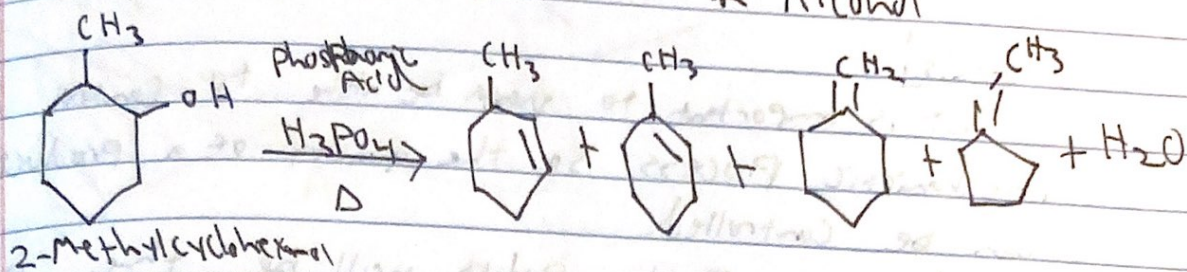


Dehydration of an Alcohol



Purpose:

Dehydrate alcohol via E_1 reaction by removing OH group from starting material

Equipment

- 13 test tubes (100mm)
- Micro Scale rxn tube
- 25 mL Erlenmeyer flask
- Heat source
- Thermowell
- thermometer w/ adapter
- Ice bath

Chemicals

- 2-Methylcyclohexanol *
- 85% Phosphoric Acid †
- 10% Sodium bicarbonate
- Anhydrous Calcium Chloride $CaCl_2$

Hazards:

- * irritant (products are irritants as well)
 - † Corrosive
- } Wash skin thoroughly

Goals:

It is important to ~~have~~ be able to control a mechanistic process so the quality of a product can be controlled.

The most stable product will be the product with the most substituted alkene which is 1-methylcyclohexene:



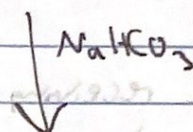
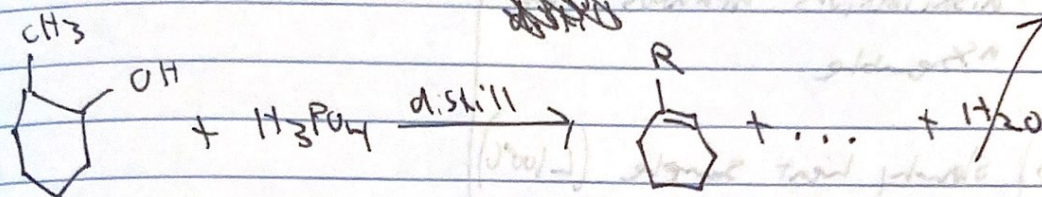
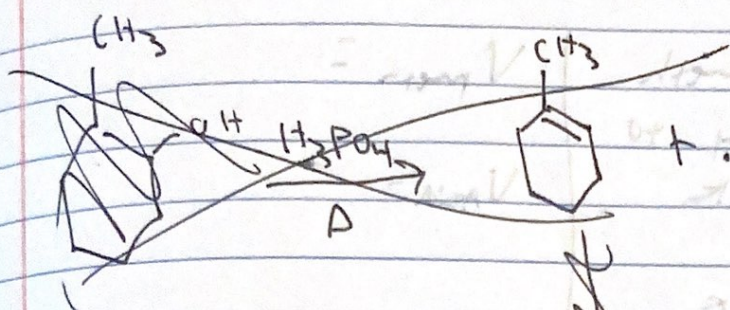
~~If the distilling vapor rises above 100°C then the water will evaporate as well and the product will be similar to starting materials.~~

~~Adding anhydrous Sodium Sulfate to Cyclohexene layer will remove excess water from the layer and will help to purify sample.~~

Increasing temp above 100°C would cause water to evaporate so the water would be carried into the receiving flask along with desired product.

