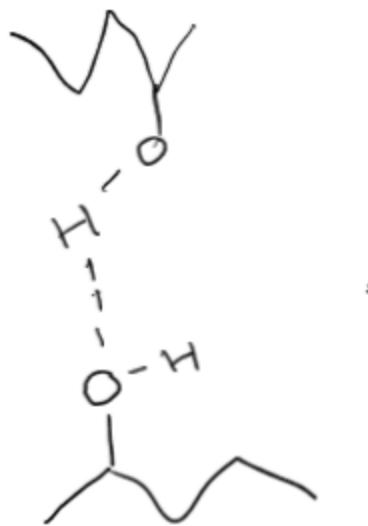
8. Use the structures provided below and what you learned during the Spartan'18 Modeling Activity 1B to answer the following questions.



- a. Pick 2 of the 3 structures above and compare their structures to explain the effect of hydrogen bonding on boiling points?

The structure of the third molecule allows it to hydrogen bond in water and in a pure solution (the oxygen has a lone pair and works as an acceptor and the hydrogen attached to the oxygen works as a donor). The structure of the first molecule (polar C=O bond) allows it to form dipoles with other molecules but not other hydrogen bonds. These dipoles are much weaker than hydrogen bonds which means that less energy is needed to break them and thus the boiling point is lower than compounds with stronger intramolecular forces. This is seen in the comparison in the boiling points of the 1st (102 °C) and 3rd (130 °C) compounds.

- b. Choose one of the structures above and draw two of its molecules hydrogen bonding. Represent the hydrogen bonding between the molecules by dashed lines.



Record Boiling Point!

"Still" - equip. used for dist.



then only provides info abt. the gas

(can be used to (1) measure bp (2) sep. liq./solid
(3) sep. & liq.)

a liquid's temp. can't be raised above its bp

Practical - sep. part of distillation

vapor pres. b/w mixtures

procedure

1.) get 44ml of mixture, split w/ paraffin (23ml), germane

2.) clean/dry glassware

3.) mark line on each test tube

4.) assemble the dist.

a. clamp boiling flask to monkey legs

b. place 31g chips in the boiling flask

c. add sample to flask using dropper funnel

d. add remaining glassware

e. adjust the height of the therm. so the bulb is below the opening leading to the condenser (but don't let the bulb touch the glass wall)

f. attach water hoses to the condenser w/ hose clamps

g. attach condenser to adjoining glassware w/ plastic joint clamps

h. collect distillate in a 10ml graduated cyl. (+ bring extra)

(check glass joints to see if they fit)

i. plug heater into controller

j. heat mixture to gentle boil (adjust heat settings until collection is one drop per sec.)

k. record the temp. for every 1ml that comes over

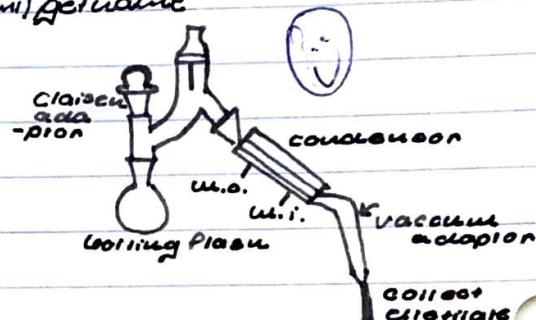
l. when 9ml is reached briefly cover the heater and transfer the liquid to the storage container

m. when 1/2ml remains in the flask turn off heater

n. assemble practical distillation

a. same as above w/ the following:

b. allow flask to cool comp.



Distillation of a Liquid Mixture

Sep. 10th, 21

Materials

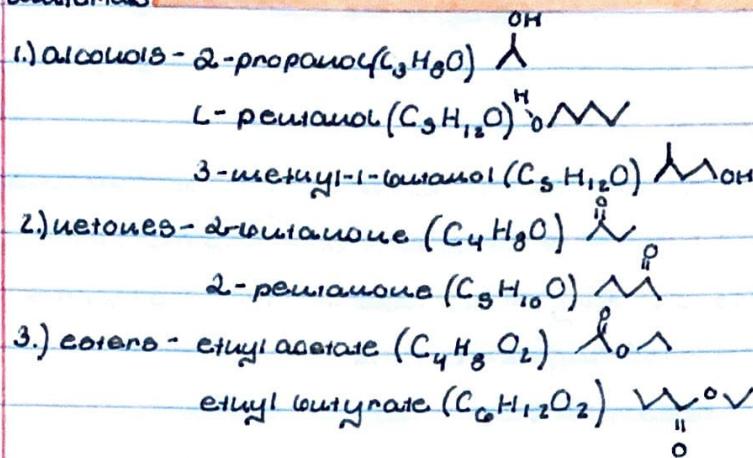


Table of physical properties

Compound	Molar Mass	mp ^(finc)	bp ^(finc)	Density (g/ml)
2-propanol	60.10	-90°	82°	0.785
L-pentanol	88.148	-79°	136°	0.812
3-methyl-1-butanol	88.148	-117°	130°	0.809
2-butanone	72.106	-87°	80°	0.805
2-pentanone	86.132	-78°	102°	0.813
Ethyl acetate	88.105	-84°	77°	0.902
Ethyl butyrate	116.168	-93°	121°	0.879

