Chem 220 Organic Chemistry I Fall 2024 - Ilana Berlin/Assignments/Week 10: Williamson Ether Synthesis/Williamson Ether Experiment

Paul Gladen - Nov 14, 2024, 11:38 AM CST

Assignment #2 - Williamson Ether Experiment

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 **10 / 10**

Graded on Nov 14, 2024, 11:38 AM CST

Ilana Berlin - Nov 12, 2024, 12:04 PM CST

Williamson Ether Synthesis

11/12/2024

Ilana Berlin

Caroline Slone - Aug 23, 2022, 3:52 PM CDT

Title & Date

Caroline Slone - Aug 23, 2022, 3:52 PM CDT

Purpose

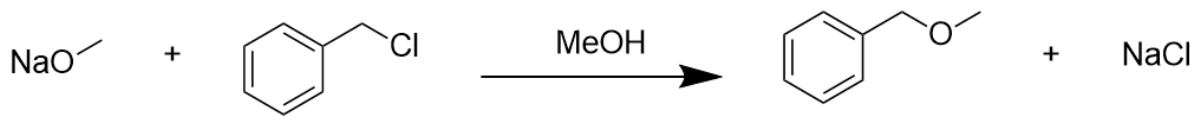
Ilana Berlin - Nov 12, 2024, 11:13 AM CST

Use a Sn2 reaction mechanism to form benzyl methyl ether and sodium chloride from benzyl chloride and sodium methoxide using methanol as a solvent. Separate the benzyl methyl ether using a separatory funnel and dry it using anhydrous sodium sulfate. Characterize product using IR and H NMR

Caroline Slone - Aug 23, 2022, 3:52 PM CDT

Net Reaction Equation(s)

Ilana Berlin - Nov 12, 2024, 11:16 AM CST



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References

Ilana Berlin - Jan 16, 2025, 10:05 AM CST

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

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Reagent Table & Calculations

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Include reagent table from Pre-Lab sheet.

reagents, solvent, & product	FW, g/mol	density, g/mL at 25 °C	bp, °C	mmol	mass, mg	volume, µL
methanol (solvent)	32.03	0.791	67.4	excess	NA	NA
sodium methoxide	54.02	NA	NA	13.0	702	NA
benzyl chloride	126.59	1.10	179	9.99	1.27×10^3	1.15

benzyl methyl ether	122.17	0.987	174	9.99 (theoretical)	1.22×10^3 (theoretical)	1.24

Remember to <u>recalculate</u> the amounts based on the reagent amounts actually used in the experiment!

Show all your calculations here.

Sodium methoxide: 702mg (1mmol/54.02mg) = 13.0mmol

benzyl chloride: 1.15mL (1.10g/1mL)(1mg/1x10^{-3}g) = 1.27 x 10^3mg(1mmol/126.59mg) = 9.99mmol

benzyl methyl ether (theoretical) : 9.99mmol(122.17mg/1mmol) = 1.22 x 10^3mg (1 x 10^{-3}g/1mg)(1mL/0.987mg) = 1.24mL

percent yield: (1.09 x 10^3/ 1.22 x 10^3) x 100 = 89.3%

Include both your theoretical yield and experimental percent yield calculations here.

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Experimental Section

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Include a **experimental** based on the experiment.

The reflux apparatus was constructed. A long pipe was attached to a spout (Claisen adaptor) with a rubber joint. The spout was pointing up toward the long pipe. A round bottom flask was attached to the other side of the spout with a rubber joint with a red handle attached. A rubber septum was placed at the end of the spout. The apparatus was clamped to the stand by the red handle and a claw holding the top of the long pipe.

