Chem 220 Organic Chemistry I Fall 2024 - Ilana Berlin/Assignments/Week 9. Hydrogenation, Mixed Spectroscopy/Hydrogenation Notebook Entry

Paul Gladen - Nov 08, 2024, 1:46 PM CST

Assignment #19 - Hydrogenation 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 **8.5 / 10**

Graded on Nov 08, 2024, 1:46 PM CST

Caroline Slone - Nov 11, 2020, 1:50 PM CST

Title

Ilana Berlin - Oct 29, 2024, 12:14 PM CDT

Catalytic Hydrogenation of A Carbon = Carbon Double Bond

Ilana Berlin

10/29/2024

Caroline Slone - Nov 11, 2020, 1:50 PM CST

Purpose

Ilana Berlin - Oct 29, 2024, 12:27 PM CDT

Use hydrogenation mechanism to form hydrocinnamic acid from *trans*- cinnamic acid using palladium as a catalyst in a methanol solution. Isolate product using vacuum filtration to remove catalyst and recrystallization.

Paul Gladen
Nov 07, 2024, 11:27 AM CST

characterize by IR and NMR -1/2

Write a comment...

@ Add Comment

Caroline Slone - Nov 11, 2020, 1:51 PM CST

References

Ilana Berlin - Oct 29, 2024, 12:28 PM CDT

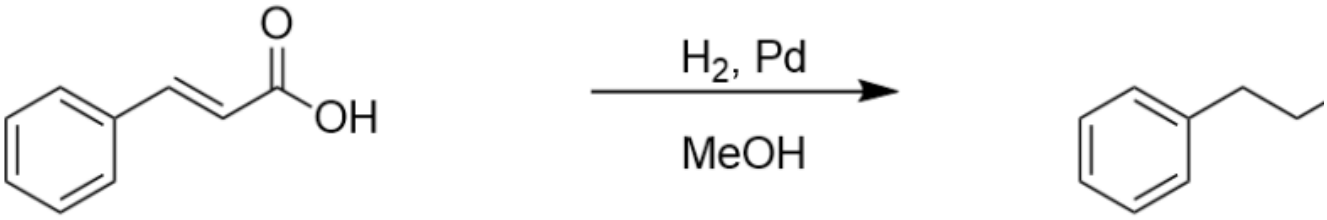
Kateley, L. J., *Guide for Organic Chemistry Laboratory*, Seventeenth edition, Lake Forest College, 2011

Caroline Slone - Nov 11, 2020, 1:51 PM CST

Net Reaction Equation

Ilana Berlin - Oct 29, 2024, 1:23 PM CDT

Insert drawing of the net reaction equation for the catalytic hydrogenation reaction (no mechanisms here).



Caroline Slone - Nov 11, 2020, 1:52 PM CST

Reagent Table & Calculations

Ilana Berlin - Oct 29, 2024, 2:23 PM CDT

Insert Reagent Table from the Pre-Lab sheet.

reagent & product	FW (g/mol)	mp ^{o}C	mmol	mass (mg)
trans - cinnamic acid	148.16	133 - 134	1.04	154
hydrocinnamic acid	150.18	47 - 49	1.04	156 theoretical yield

Calculations from the Reagent Table:

Show your calculation for the mass of *trans*-cinnamic acid that corresponds to 1.00 mmol of *trans*-cinnamic acid .

154mg(1mmol/148.16mg) = 1.04mmol

Starting with 1.00 mmol of *trans*-cinnamic acid, show your calculations for the theoretical mmoles of hydrocinnamic acid product and the theoretical yield in mg.

1.04mmol trans = 1.04mmol hydro (150,18mg/1mmol) = 156mg

Percent Yield Calculations for the Experiment:

mass of trans-cinnamic acid reagent used: _____154_____ mg

mass of filter flask (tare): _____30574_____ mg

mass of filter flask + hydrocinnamic acid product: _____30717_____ mg

mass of hydrocinnamic acid product: _____143_____ mg

Using the actual amount of trans-cinnamic acid used in the experiment (see video), recalculate the theoretical yield below.

Theoretical Yield: 156mg

Use this theoretical yield value and the mass of the hydrocinnamic product obtained from the reaction to calculate the percent yield for the reaction below.