Adding Bicarbonate allows separation to occur so the desired product can be extracted.

The Anhydrous CaCl_2 will absorb excess water from product allowing it to "dry".



Mix

discard
 Aqueous
 layer

$\downarrow CaCl_2$
 Product

remove



Procedure:

- 1) Mix 3 mL 2-meth and 1 mL H_2PO_4 to round bottom flask
- 2) Grease joints of distillation apparatus and assemble
- 3) slowly heat sample ($<100^\circ\text{C}$)
- 4) place receiving flask into ice bath
- 5) when 2-3 mL are collected, transfer to test tube
- 6) Remove water layer (bottom) with pipette
- 7) wash organic layer with equal volume NaHCO_3
- draw small amounts into pipette and squirt into organic layer to mix
- 8) Remove aqueous layer

$V_{\text{meth}} =$

$V_{\text{acid}} =$

$V_{\text{product}} = 1.8 \text{ mL}$

no aqueous layer

$V_{\text{NaHCO}_3} = 1.8 \text{ mL}$

Procedure

9) Transfer organic layer to flask and add CaCl_2

10) Filter mixture with cotton onto flask

$$m_{\text{flask}} = \frac{41.85}{28.88}$$

11) IR spec of product AND Starting material

$$m_{\text{tot}} = 29.592$$

12) Dissolve 1 drop of product in 1 mL Hexane and Submit for GC

13) Add 2-3 drops of product to tube with 1 mL THF

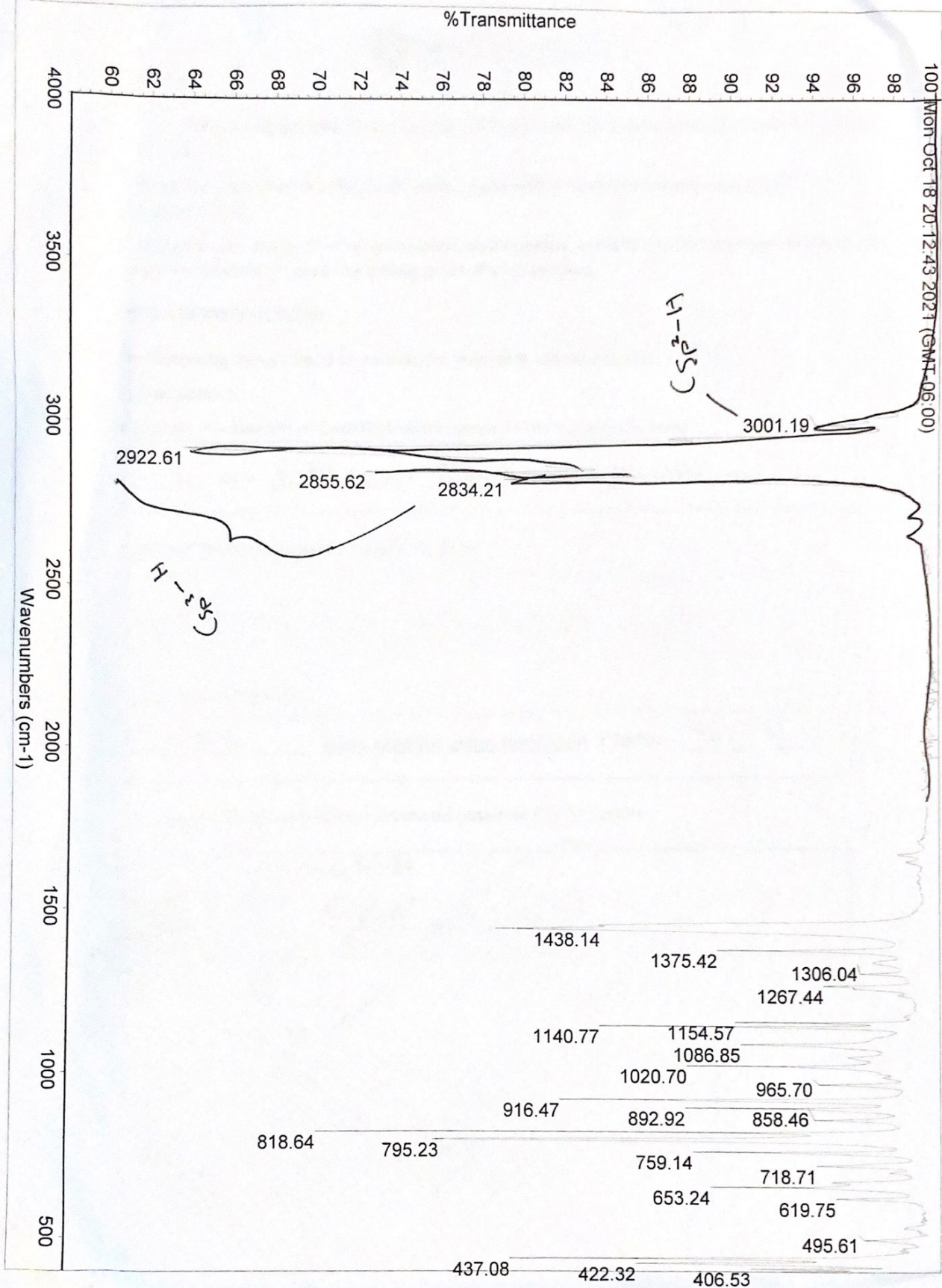
~ 1 mL

- Add 0.05 M Pyridinium tribromide drop wise until soln stays orange

11-2

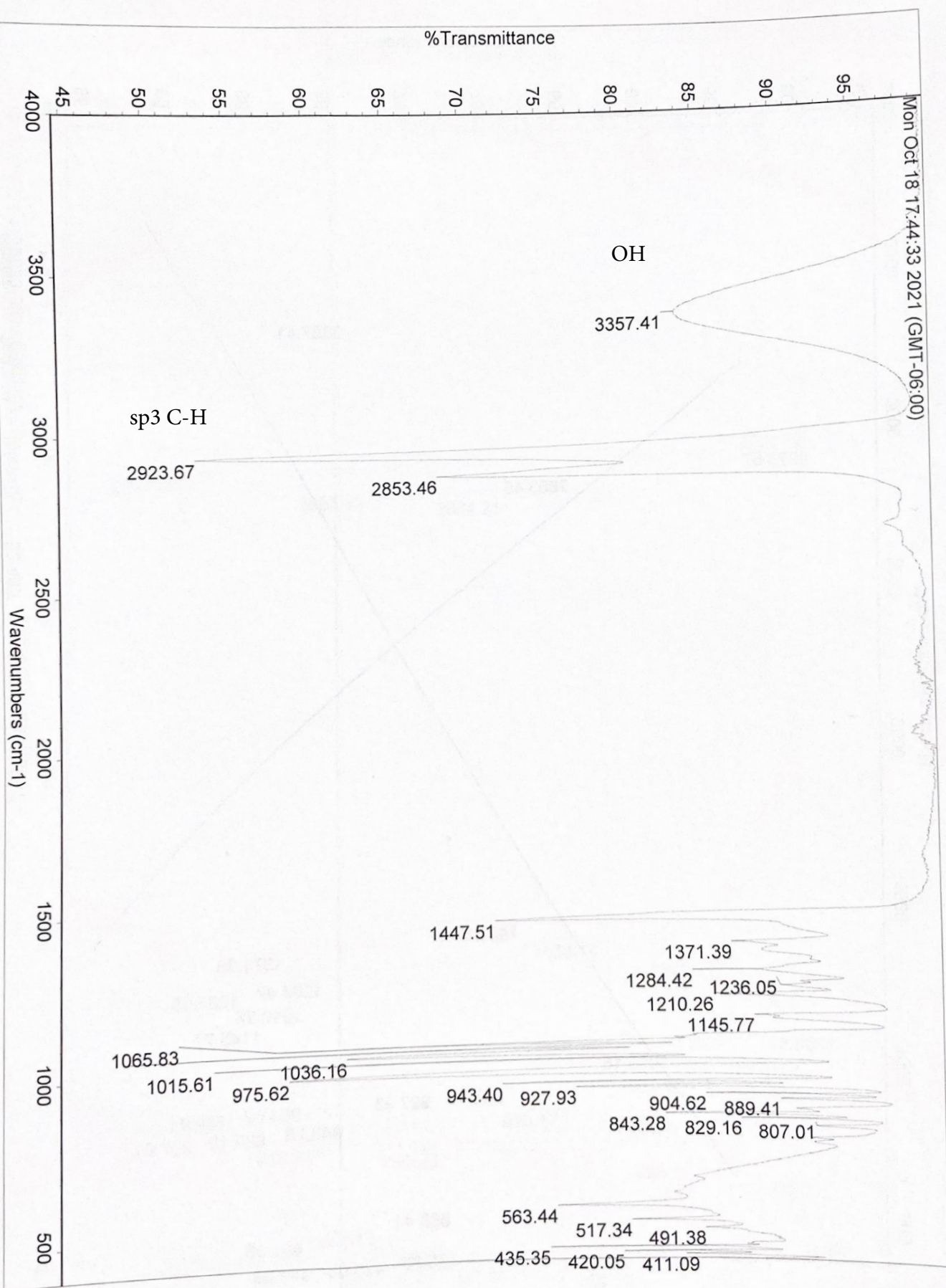
turned yellow ~ then clear
white precipitate