Safety hazards/disposal

- 2-propanol → Flammable liquid (необходимо远离 open flame, work under fume hood) eye-irr.(mean goggles), Avoid breathing vapors
- L-pentanol + Flammable vapors (необходимо远离 open flame, work under fume hood) eye-irr.(mean goggles), avoid inhalation (fume hood)
- 3-methyl-1-butanol → Flammable (необходимо远离 open flame, work under fume hood) eye-irr.(mean goggles) resp. tract danger (fume hood)
- 2-butanone → Flammable (необходимо远离 open flame, work under fume hood)
- 2-pentanone → Flammable (необходимо远离 open flame, work under fume hood) vapors cause drowsiness (fume hood)
- Ethyl acetate → Flammable (необходимо远离 open flame, work under fume hood) eye-irr.(mean goggles)
- Ethyl butyrate → Flammable (необходимо远离 open flame, work under fume hood) vapors irritating to upper respiratory (fume hood)



- c. put both samples in the flask w/ 2-3 boiling cups
- d. Pasteur or screw clamp tightly to the boiling flask and loosely Pasteur a quick clamp to the upper part of the fractionating column.

- (e.) be slow, gradually turn up the heat until boiling + adjust heater settings so that the ring of vapor rises slowly up the column; apply more heat once the ring of condensation was stopped rising
- 11.) get the heat to 20 drops(min) per minute
- 12.) record temp. at 1st drop and 2nd
- 13.) after 2nd stop it + let the column drain, measure the volume of distill. liquid

if flood + lower heat/allow draining/start over
 if heat loss + better insulation/more heat
 → heat lost

- 14.) identify samples

- 15.) put in round bottom flask, stopper + wrap the top, stand it up on glassware in the lab cabinet

- 16.) disassemble remaining fractionating P. liquid, disassemble + clean w/ acetone, return it to lab desk, wipe down wood materials

- 100 mL 14/20 neck
- glass beaker w/ side tube
- therm. w/ 14/20 adapter
- reflux condenser + water jacket + loose clamps
- vacuum adapter
- 50mL graduated cyl. (to collect dist.)
- fractionating column heads
- 10 test tubes + rack
- 325 mL Erlenmeyer flasks

(boiling cups/care, jack/ceramic heater + controller/screw clamp/s, controller/screw clamp + plastic joint clamp/water jacket)

Distribution of a liquid mixture

modifying activity

ethyl acetate

min/max (-176.24 μJ/mol to 88.02 μJ/mol)

range (264.24 μJ/mol)

Oxygen close to min, carbon close to max

area (124.72 Å²) para (0.59 Å²)

2-butanone

min/max (-178.02 μJ/mol to 97.98 μJ/mol)

range (275.04 μJ/mol)

Oxygen close to min, carbon close to max

area (114.87 Å²) para (0.94 Å²)

2-pentanone

min/max (-176.76 μJ/mol to 95.63 μJ/mol)

range (272.09 μJ/mol)

Oxygen close to min carbon close to max

area (133.05 Å²) para (10.10 Å²)

2-propanol

min/max (-181.94 μJ/mol to 199.61 μJ/mol)

range (381.55 μJ/mol)

Oxygen close to min hydrogen close to max

area (101.33 Å²) para (11.02 Å²)

Cyclohexanol

min/max (-175.18 μJ/mol to 223.42 μJ/mol)

range (398.64 μJ/mol)

Oxygen close to min hydrogen close to max

area (41.43 Å²) para (0.67 Å²)

3-methyl 1-butanol

min/max (-178.51 μJ/mol to 218.16 μJ/mol)

range (396.37 μJ/mol)

Oxygen close to min hydrogen close to max

area (136.74 Å²) para (0.95 Å²)



videos

- (1) start w/ round bottom flask, buildup
- (2) don't use silver mercury then.
- (3) measure temperature every min
- (4) insulate the joints



Questions

- 1) one group: 2-propanol / 2-butanone / ethyl acetate
(bp around 80°C)
another group: 1-pentanol / 3-methyl 1-butanol / 2-pentanone
(bp around 130°C)
- 2) The prediction for 2-pentanone is an outlier
- 3.) 2-propanol, 1-pentanol, 3-methyl 1-butanol
- * 4) The model results support the notion that hydrogen bonding is important in raising the boiling point. All molecules with hydrogen bond ability show a greater range (neg to pos) with the extremes being near the hydrogen and the oxygen, meaning that these regions have a stronger intramolecular force than those with less of a range and a maximum only on oxygen/carbon.