Percent Yield : 143mg / 156mg x 100 = 91.7%

Experimental

Ilana Berlin - Oct 30, 2024, 7:32 PM CDT

A balloon was attached to a provided side arm flask. A rubber band was used to ensure that the seal between the side arm and the balloon was secure as possible. The reactant, 0.154g of *trans* cinnamic acid, the solvent, 5mL of methanol, and the catalyst, 0.010g of 5% palladium, were added to the flask. A stir bar was also added. The flask was capped with a rubber septum.

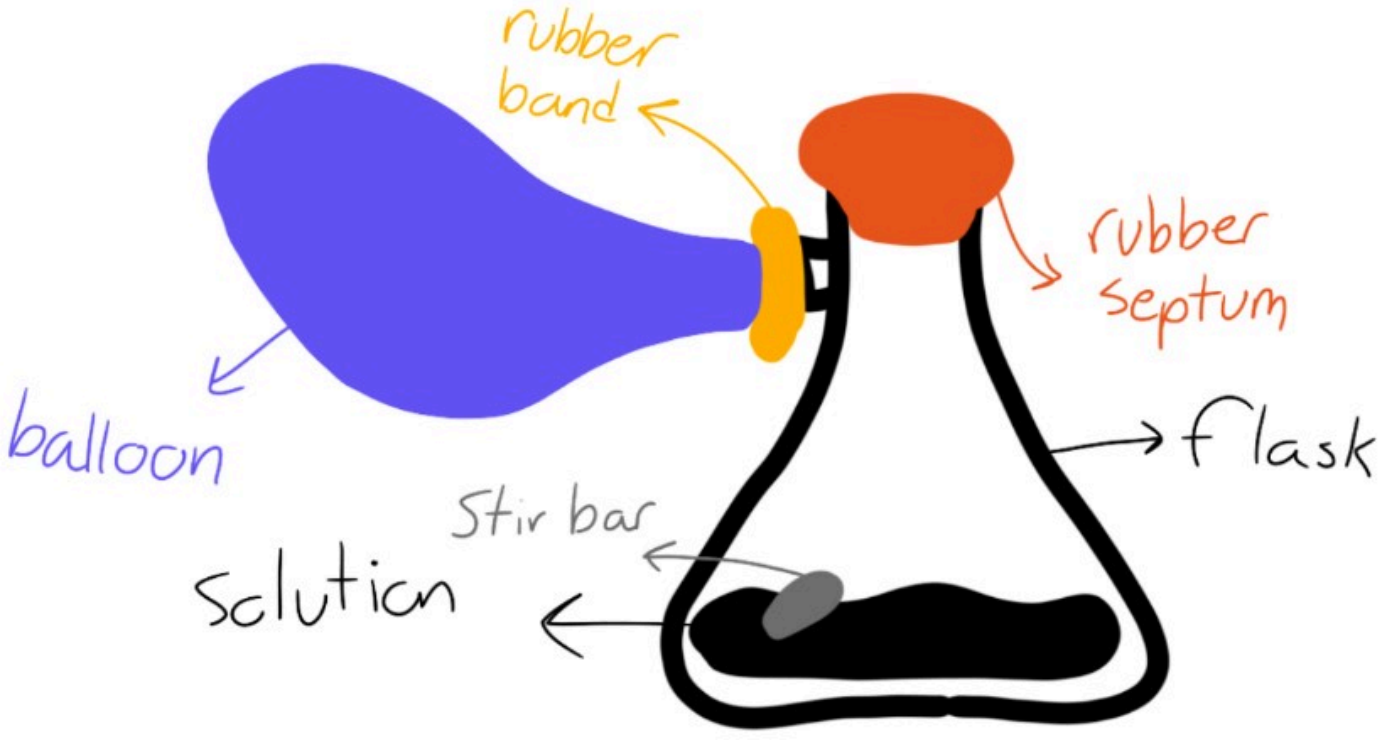
The flask and attached balloon were filled and flushed with H_2 gas. After the H_2 began quickly escaping the apparatus, the rubber septum was replaced to ensure a good seal and the system was refilled and flushed with gas. The flask was left to stir for around 30 minutes.

While the flask was stirred, a clean dry side arm flask, later used for vacuum filtration, was weighed and the apparatus for vacuum filtration was constructed. An additional fiber glass filter was added to the Hirsch funnel to ensure filtration. The solution was carefully transferred from the side arm flask to the vacuum filtration apparatus. A glass pipet was used to transfer the solution onto the fiber glass filter to ensure as much of the catalyst as possible was removed.

