Chem 220 Organic Chemistry I Fall 2024 - Ilana Berlin/Assignments/Week 8: Dehydration and 13C NMR/Dehydration Notebook Entry

Paul Gladen - Oct 28, 2024, 1:03 PM CDT

Assignment #16 - Alcohol Dehydration Notebook Entry

You cannot edit this entry after it is graded.

Description	Due at 5:00 pm the day following your lab section.
I worked in a group with	
The work for this assignment is in	My notebook

Grade **9 / 10**

Graded on Oct 28, 2024, 1:03 PM CDT

Caroline Slone - Oct 28, 2020, 1:06 PM CDT

Title

Ilana Berlin - Oct 22, 2024, 12:17 PM CDT

Dehydration of Cyclohexanol

Ilana Berlin

Tuesday, October 22 2024

Paul Gladen - Oct 13, 2022, 2:08 PM CDT

Purpose (goal, technique, measurement)

Ilana Berlin - Oct 22, 2024, 12:29 PM CDT

Obtain cyclohexane by dehydration of cyclohexanol using sulfuric acid as a catalyst. Characterize product of dehydration reaction using IR and proton NMR spectra to check if the wanted product, cyclohexane, was obtained.

Paul Gladen
Oct 28, 2024, 1:02 PM CDT

isolation via fractional distillation

Write a comment...

@ Add Comment

Caroline Slone - Oct 28, 2020, 1:06 PM CDT

References

Ilana Berlin - Oct 29, 2024, 12:38 PM CDT

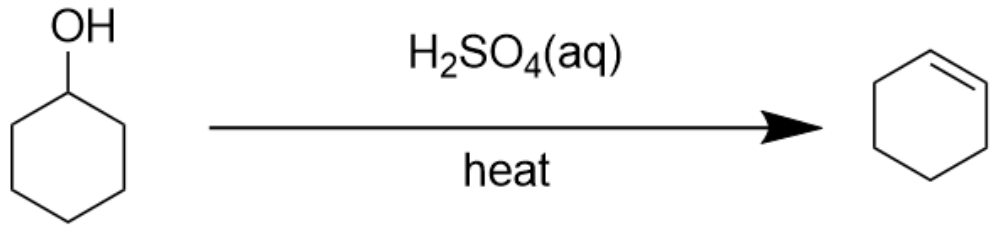
Kateley, L. J., *Guide for Organic Chemistry Laboratory*, Seventeenth edition, Lake Forest College, 2011

Google

Caroline Slone - Oct 28, 2020, 1:07 PM CDT

Net Reaction Equations

Ilana Berlin - Oct 22, 2024, 1:01 PM CDT



Insert drawings of the net reaction equations using ChemDraw (no mechanisms here).

Caroline Slone - Oct 28, 2020, 1:10 PM CDT

Reagent Table & Calculations

Ilana Berlin - Oct 22, 2024, 5:33 PM CDT

Reagent/Product	MW (mg/mmol)	Density (g/mL)	Equiv.	bp (°C)	mmol	mass (mg)	volume (mL)

Cyclohexanol	100.158	0.9624	1	161.8	30.25	3030	2.916
Sulfuric Acid (9M)	n/a	n/a	0.46	n/a	14.00	n/a	1.556
Cyclohexene (theoretical)	84.16	0.7785	0.0119	74.5	6.82	574	0.737

Calculations:

2916uL cyclohexanol = 2.916mL (1mL/0.9624g) = 3.030g = 3030mg (1mmol/100.158mg) = 30.25mmol

