CHEM 2212L Experiment 8 - The Wittig Reaction Lab Report

Graded

Student

Siddiqui Zohaib M.

Total Points

92 / 94 pts

Question 1

Title/Introduction

10 / 10 pts

✔ **– 0 pts** Correct

Question 2

Balanced Equation

6 / 6 pts

✔ **– 0 pts** Correct

Question 3

Reaction Mechanism

6 / 6 pts

✔ **– 0 pts** Correct

Question 4

Table of Reagents

5 / 5 pts

✔ **– 0 pts** Correct

Question 5

Safety Information

5 / 5 pts

✔ **– 0 pts** Correct

Question 6

Experimental Procedure

6 / 6 pts

✔ **– 0 pts** Correct

Question 7

Data and Observations

10 / 10 pts

✔ **– 0 pts** Correct

Question 8

Results

10 / 10 pts

✔ **– 0 pts** Correct

Question 9

Discussion and Conclusions

12 / 12 pts

✔ **– 0 pts** Correct

Question 10

Post Lab Questions

22 / 24 pts

10.1 **Question 1**

6 / 6 pts

✔ **– 0 pts** Correct.

10.2 **Question 2**

10 / 10 pts

✔ **– 0 pts** Correct

10.3 **Question 3**

2 / 4 pts

✔ **– 2 pts** Incorrect 2nd product

10.4 **Question 4**

4 / 4 pts

✔ **– 0 pts** Correct

Questions assigned to the following page: 1 and 2

Zohaib Siddiqui

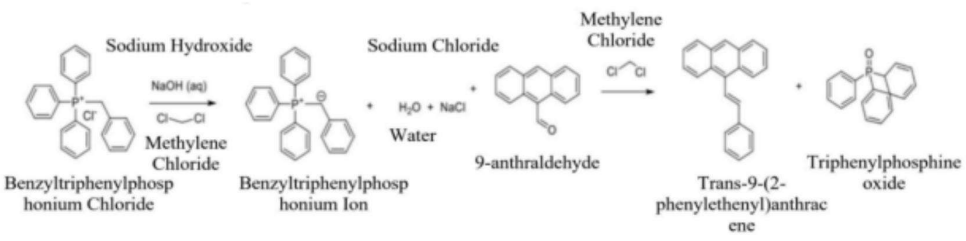
Zms05515

Title: Wittig Reaction

Introduction:

This lab requires an aldehyde and the conversion of it into an alkene using the Wittig reaction. The Wittig reaction formulates a double bond in between an electrophilic carbonyl and a nucleophilic phosphorus ylide that is neutral and a dipolar molecule with positive and negative charges. The ylide devised in the experiment is benzyltriphenylphosphonium chloride. Sodium hydroxide will be used to deprotonate the ylide, and then the reaction will occur when separating the organic and aqueous layers. The use of liquid-liquid extraction will be used to separate the organic and aqueous layers, and decanting will get rid of the calcium chloride from the organic layer. The use of simple distillation will remove the solvent from the product mixture. Then, recrystallization will help form the product crystals starting from the liquid form, and suction filtration will be used to dry and purify them. IR spectroscopy will be utilized in order to confirm if the reaction was complete in order to find the correct product along with the correct functional groups.

Balanced Equation:



Reaction Mechanism:

Questions assigned to the following page: 3 and 4

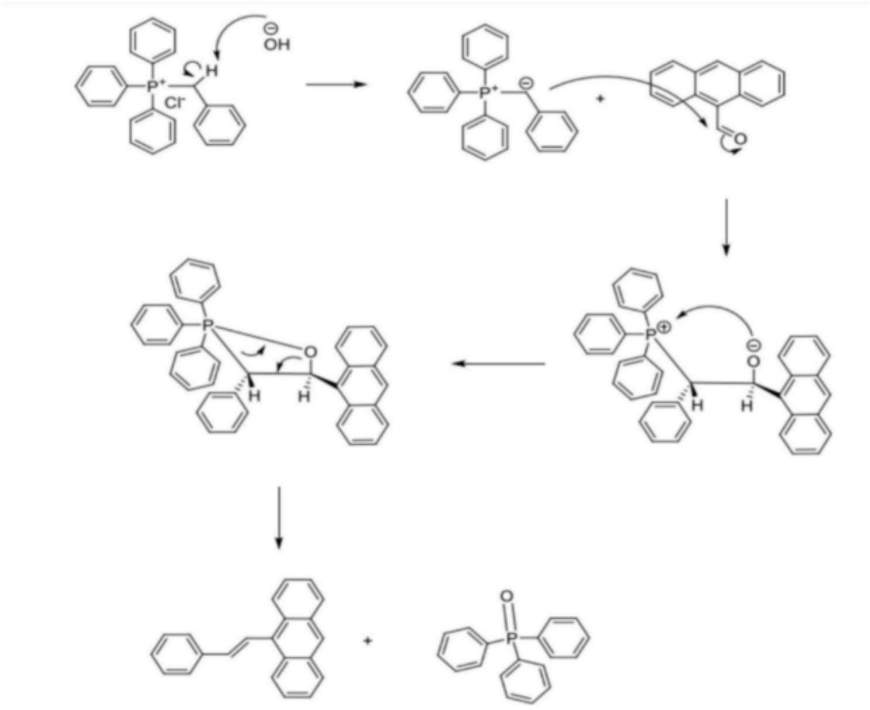
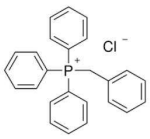
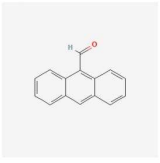
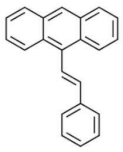
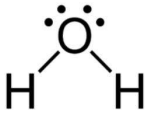
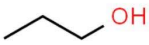
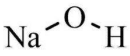
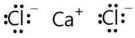


Table of Reagents:

Compound	Structure	Molecular Weight (g/mol)	Boiling/Melting Point	Density (g/mL)
Benzyltriphenylphosphonium chloride		388.87	314	
9-Anthraldehyde		206.24	103-107	

Questions assigned to the following page: 4 and 5

Trans-9-(2-phenylethenyl)anthracene		280.73	130-133	
Methylene chloride		84.93	39	1.33
Water		18.02	100	1.00
1-Propanol		60.10	97	.803
Sodium hydroxide		40	318	
Calcium chloride		110.98	176	

Safety Information:

- Always wear safety goggles, lab gloves, and lab coats at all times. Be cautious with glassware and other volatile liquids.
- Any exposure to vapor or liquid must be informed to TA immediately.
- Never seal a reflux apparatus airtight.
- Benzyltriphenylphosphonium chloride and 9-Anthraldehyde can cause eye irritation and can be fatal if swallowed.
- Trans-9-(2-phenylethenyl)anthracene should be avoided by the skin and eyes
- Water should be normal, can be hazardous if boiled

Questions assigned to the following page: 5 and 6

- 1-propanol can cause eye irritation and dizziness.
- Sodium hydroxide can cause severe burns with contact to eyes and skin
- Calcium chloride can cause serious eye damage

Experimental Procedure:

1. Add 1.0 of benzyltriphenylphosphonium chloride, 0.590 g of 9-anthraldehyde, and a spin vane to a clean, 10 ml round bottom flask. Dissolve the starting materials in 3.5 ml of methylene chloride and begin to slowly stir the reaction mixture using the spin vane.
2. Over a three minute period, slowly add 1.3 ml of 50% sodium hydroxide solution to the reaction flask in a dropwise fashion.
3. Once the hydroxide solution has been added allow the flask to stir at room temp for an additional 30 minutes.
4. Remove the spin vane and carefully pour the contents of the flask into a separatory funnel. Rinse the round bottom reaction flask once with 5 ml of methylene chloride and once with 5 ml of DI water. Pour reach rinsing into a separatory funnel, and allow the layers to separate once again.
5. Drain the organic layer into a clean, dry 25 ml Erlenmeyer flask. Extract the remaining aqueous layer with a fresh 10 ml portion of methylene chloride. Drain and collect the methylene chloride extract in the 25 ml Erlenmeyer flask.
6. Drain the aqueous solution out of the separatory funnel and save it until the end of the experiment as a precautionary measure. Add calcium chloride pellets to the organic extract in order to remove any residual water.
7. Decant it into a clean, dry 50 mL round bottom flask. Construct a simple distillation apparatus and use it to separate the methylene chloride from the crude product.