After there was no sign of the catalyst (black spots) in the solution, the vacuum side arm flask was removed from the apparatus and placed in a bath of warm tap water. The solution was dried using a gentle stream of air until the methanol evaporated and the remaining cinnamic acid crystallized. The flask was reweighed and the mass and percent yield of the cinnamic acid was calculated.

IR and NMR spectra were run and analyzed. Lab station was cleaned and waste was properly disposed of.

Include your description of the experiment & procedure here.



Paul Gladen
Nov 08, 2024, 1:42 PM CST

"waste was properly disposed of" be more specific

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@

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Caroline Slone - Nov 11, 2020, 2:31 PM CST

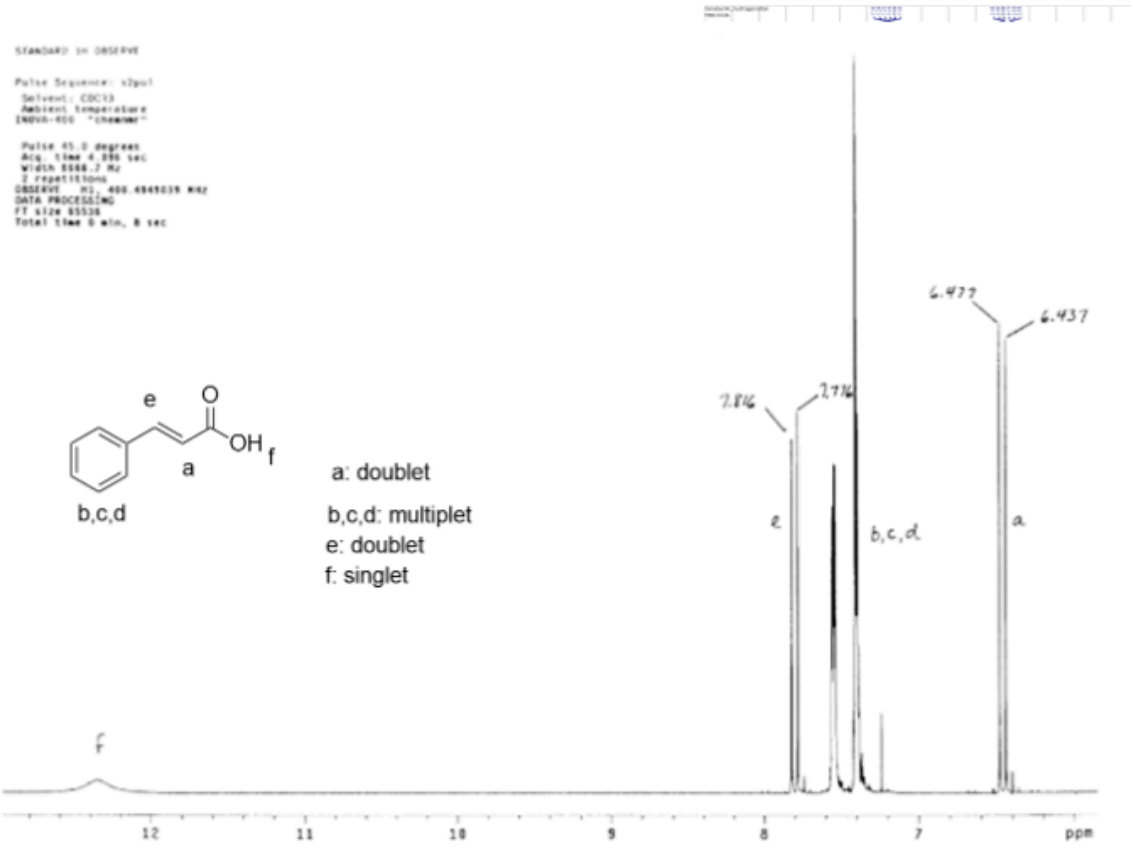
sketch of apparatus here

Caroline Slone - Nov 11, 2020, 2:32 PM CST

Data Analysis

Ilana Berlin - Oct 29, 2024, 7:19 PM CDT

Insert the ^{1}H NMR spectrum of trans-cinnamic acid substrate here. The substrate has two vinyl hydrogen doublets, one at ~6.4 ppm and the other at ~7.8 ppm. Draw and label the substrate structure below; indicating which vinyl hydrogen results in which doublet. Use peak labels on this spectrum to calculate the coupling constant ($^{3}J_{HH}$) for the vinyl hydrogens and thus verify stereoisomeric orientation of these vinyl hydrogens. A typical $^{3}J_{HH}$ value for cis vinyl H is about 8 Hz and a typical $^{3}J_{HH}$ value for trans vinyl H is 16 Hz. Include the vinyl $^{3}J_{HH}$ calculations here.



