Experiment 4. Reduction of Benzyl

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Section: C02
Date conducted: March 6, 2018
Date Due: March 13, 2018

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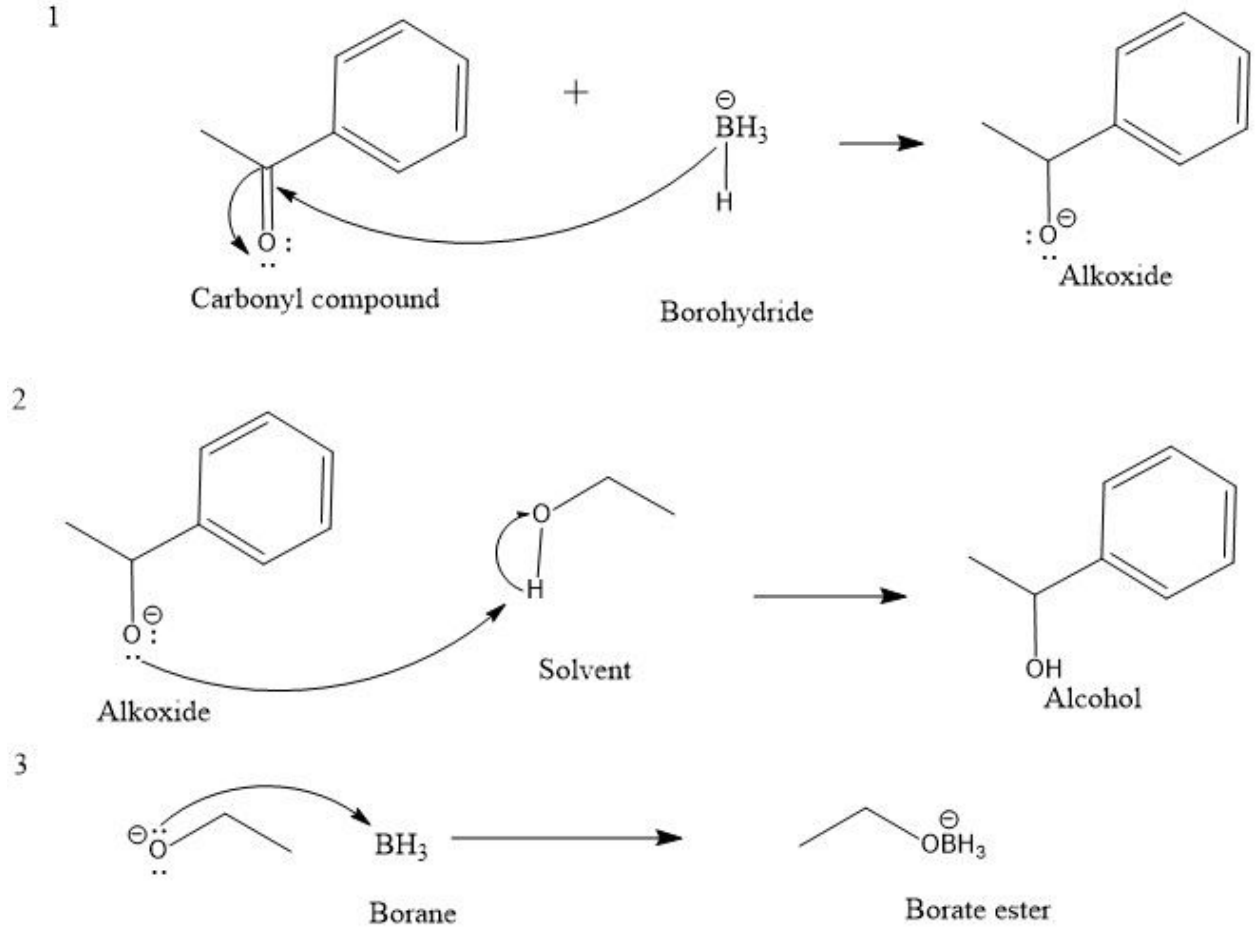
Introduction

Reaction mechanism of the reduction of $NaBH_4$



Procedure

Refer to introductory to Organic Chemistry Laboratory Manual 2017. By Prof. William Ogilvie and Prof.Tony Durst. Experiment 4: Stereochemical Analysis of the reduction of Benzyl. Page 34- 41

Observations

Part A:

1g of benzil with 10 mL of Ethanol

- When solution was on stir plate and in the ice bath it was slowly dissolving
- Color of benzil is yellow which turned solution to be yellow
- Color yellow did not disappear\