Questions assigned to the following page: 6 and 7

8. Remove the heating mantle and allow the apparatus to cool to room temperature once the product forms a thick yellow slurry in the bottom of the flask.
9. Recrystallize the crude product with a minimal amount of 1-propanol. Use an ice water bath to aid in the final precipitation of the crystals. Pull air through the filtration apparatus for 5 minutes, after the crystals have been collected.
10. Transfer the final product onto a watch glass and place the crystals in a drying oven for 5 minutes. Determine final mass of product and melting point range.

Data:

Initial Weight of benzyltriphenylphosphonium chloride used: 1.02 g

Initial Weight of 9-anthraldehyde used: 0.593 g

Volume of methylene chloride used to dissolve the starting materials: 15 ml

Volume of aqueous sodium hydroxide used: 1.35 ml

Volume of methylene chloride used to rinse the reaction flask: 5.05 ml

Volume of water used to rinse the reaction flask: 5.15 ml

Volume of methylene chloride used to extract the reaction mixture: 9.91 ml

Approximate amount of calcium chloride used to dry the organic extract: 0.969 g

Final weight of the purified product: 1.741 g

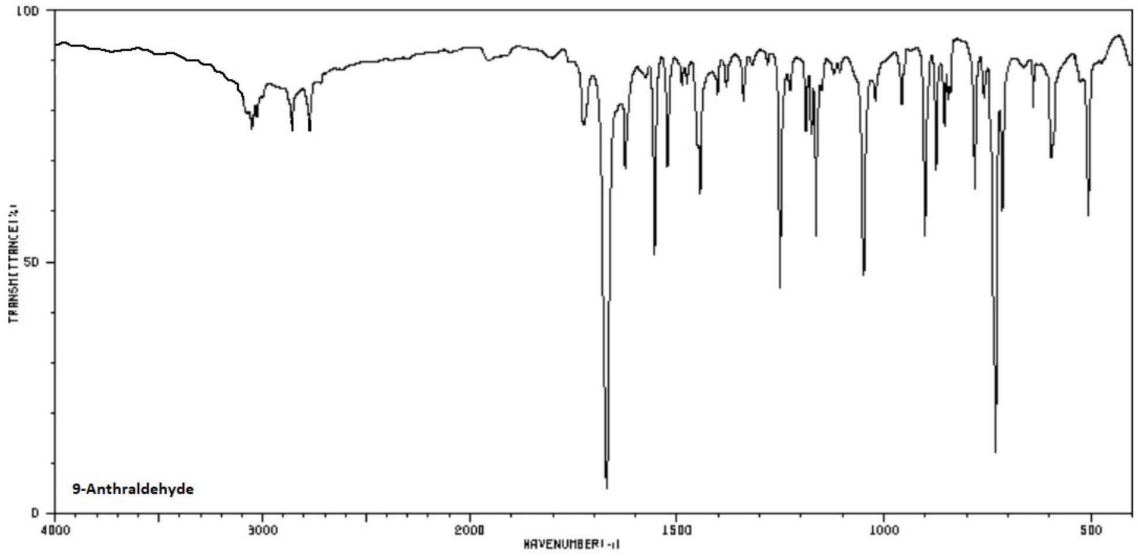
Observations:

- Solution has stayed yellow in the mixing step
- Yellow color has gotten deeper throughout stirring time
- Small droplets not mixed into two separate phases
- Organic layer was clear yellow, and the aqueous was cloudy white
- Some solution lost during extraction due to dripping