a)

(6.477 - 6.437) x 400 = 16.0Hz - trans

e)

(2.816 - 2.716) x 400 = 40Hz

Paul Gladen
Nov 08, 2024, 1:44 PM CST

coupling constant should match - both are 16 Hz

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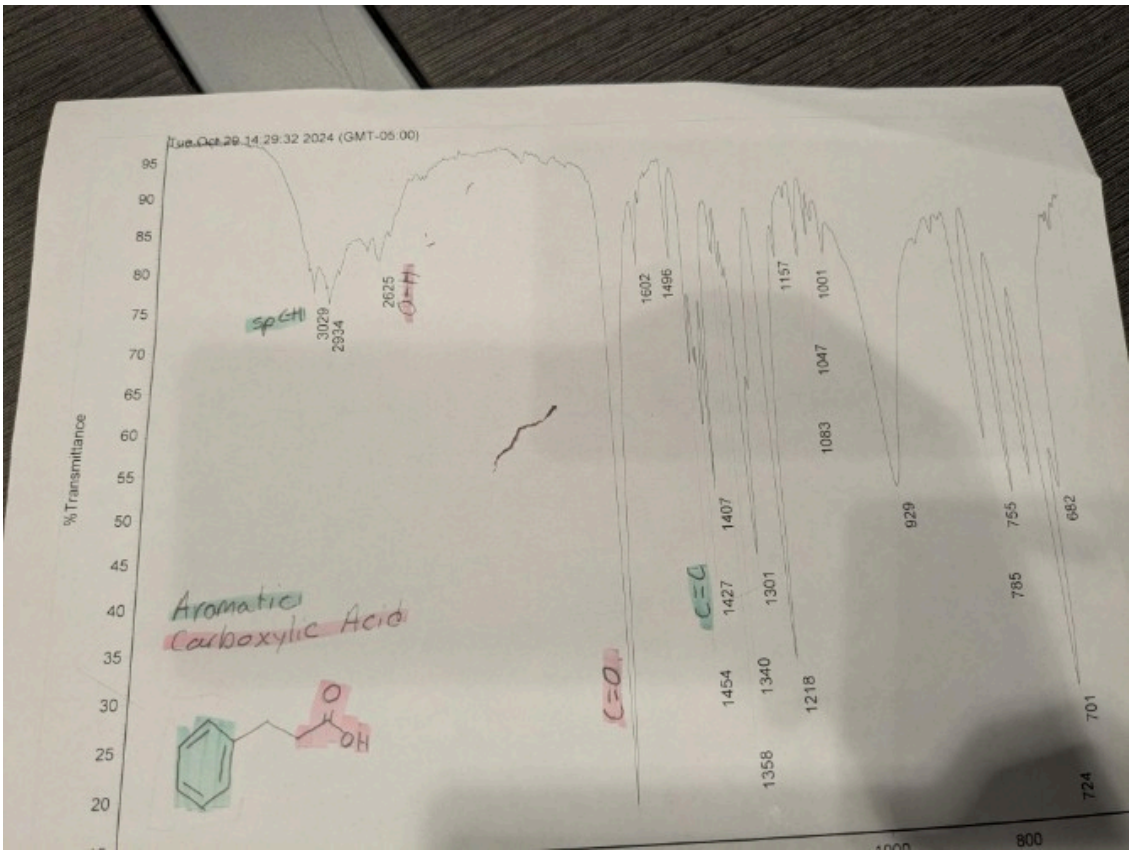
Ilana Berlin - Oct 30, 2024, 7:09 PM CDT



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Paul Gladen
Nov 08, 2024, 1:44 PM CST