	2-methylcyclohexanol	phosphoric acid	C_7H_{12}
MW (g/mol)	114.2	85% aqueous	96.10
d (g/mL)	0.93 g/mL	NA	NA
bp ($^{\circ}\text{C}$)	165	~100	~102-110
amount used/obtained (g or mL)	3 mL	1 mL	0.712 g
amount used/obtained (mol)	0.0244 mol	N/A (catalytic)	0.007409 mol



2 - methyl cyclohexanol

Mon Oct 18 17:44:33 2021 (GMT-06:00)



Qualitative Analysis Report

User Chromatograms

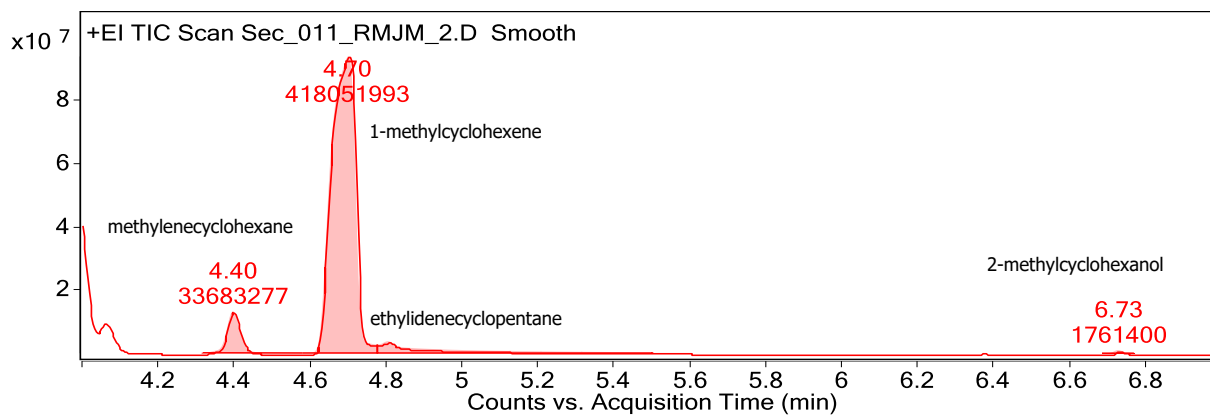
Fragmentor Voltage

Collision Energy

0

Ionization Mode

EI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	4.32	4.4	4.58	13183753	33683277	8.06
2	4.58	4.7	4.78	93449563	418051993	100
3	4.78	4.81	5.5	3749030	39204618	9.38
4	6.69	6.73	6.77	986248	1761400	0.42

--- End Of Report ---



- Why is sodium carbonate non-hazardous? Where do you find this chemical in everyday life?