When 0.1g of sodium borohydride was added and in ice bath

- Benzil still hasn't dissolved

- Solution was less yellow

When another 0.1g of sodium borohydride added

- Solution looks more cloudy
- Benzil still not dissolved

When another 0.1g of sodium borohydride added

- Solution was even more cloudy
- Benzil was starting to dissolve more
- Yellow colour was slowly disappearing by 2 min

In 10 min after

- Completely dissolved
- Solution is very light yellow

Greater than 10 min

- Cloudy white yellow colour
- Looks white like milk

TLC plates

- Reference point is benzyl dissolved in dichloromethane
- The co spot is the dissolved benzl and the reaction mixture
- On the first TLC plate the reaction mixture had a bit of benzyl which showed that the reaction was complete
- The second TLC plate showed that the reaction was complete

When 10 mL of hot water added and stirred

- Solution was pale yellow

When 20 mL of hot water added to reaction

- Solution was colourless
- Bubbles appeared

Suction filtration

- Really big crystals were formed

Part B:

- 0.6 g of diol was a light yellow powder
- CH_2Cl_2 was a clear liquid and had a fragrance smell to it
- When adding p-toluenesulfonic acid the solution turned from a light green to a light orange then to a dark orange.
- When waiting longer the solution is supposed to look dark brown.

Tables of reagent

Table 1. Table of reagents for the reduction with $NaBH_4$

Compound	Molecular weight (g/mol)	Amount (g or ml)	Density (g/ml)	Moles (mol)
$NaBH_4$	37.83	0.3g	-	0.007930
Benzil	210.23	1.0	-	0.004756
Ethanol	46.07	10 mL	0.789	0.1713

Table 2. Table of reagents for the acetalization

Compound	Molecular weight (g/mol)	Amount	Density (g/mL)	Moles (mol)
Diol product	-	0.6g	-	-
CH_2Cl_2	84.93	25 mL	1.33	0.3915
Sodium Sulfate	142.04	0.5g		0.00352
2-methoxypropene	72.11	1.0 mL	0.753	0.0104
P-toluenesulfonic acid	172.2	0.05 g	-	0.0029