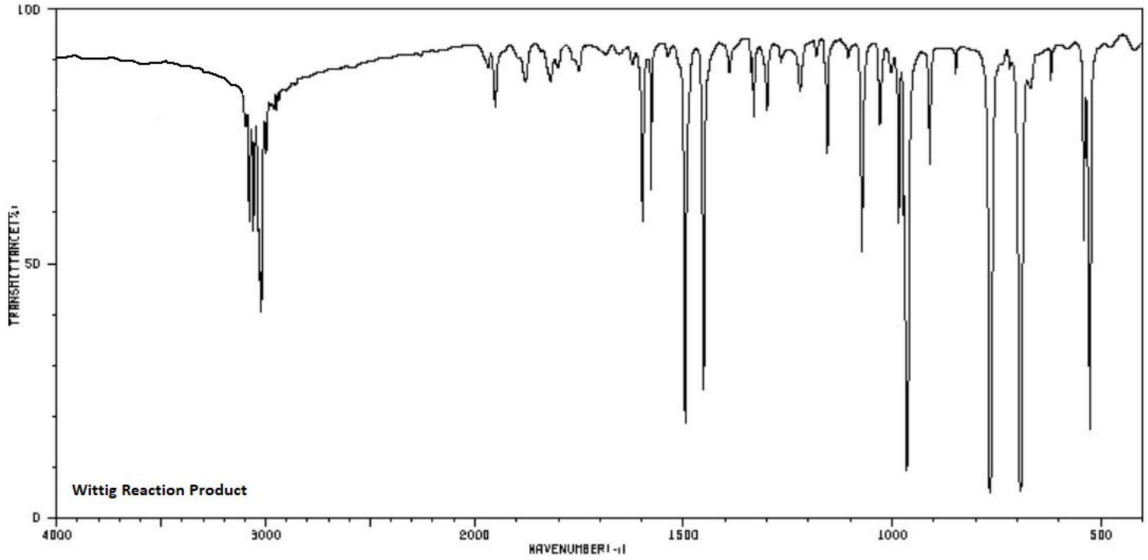
Question assigned to the following page: 8

Results:

9-Anthraldehyde IR Spectrum:



Wittig Product IR Spectrum:



Limiting reagent calculations:

Questions assigned to the following page: 8 and 9

Mass of Benzyltriphenylphosphonium chloride to grams of Trans-9-(2-phenylethenyl) anthracene product:

1.012 g * (1 mol/ 388.87 g) * (1 mol Trans-9-(2-phenylethenyl) anthracene / 1 mol Benzyltriphenylphosphonium chloride) * (280.73 g / 1 mol) = 0.731 g of Trans-9-(2-phenylethenyl) anthracene

Mass of 9-Anthraldehyde to grams of Trans-9-(2-phenylethenyl) anthracene product:

0.593 g * (1 mol/ 206.24 g) * (1 mol Trans-9-(2-phenylethenyl) anthracene/ 1 mol 9-Anthraldehyde) * (280.73 g/ 1 mol) = .807 g of Trans-9-(2-phenylethenyl) anthracene

Volume of Sodium Hydroxide to grams of Trans-9-(2-phenylethenyl) anthracene product:

1.35 ml * (2.13 g/ 1 ml) * (1 mol/ 40 g) * (½) * (1 mol Trans-9-(2-phenylethenyl) anthracene/ 1 mol NaOH) * (280.73 g/ 1 mol) = 10.09 g of Trans-9-(2-phenylethenyl) anthracene

Benzyltriphenylphosphonium chloride is the limiting reagent.

Percent Yield: (actual/theoretical)= 1.741/.731 x 100 = 238.2%

Discussion/Conclusion:

The experiment consisted of making an alkene product using the Wittig reaction.The completed final product was then put in use in order to get rid of any impurities. IR spectroscopy was used to confirm that the product alkene had successfully produced the correct product by comparing the 9-Anthraldehyde IR spectrum to Trans-9-(2-phenylethenyl) anthracene IR spectrum. The IR spectrum of the product material shows peaks around 3000-3100 cm-1 which means sp2 hybridized carbons of an alkene were present. There was also a peak around 1000 cm-1 showing