A handwritten grade 'A' is enclosed in a circle, indicating a good answer or result.

Distillation of mixture

partner: Mayasax

sep 11/7/21

- 1) got 44ml glengolditch
- 2) 22ml spirit of person
- 3) Did simple distillation w/ above product. Obtained 19ml of distillate.
- 4.) Collected maya and 1's distillation products.
- 5.) Performed fractional distillation with all 44ml of the mixture. Collected 12 tubes of product each at 2ml.
Distillation had to be stopped after 12 because
the fract. column kept overflowing
- 6.) Collected tubes 3 and 4 (low) and 10 and 11 (high)
for next week's lab.

run temp.

1 75°C

2 77°C

3 78°C

4 80°C

5 82°C

6 83°C

7 86°C

8 88°C

9 88°C

10

11

12

13

14

15

16

17

18 110°C

19 108°C

Simple distillation

experimental error:

I thought we only
had to record for the
first 9 all, so I did
so

Practical duration

<u>cycle</u>	<u>start/end (core) (temp.) (C°)</u>
1	65
1	70
2	70
2	75
3	74
3	75
4	74
4	72
5	72
5	74
6	74
6	76
7	78
7	78
8	80
8	120
9	120
9	122
10	122
10	122
11	122
11	120
12	120
12	→ flooding start rise

13 (had to stop after 12 due
to issue of flooding)

14

14

15

15

16

16

17

17

18

18

19

19

20

20

Week 2 - Characterization

- 1.) draft each chemical, try to find a common name/
smell/color/functional group etc...

User Instructions

- 1.) Open airac
- 2.) make sure exp field says chem 2011202
 "CP will click right arrow"
- 3.) Click "sample"
- 4.) Give sample a little
- 5.) Ensure there is no sample in the air, click away
- 6.) When this is done a box will appear asking
to collect your sample, draw up a small amount
via a glass pipette, pipette 3-7 drops onto the
center of the air accessory, do not touch the surface
- 7.) After it ends click yes
- 8.) Clean air first by dry cotton and then by ethanol
cotton
- 9.) Print peaks by clicking "Print pic"
 "Move line by clicking next spot"
- 10.) When everything looks good hit replace
- 11.) Create a previous by using 'prev rpt'
 "Clear part writer"
- 12.) Clear spectrum
- 13.) Log out of mail, email PDF to self

BTW

mapping test

2-propanol - no smell

2-butanone - sweet

6-pentanol - sour

ethyl crotonate - very sweet

2-pentanone - like acetone

3-methyl-1-butanol - sour

ethyl acetate - very like acetone

←

-ol

Low: smells like acetone

High: sweet, but not strongly

1.) Chemical mapping (sample own own)

2.) IR never looks like acetone with samples.

IR activity questions

(questions 7-9)

analyzing + predicting spectra

bond	Frequency range(cm^{-1})	intensity	shape
O-H	3690 - 3680	S	broad, wosuper normal
C-H	2950 - 3100	M?	sharp(?)
C=C	2100 - 2260	S?	sharp

q 8) What do you see? a.) see p1P for peak classification

bond	Frequency range(cm^{-1})	intensity	shape
N-H	3,900	M	w-shaped
C=O	1,800	S	w-shaped

q 9) The molecule contains an amide.

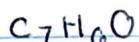
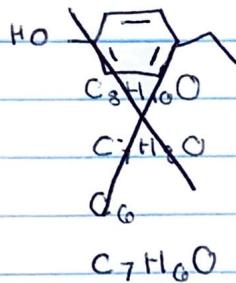
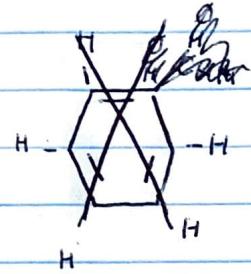
(Because it has both N-H and C=O bonds)

molecular weight = 106

no O-H (nothing at 3,900)

bond Frequency range(cm^{-1}) intensity ~~excepted~~ $\text{C}_8\text{H}_8\text{O}$ C-H $\sim 2,900 - 3,000$ W ~~W~~C=O ~ 1700 SC=C ~ 1600 ~~and M~~

benzaldehyde?



Spartan 11.0 modelling activity

table 1.) ethyl acetate

functional group / stretching vibration		freq.(cm ⁻¹)
carboxyl	C=O	1710
acyl	(O=)C-O	1439
alanyl	O-C(H ₂ CH ₃)	3045

table 2.) 3-methyl-1-butanol

functional group/streching vibration		freq.(cm ⁻¹)
O-H		3608
C-O		1066

table 3.) ketone group

functional group	freq.(cm ⁻¹)
C=O(ketone)	1800-1680