A pair of blue nitrile gloves were put on before any of the chemicals were handled. All chemicals were handled inside the hood. The round bottom flask was removed from the apparatus. A stir bar was added to the flask. 0.702 g of sodium methoxide was added to the flask followed by 3mL of methanol. A funnel was used to reduce spilling. The methanol helped wash any residue on the funnel into the flask. The flask was placed back on the apparatus with the hot plate set to 100ºC and the stir bar at a low speed. 1.15mL of benzyl chloride was added to the flask. It was by syringed through the rubber septum at the end of the funnel. It was added all at once but it should have been slowly dripped of the course of a minute. The syringe was cleaned with acetone, dried, and another 0.5mL of methanol was added via syringe through the rubber septum to wash down any benzyl chloride. The solution bubbled and turned foamy and slightly yellow in color. The flask was left to reflux and stir for around 25 minutes.

The heat was turned off and the apparatus was lifted above the hot plate. The solution was cooled to room temperature and transfer to the centrifuge tube. The round bottom flask was washed with methanol to ensure as much product as possible was retained. The tube was placed in a bath of warm tap water. A gentle stream of air was used to evaporate the methanol.

Around 3mL of anhydrous diethyl ether was added to the centrifuge tube. The tube was gently shaken to dissolve the dried mixture in the ether. Around 3mL of water was added to the solution in the tube. The tube was gently mixed again. The solution was transferred from the tube to a 30mL separatory funnel. More ether and water was used to rinse the tube to ensure as much product as possible was retained. The claw on the stand was switch for a small ring stand to hold the separatory funnel. The separatory funnel was gently inverted, burped, and the bottom water layer was drained off and saved in a 30mL beaker. The ether layer was drained into a separate 25mL flask. The water was added back into the separatory funnel and around 2 more mL of ether was added. The funnel was inverted to mix. The water layer was drained back into the 30mL beaker and the ether layer was added to the 25mL flask. This process was repeated once more to ensure as much of the product as possible was captured in the ether solution.

The ether was dried using anhydrous sodium sulfate. While the solution was drying an empty 25mL flask was weighed, 23.729 g. The dried solution was transferred to the weighed flask, using more ether to wash. The beaker was placed in a bath of warm tap water and the ether was evaporated with a gentle stream of air. The flask was reweighed and the mass of the product was calculated by subtracting the final mass from the initial.

Percent yield was calculated by divided the mass of the product by the theoretical. An IR spectra was run and analyzed. An NMR tube was prepared by adding 3 drops of the product to 650 microliters of $CDCl_3$. The H NMR spectra was run and analyzed.

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Apparatus

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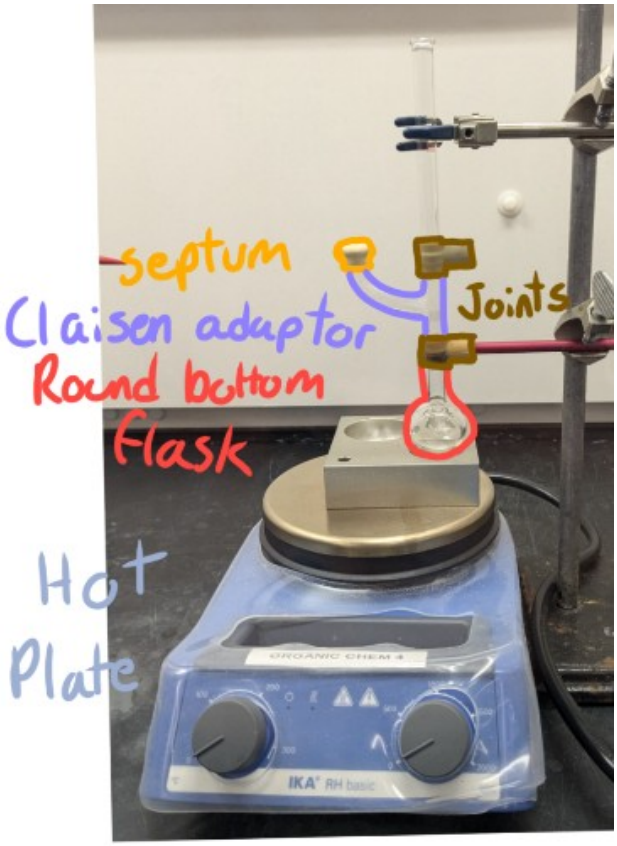
Insert drawing or photo of the synthesis set-up and **label all parts**.