sp2 and sp3 C-H? -1/2

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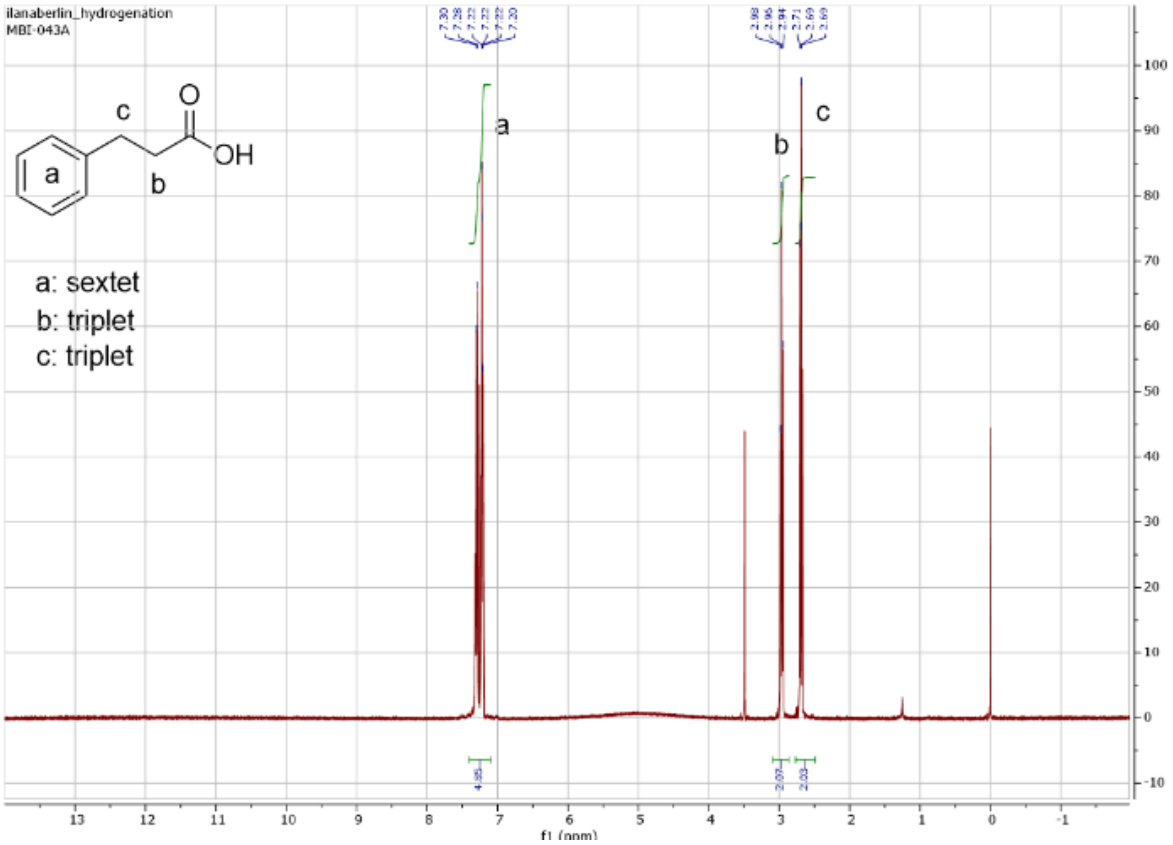
Ilana Berlin - Oct 29, 2024, 7:19 PM CDT

Insert ^{1}H NMR spectrum of the hydrocinnamic acid product. Draw the structure of hydrocinnamic acid and label the 2 methylene groups on the structure and on the NMR spectrum. Calculate the coupling constants for the methylene (CH_2) triplets.



coupling constant:

c)

(2.98- 2.96) x 400 = 8.00Hz

(2.96 - 2.94) X 400 = 8.00Hz

d)

(2.71 - 2.69) x 400 = 8.00Hz

(2.69 - 2.69) x 400 = 0 Hz

Paul Gladen
Nov 08, 2024, 1:45 PM CST

0 Hz? -1/2

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Caroline Slone - Nov 11, 2020, 2:32 PM CST

Conclusions

Answer the following in your conclusions.

Did the reaction go to completion? What evidence is there to support that the reaction took place?

What is the percent yield? Discuss possible sources for loss of product.

Conclusion:

The balloon continued to deflate relatively quickly after being filled with H_2, even after the rubber septum was replaced, so it is possible that the reaction was not able to go to completion and turn all the *trans*-cinnamic acid into cinnamic acid. The reaction definitely took place, indicated by the change in temperature of the side arm flask. It started at room temperature and got colder as the reaction preceded till it was removed from the stir plate.

The percent yield was likely higher than the real percent yield because remaining moisture added to the mass even after the solution had been dried. The percent yield was also effected by loss of product to the residue left in the side arm flask where the reaction took place and the small crystals that were blown out of the vacuum filtration side arm flask while the methanol was being dried off. There was no visible catalyst residue, black spots, left in the solution after vacuum filtration and the crystals formed after drying were a clean white so mass of catalyst should not have skewed the percent yield.