1556uL sulfuric acid = 1.556mL = 14.00mmol

74.5ºC

Vial Weight: 9.966g Empty 10.570 with solution

solution mass: 0.574g (1mL/0.7785g) = 0.737mL = 574mg (1mmol/84.16mg) = 6.82mmol

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Experimental

Ilana Berlin - Oct 22, 2024, 5:43 PM CDT

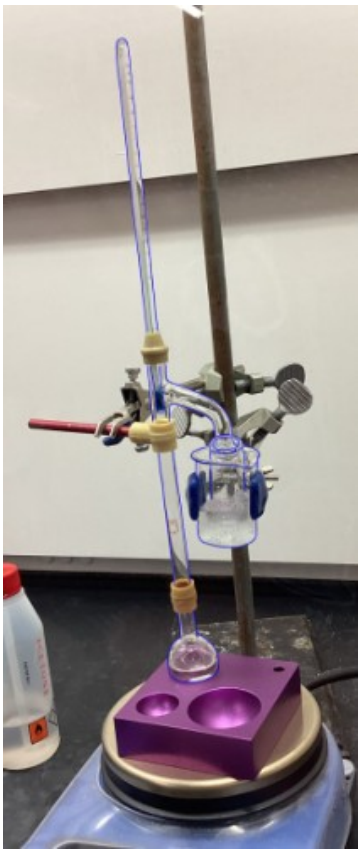


Figure 1. Apparatus

A long pipe was connected to spout with rubber joint. Funnel was placed in rubber holder on the other side of the spout. Thermometer was lowered until the tip was around even with the base of the spout. 30mL beaker was filled with ice for ice bath and empty vial was placed inside the ice bath. More ice was added as needed throughout the experiment to maintain the ice bath. The beaker was raised till the mouth of the spout was inside the empty vial.

The appropriate amounts of cyclohexanol (2916µL) and sulfuric acid (1556µL) were pipetted into a small round bottom flask. A stir bar was quickly rinsed with acetone, dried, and added to the round bottom flask. The flask was attached to the long pipe with a rubber joint. The entire apparatus was leveled so that bottom of the round bottom flask settled in the holder on the hot plate. The hot plate was turned on to a temperature of around 140°C and a slow stir. The temperature of the hot plate was increased to around 250°C after a long amount of time passed with nothing happening. The solution in the round bottom flask boiled, moved up the pipe, and condensed. The solution dripped from the spout starting when the thermometer showed a temperature of 74.5°C.

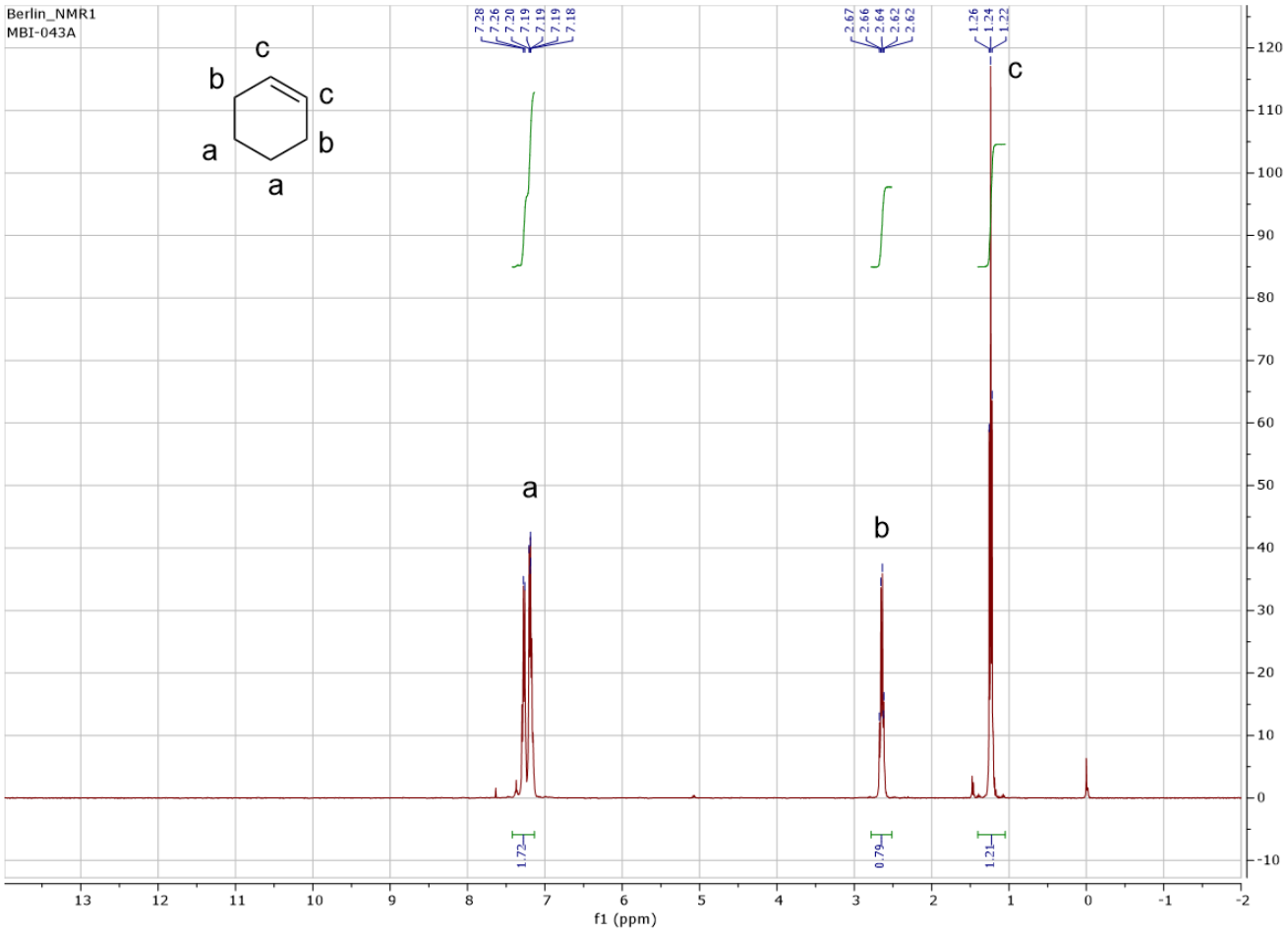
~2mL of solution dripped in to the empty vial. Then the vial was capped and removed from the apparatus. Anhydrous Na_2SO_4 was added to remove the remains of water from the solution. The vial with anhydrous Na_2SO_4 was set in a fresh ice bath to rest for 10 minutes. A clean vial and cap were weighed for a total mass of 9.966g. The solution was poured from the anhydrous Na_2SO_4 vial into the clean vial. The vial was reweighed to get a mass of 10.570g. The mass of the solution, 0.574g, was calculated by subtracting the mass of the vial with solution by the mass of the empty vial.

