Rowan College at Burlington County

CHE 241

Lab Three: Synthesis of Cyclohexanol

Video Explanation of this lab

What is Refractive Index?	Here's a video Here's the accompanying slideshow Here's
What is GC?	Here's a video Here's the accompanying slideshow

In the lab you carried out the procedure given in the lab and as written below.

> **"Reaction:** In a 500 ml standard taper round bottom flask, add 16 mls of concentrated sulfuric acid and 8 mls of water. Cool the reaction flask in an ice bath until cold to the hand and then add 20 ml of reagent grade cyclohexene.
>
> Stopper the flask with a greased ground glass stopper and shake vigorously to mix for at least 15 minutes or until there is one homogeneous layer, holding the stopper in. Let the reaction mixture sit an additional 10 minutes to increase completeness of reaction.
>
> **Purification:** Add 250 ml of water and distill the reaction mixture via a simple distillation using a heating mantle as the heat source and a 250 ml standard taper round bottom flask as a receiver. Distill until the distillate in the condenser is clear, with no oily droplets present, and one phase is left in the pot.
>
> Saturate the distillate with sodium chloride and let this mixture stand for 15 minutes.
>
> Transfer the mixture to a 250 ml separatory funnel. Rinse the reaction flask three times with 10 ml of diethyl ether and add all the ether rinsings to the separatory funnel. Extract the reaction mixture with the ether, separate the layers and pour the ether layer into a 125 ml erlenmeyer flask. Add anhydrous potassium carbonate to the ether layer until free flowing and allow it to stand for at least 10 minutes.
>
> Gravity filter this mixture through fluted filter paper into a 250 ml suction filtration flask.
>
> Vacuum evaporate the ether from the mixture using the set-up demonstrated in the laboratory. Transfer the remaining liquid to a 50 ml standard taper round bottom flask. Distill via a simple distillation using a heating mantle as the heat source. Collect the low boiling fraction in a 100 ml standard taper flask. Change the flask at 120 °C to a labelled, weighed bottle and collect from 120-162 °C. Adjust the variac setting to control frothing. Watch the liquid level in the pot. The distillation should be stopped when the liquid level becomes too low, the temperature drops and/or signs of charring are seen. Weigh the sample and sample bottle to obtain the number of grams of cyclohexanol produced.
> **Characterization**: Determine the refractive index of the product. Determine the infrared spectrum of both the reactant and the product. The instructor or instructional assistant will demonstrate the operation of the Abbe refractometer and the infrared spectrophotometer."

During characterization, you were able to get this information about the final product:

Corrected Refractive Index of product: 1.4605

Literature refractive index of cyclohexanol: 1.4641
Literature refractive index of cyclohexene: 1.4465

Mass of final product: 10.32 grams

Percent yield of product: 53%

Infrared spectra of your product, cyclohexanol

Infrared spectra of the pure reactant, cyclohexene

A GC of the reaction mixture

You were able to get all your data when, unfortunately, at the end of the lab, you dropped your lab notebook and your spectra was all mixed up. So you have some spectra and your lab book of data, and now you have to write a lab conclusion. Here's your task:

1) Sort out the spectra.

    a) Identify whether we are looking at the IR of cyclohexene (the reactant) or cyclohexanol (the product) or the GC of the product mixture.

    b) For each IR, identify the characteristic peaks.

    c) For the GC, identify the peak that corresponds to the product, cyclohexanol.

2) Write a thorough conclusion.

    It is not up for debate that you made cyclohexanol. You did. Great job! What you do need to do is defend how you know that you did using the data in the lab. Just like always, defend your answer to the purpose. The purpose of the lab is to "synthesize pure cyclohexanol" Your lab conclusion should be two-fold. Support how you know you made cyclohexanol. Support how you know its pure.

    a) How do you know that the product you made is cyclohexanol? This can be done using IR spectra and refractive index data.

    b) How do you know the purity of your product? Explain whether the product is pure or not and how you know. You should use refractive index data and GC data to come to this conclusion.

c) Comment on the percent yield. Based on your purity, is 53% a good yield or a poor yield? How do you know?

3) Identify potential errors and explain their impact on the lab

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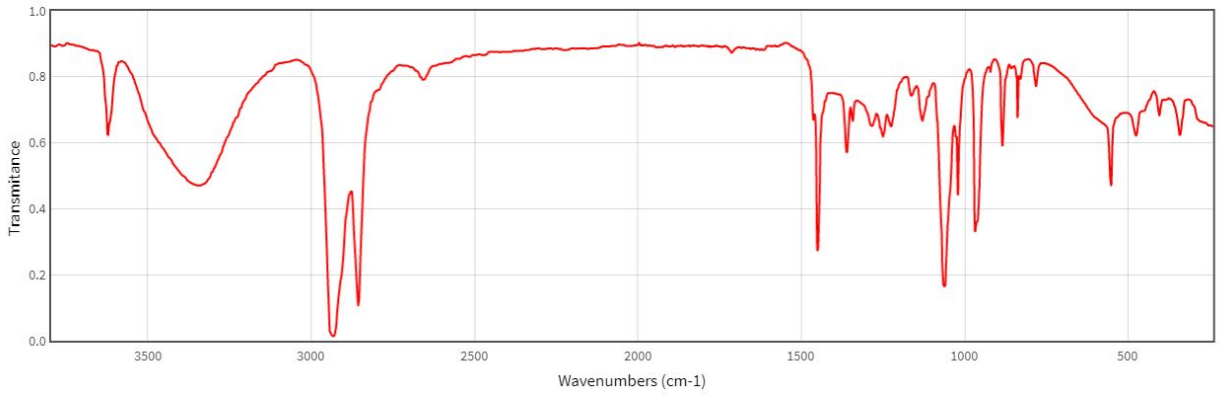
I get it. We aren't doing this lab live. But, that still doesn't mean that there aren't some places that you could have potentially screwed up. Take some time to read over the procedure and identify three (3) specific errors that could have been made and what the impact they would have had on the outcome of the lab. Here is an example::