5. Show the flow chart for the purification, begin with 2-methylcyclohexanol and 85% phosphoric acid.

6. Record EXACT amounts of reagents used, observations, and any changes you have made to the written procedure. Record the boiling point of your product.

POST LAB INSTRUCTIONS

The following items should be included in your post-lab conclusions:

1. Calculations.

Calculate the **amount** of 2-methylcyclohexanol (in moles) actually used:

$$\underline{3} \text{ mL} \times \underline{0.93} \text{ g/mL} \times \underline{1/114.2} \text{ mol/g} = \underline{0.0244} \text{ mol}$$

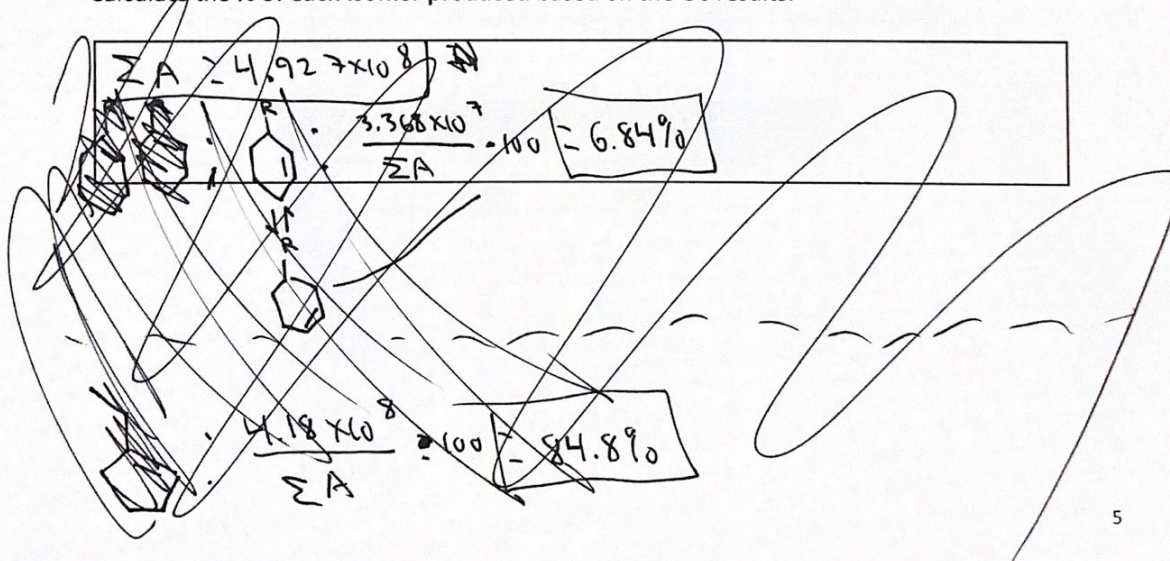
Calculate **theoretical yield** of alkene isomers:

$$\underline{0.0244} \text{ mol} \times \underline{96.1} \text{ g/mol} = \underline{2.348} \text{ grams}$$

Calculate the **% yield**:

$$\underline{0.712} \text{ grams isolated} / \text{grams theoretical} \times 100\% = \underline{30.33} \%$$