IR and Proton NMR spectra were run and percent recovery of 22.5% was calculated.

Caroline Slone - Oct 28, 2020, 2:36 PM CDT

Data Analysis

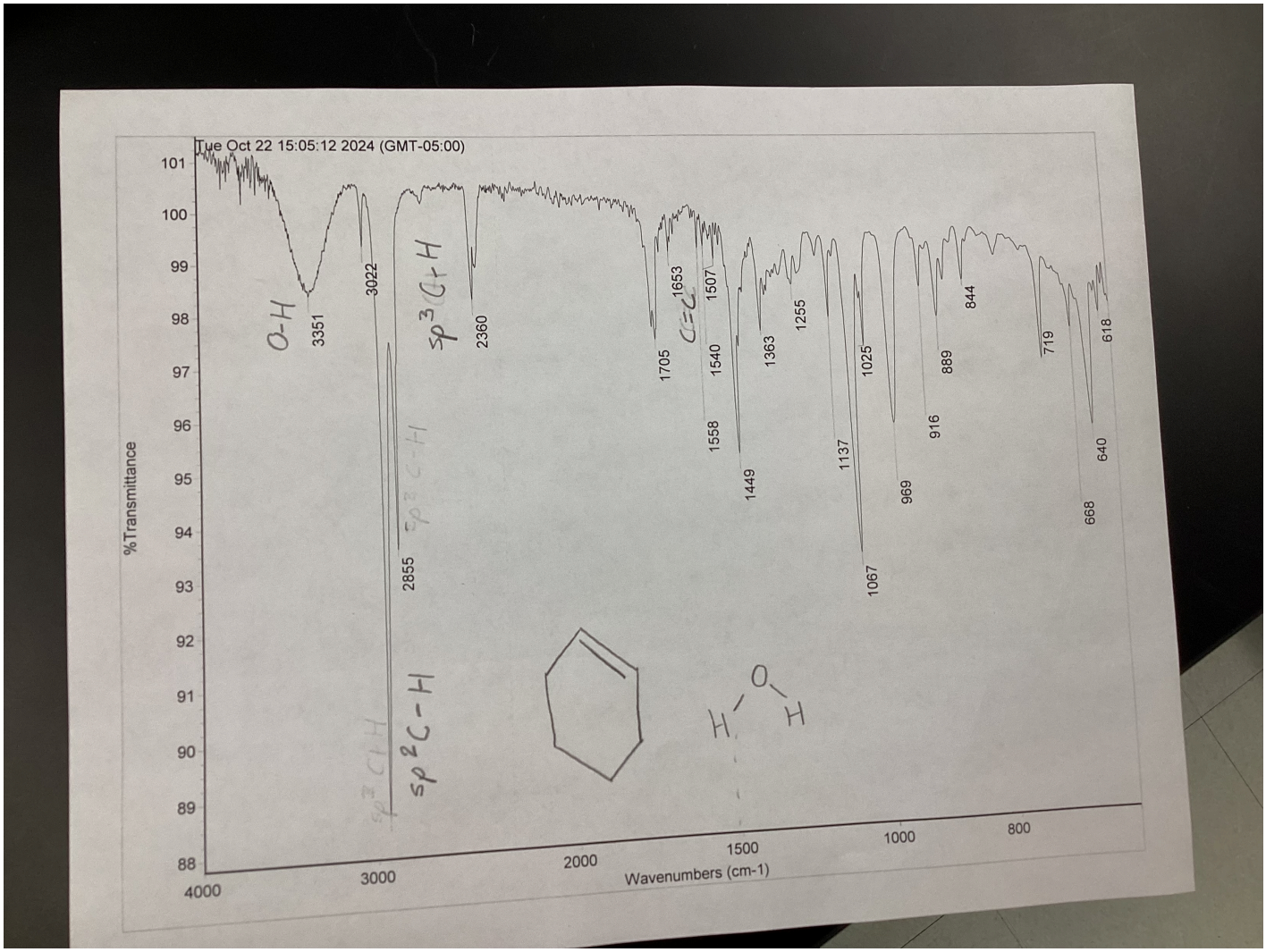
Ilana Berlin - Oct 22, 2024, 5:47 PM CDT