Question assigned to the following page: 10.1

the C=C double bonds of an alkene. There are aromatic overtones around 1750-1900 cm-1 which also indicates a benzene interaction. The IR of the starting material has almost the same peaks, but a C=O double bond stretch is present near the 1600 cm-1 which isn't present in the product IR. This indiciated correct following of the Wittig reaction since the product IR has no double bonds, meaning that 9-Anthraldehyde did not stay in the product. The percent yield obtained in the experiment was only 238.2% , which is very high and more than expected. This could be due to a multitude of experimental factors. To begin with, the refluxed reaction may not have been fully mixed due to moving of the beaker and changing of the hot plate since the heat was accidentally on the first go round. Another reason the yield might have been higher was due to a slight excess in starting materials since slightly more was added than what the procedure had told us to. Along with this, the use of the separatory funnel may not have fully extracted and separated the aqueous and organic layers which could have potentially led to us getting more of the organic to the final product, and after decanting more product was also left over.

Post Lab Questions:

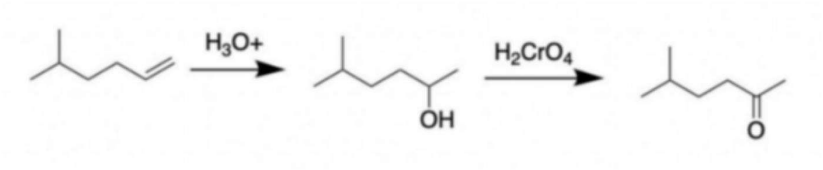
1. (4.15 g (S)-(4-ethyl-2-methylhexane) triphenylphosphonium) * (1 mol/ 389 g) * (100/81) * (1 mol (S)- 1-chloro-4-ethyl-2-methylhexane/ 1 mol (S)-(4-ethyl-2-methylhexane) triphenylphosphonium) * (162.7 g/1 mol) = **2.12 g of (S)-1-4-ethyl-2-methylhexane are required to produce 4.15 g of (S)-(4-ethyl-2-methylhexane) triphenylphosphonium at a 81% yield.**

   (4.15 g (S)-(4-ethyl-2-methylhexane) triphenylphosphonium) * (1 mol/ 389 g) * (100/81)*(1 mol triphenylphosphine / 1 mol (S)-(4-ethyl-2-methylhexane) triphenylphosphonium)*(262.29 g/ 1 mol) = **3.41 grams of triphenylphosphine are**

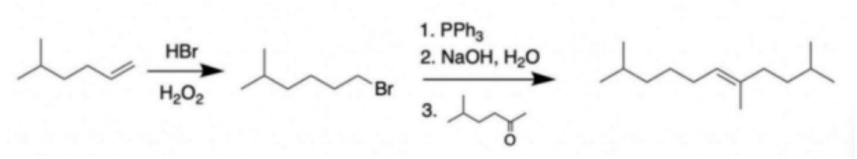
Questions assigned to the following page: 10.2, 10.3, and 10.4

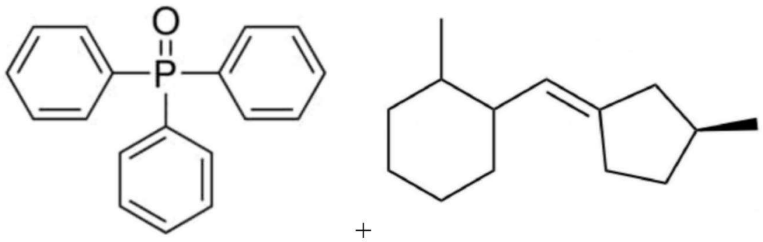
required to produce 4.15 g of (S)-(4-ethyl-2-methylhexane) triphenylphosphonium at a 81% yield.

2. Part 1:



Part 2:



3. 

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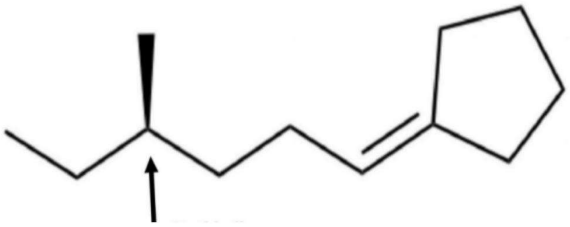
4. 
Z Isomerism and R Chiral Center