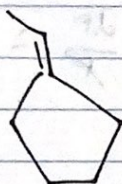
Calculate the **% of each isomer produced** based on the GC results:




$$\frac{3.37 \times 10^7}{\Sigma A} \cdot 100 = 6.84\%$$



$$\frac{4.18 \times 10^8}{\Sigma A} \cdot 100 = 84.8\%$$



$$\frac{3.92 \times 10^7}{2A} \cdot 100 = 7.96\%$$



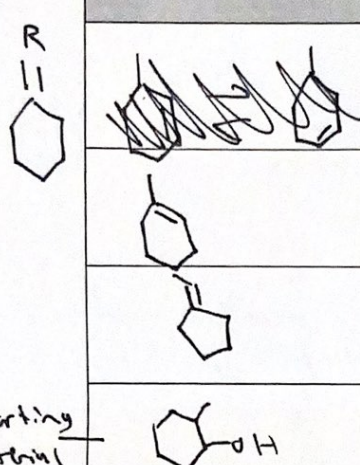
$$\frac{1.76 \times 10^6}{\sum A} = 100 \sqrt{\quad} = 0.35890$$

$$\Sigma A \approx 4.927 \times 10^8$$

2. Results.

theoretical yield of isomers	percent yield of isomers	appearance of product
2.348 g	30.33 %	Clear

GC/MS Results

	isomer	retention Time	area percentage
	isomer	4.4	8.06
	isomer	4.7	100
	isomer	4.81	9.38
	Starting material	6.73	0.42

IR

functional group	cm ⁻¹
C _{sp} ² - H	2834, 2855, 2922
C _{sp} ² - H	3001

Experiment 7 "Dehydration"

The IR spectra had about 3 peaks at 2834, 2855, and 2922 cm^{-1} which correspond to $\text{C}_{\text{sp}^3}\text{-H}$ groups and one peak at 3001 cm^{-1} which corresponds to $\text{C}_{\text{sp}^2}\text{-H}$ groups. This result is expected because each isomer only contains sp^2 and sp^3 carbons bonded to hydrogens. The GC spectra has peaks at retention times of 4.4 (a), 4.7 (b), 4.81(c), and 6.73(d). Comparing these results to the sample results, (a) corresponds to methelenecyclohexane, (b) corresponds to 1-methelcyclohexene, (c) corresponds to ethyldenecyclopentane, and (d) corresponds to the starting material: 2-methylcyclohexanol. (b) was the peak with the largest area so 1-methelcyclohexene is the major product.

The IR spectra for the starting material only has peaks correlating to $\text{C}_{\text{sp}^3}\text{-H}$ groups and an OH stretch. The IR spectra for the product has similar peaks for $\text{C}_{\text{sp}^3}\text{-H}$ groups but instead of a peak for the OH stretch it has a peak for the $\text{C}_{\text{sp}^2}\text{-H}$ groups or the alkenes. Even though there was a peak corresponding to the starting material in the GC spectra, it has a very small area compared to the other products. This suggests that our product was pure but there was some starting material mixed with the product.

It was hypothesized that the most stable product would follow Zaitsev's rule and be the isomer with the most substituted alkene which is the 1-methelcyclohexene. 1-methelcyclohexene shows on the GC spectra with the largest peak with a product ratio of 84.8% so the results match the prediction. The next most stable isomer would be the ethyldenecyclopentane with a product ratio of 7.96%. This again matches Zaitsev's rule because the alkene is just as substituted as the 1-methelcyclohexene but it has a 5 carbon ring rather than a 6 carbon ring which is not as stable in this case because of the steric strain. The next most stable isomer was methelenecyclohexane which has a less substituted alkene and a product ratio of 6.84%. 3-methelcyclohexene did not appear on the GC spectra. Zaitsev's rule applies to this experiment because it's an elimination reaction so the products have an alkene.

When adding THF and pyridinium tribromide to the product, the solution turned yellow-orange for a few seconds before returning clear. This was done twice and after the second time a white precipitate was observed. This precipitate was possibly due to some starting material mixed with the product.