reflux apparatus:



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Data & Analysis Section

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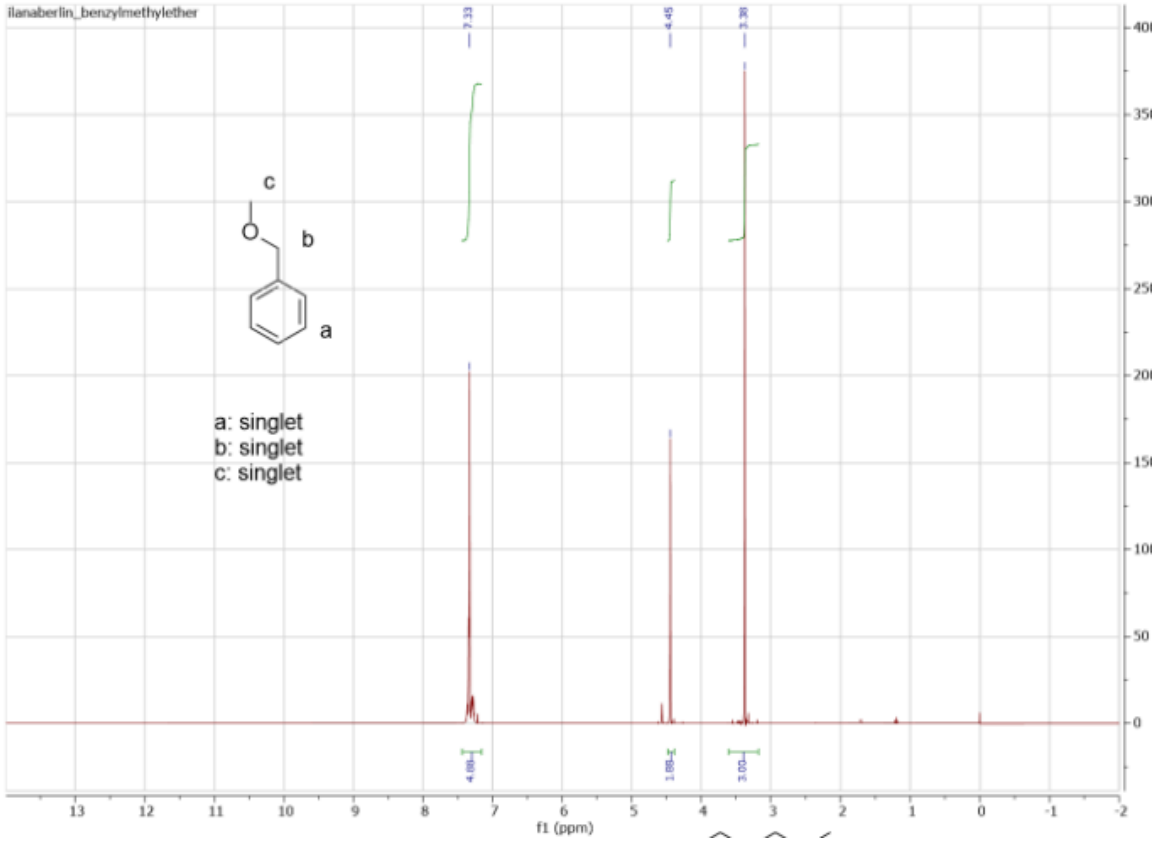
Insert all spectra and chromatograms here.