The mixture was not distilled long enough. Because of this, there was some product left behind in the pot. This would have lowered the overall percent yield.

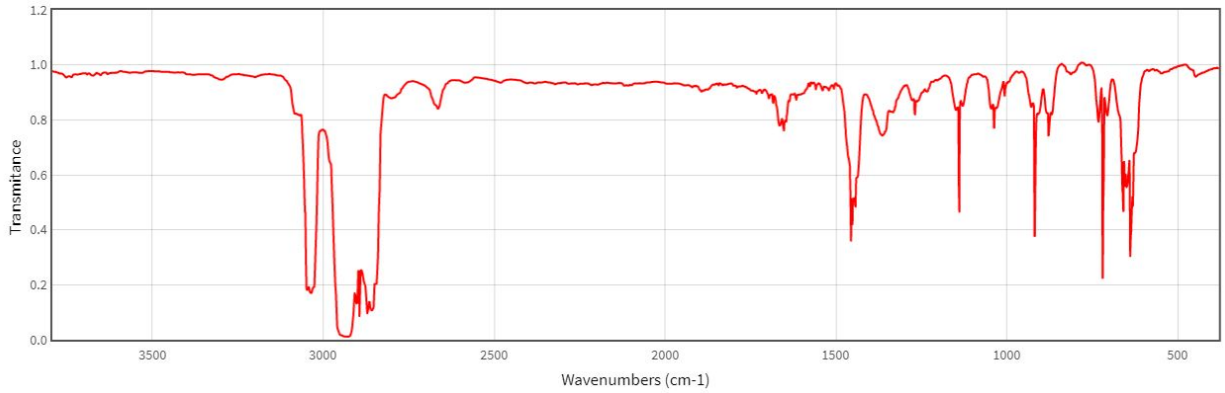
Spectra 1 -



What is this?

Identify the important peaks.

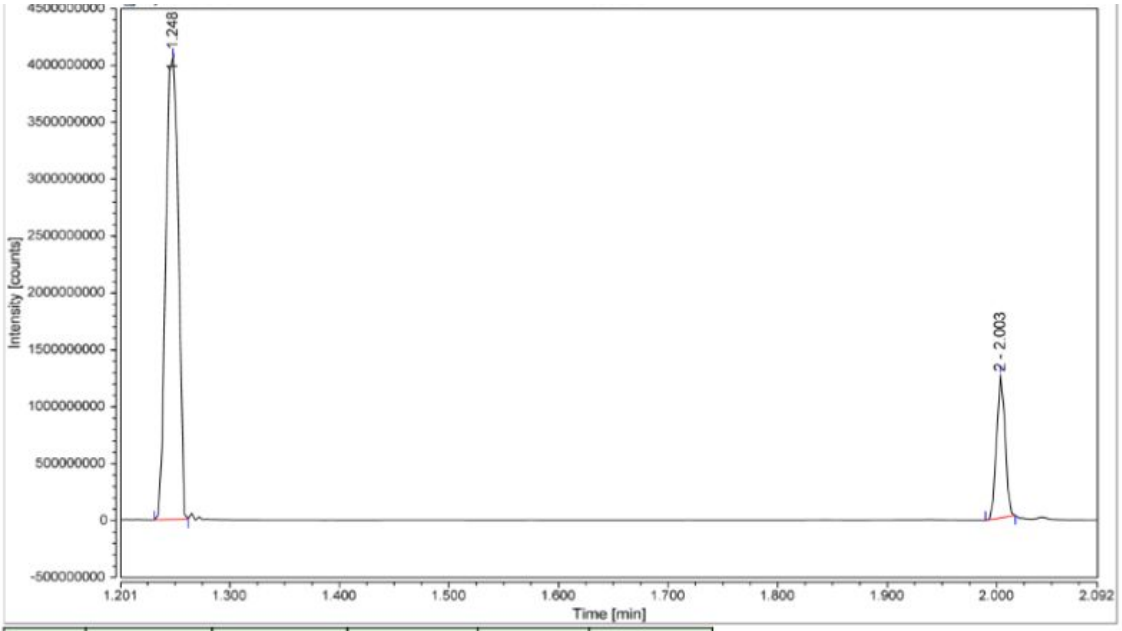
Spectra 2 -



What is this?

Identify the important peaks.

Spectra 3 -



No.	Retention Time min	Area counts*min	Height counts	Relative Area %	Relative Height %
1	1.248	54369801.483	4057018622.986	82.70	76.35
2	2.003	11374120.819	1256530997.205	17.30	23.65
Total:	**65743922.302**	**5313549620.191**	**100.00**	**100.00**	

What is this?

Identify the important peaks.