a: septet 2

b: quintet 1

c: triple 2



Insert your annotated IR (hand drawn) and NMR (MNova) here.

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Oct 28, 2024, 1:03 PM CDT

This spectra is ethyl benzene -1

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@

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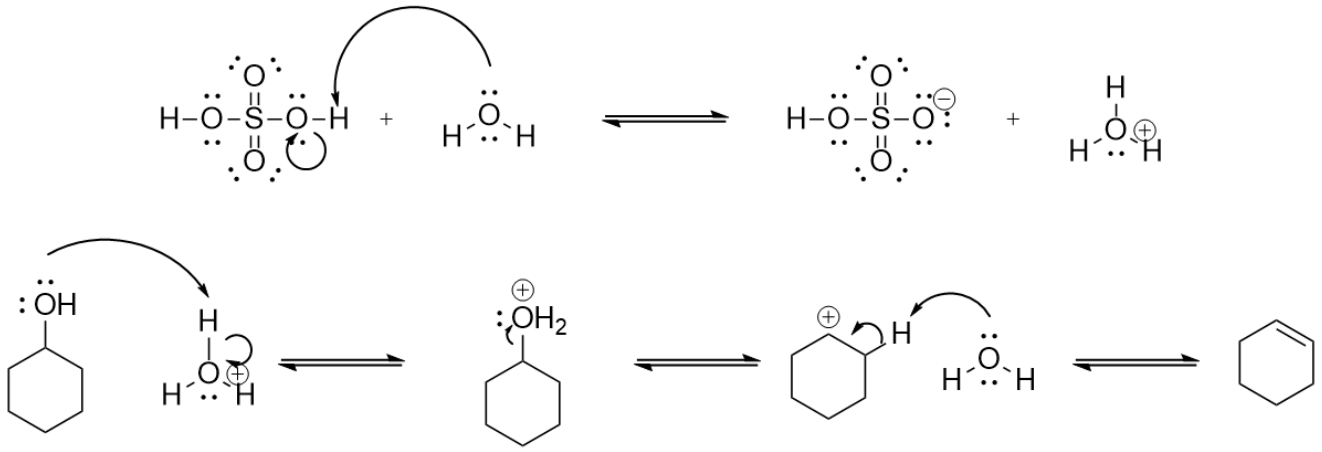
Paul Gladen - Oct 13, 2022, 1:57 PM CDT

Reaction Mechanism

Ilana Berlin - Oct 22, 2024, 5:48 PM CDT



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Conclusions

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Cyclohexene has a much lower boiling point than cyclohexanol. As the solution heats the cyclohexene vaporizes before the cyclohexanol. By isolating the cyclohexene all of the cyclohexanol should theoretically be converted to cyclohexene.

The percent yield was 22.5%.

The initial temperature of the hot plate should have been much higher has the temperature shown on the thermometer did not start increasing until the hot plate was turned up to over 200°C. As the temperature took so long to increase, the ice in the ice bath melted really quickly and eventually there was no room for more ice without overflowing the beaker. The ice bath should have been colder and water from the ice bath may have spilled into the vial because even after dehydrating there was still water in the solution. To improve results a larger beaker should be used for the ice bath and more dehydrant should be added.

Paul Gladen - Oct 13, 2022, 1:57 PM CDT

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