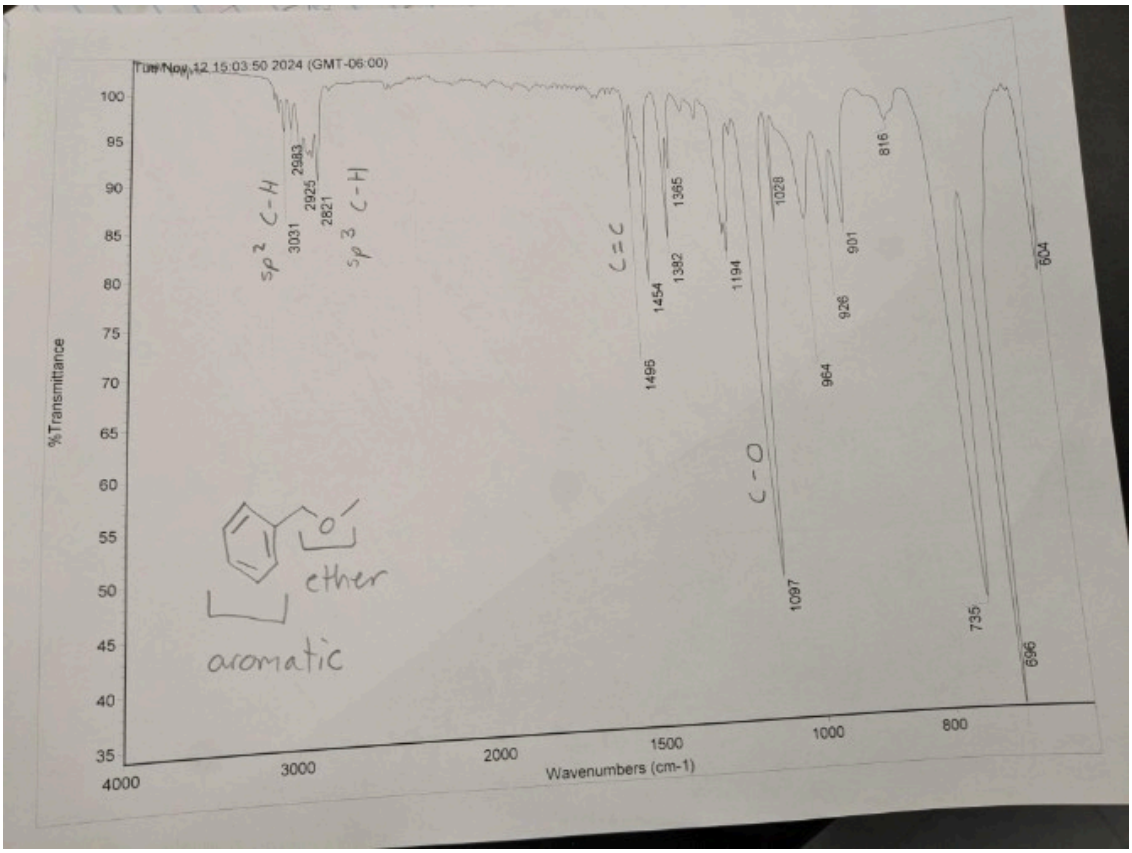
Label all data appropriately

- FTIR - label stretches for key functional groups
- NMR - label each peak (A, B, C...) and assign each peak on a drawing of the compound
- GC - label key peaks and record retention times and percent areas

Include a brief analysis for each piece of collected data.



The NMR supports the proposed spectra from the pre-lab.



The ether shift is in the fingerprint region of the IR spectra.

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labels for NMR signals?

Write a comment...

@

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Conclusions

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In the Sn2 reaction the chlorine in the benzyl chlorate acted as a leaving group while the methyl oxide of sodium methoxide acted as a nucleophile. Methanol, a polar protic solvent weas used to reduce the nucleophilcity of the methyl oxide since it is such a strong nucleophile, usually Sn2 reactions are favored in polar aprotic solvents.

The loss of product was minimized by washing containers with solvents whenever the solution was transferred, even so there may have been residue left behind. The tip of the separatory funnel I used was broken which lead to a bit of spillage of the water during the separation process, product remaining in the water may have been lost.

Evidence of a reaction happening includes the foaming of the solution, the temperature change of the round bottom flask, and IR and NMR spectra supporting that the product is different from the reactant.

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