TLC Plates

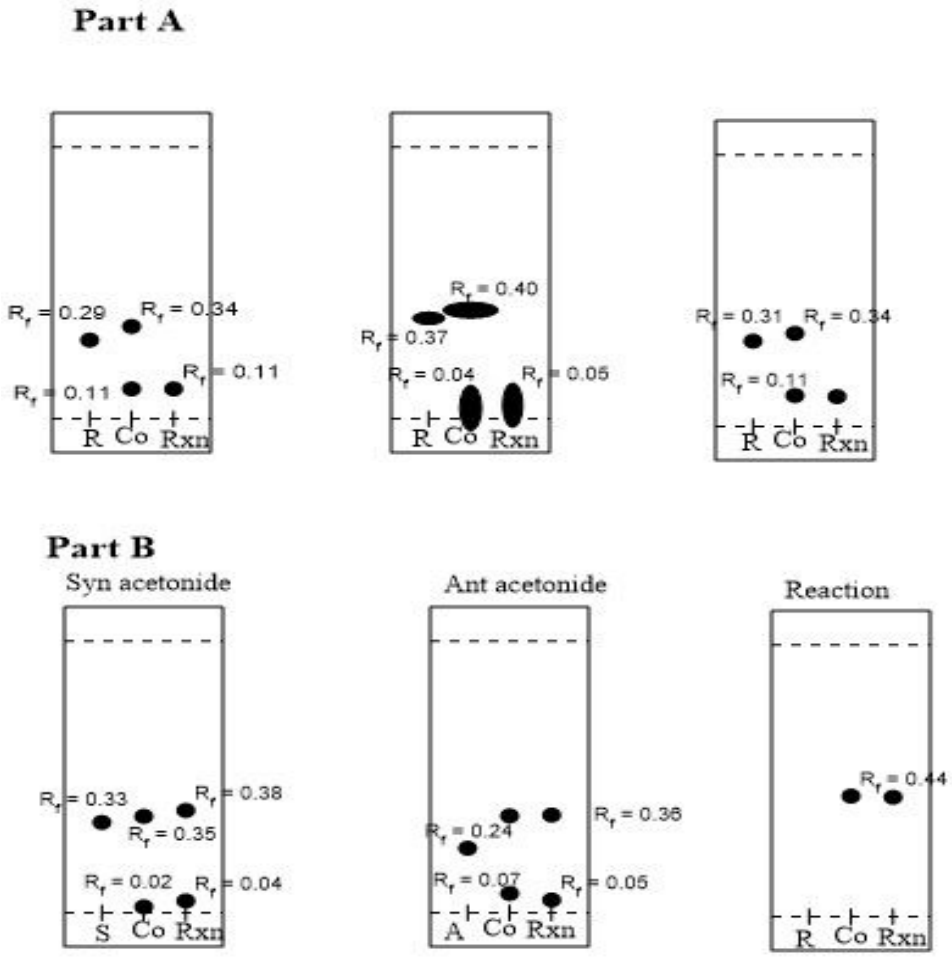


Table of results

Table 3. Table of results

Product	Amount(g)	Molecular Weight(g/mol)	Moles(mol)	Percent Yield
Hydrobenzoin	0.69	212.26	0.22	67.699%

Calculations

Number of mol of benzil= m/M

m= 1.0 g

M= 210.232 g/mol

=1g ÷ 210.232 g/mol = 4.757 X 10^{-3}

Number of mol of hydrobenzoin= m/M

= 0.69g ÷ 212.26 g/mol = 3.220 X 10^{-3}=0.22

Percent Yield= (mol of hydrobenzoin/ mol of benzil) X 100

=(3.220 X 10^{-3} ÷ 4.757 X 10^{-3}) X 100

=67.699%

Discussion

Part A:

- The purpose of this lab was to determine the percent yield through calculations of a benzil product with the use of recrystallization and suction filtration as well as the reduction of sodium borohydride
- Reduction is when a compound has gained electrons and oxidation would be defined as the loss of electrons.
- In order for this to happen, sodium borohydride has to act as a reducing agent to benzil in a protic solvent.
- In this experiment, the benzil was not dissolving at first. This is due to benzil being non polar and ethanol being polar. Thus like dissolves like, and nonpolar molecules are not miscible with polar ones.
- After adding 0.3 g of sodium borohydride, the benzil started to dissolve and the colour started to fade away. This is due to sodium borohydride acting as a reducing agent.

Sodium borohydride can be used as a reducing agent if alcohol is present. In this case ethanol is the alcohol present.

- This also helped change the color from yellow to white.
- The TLC plates helped show if the reactions were complete. The first TLC plate showed that there was still benzil in the reaction mixture. Therefore the reaction was not complete.
- After this the solution was stirred more and another TLC plate was made. In this TLC plate the benzil was not shown in the reaction. This shows that the reaction was complete. A third TLC plate was taken and the same thing was proved.
- When suction filtration was done, really big crystals were formed. This was done by recrystallization.
- Recrystallization was then done to remove impurities allowing the compound to crystalize when cooling.
- Another TLC was made to see if the final product was in the reaction mixture

Part B:

- The purpose of this experiment was to determine the major product of this reaction which could be either syn acetonide or anti acetonide
- Crystals retrieved from the suction filtration were mixed with 2-methoxypropene and P-toluenesulfonic acid
- The diol product was converted into acetonide in order to better identify the stereoisomers.

- Acetonide allowed a greater change in polarity between the two stereoisomers as it fixed oxygens in place, resulting in only one conformer, as rotations along the bond axis bonding the phenyl group and the oxygen will be limited
- This is what caused the colour change
- The first TLC showed that the syn acetonide was in the reaction mixture
- The second showed that the ant acetonide was not in the mixture
- Due to the distance travelled by both syn and anti-stereoisomer, it is also revealed that anti is less polar than syn, as it travelled a greater distance up the stationary phase.
- The percent yield was low and this can be due to the loss of substance during transfer of solution. What could've been done is carefully transferring the substance and not rush. This would make it more efficient and less product would have been loss.

Questions

1.

- The experimenter is given 3.5 g of A and 10 g of B

The minimum volume of solvent needed to dissolve both is given by the solubility of compound A& B: B= 10g(100ml/16g) = 62.5ml

The solution is cooled to 20 degrees celsius and crystals are collected

- At 20 degree we will have in solution:

1g of A/100ml= (x)g of A/62.5ml

(x)g of A = (1/100ml)(62.5)= 0.625 of A in solution

1g of B/ 100ml = x(g) of B/ 62.5ml

(x)g B= (1/100ml)(62.5)= 0.625g of B in solution

The sample is composed of 3.5g of A and 10g of B hence these amounts of solids A and B will be in the crystals that have been isolated.

3.5g of A - 0.625g A in solution = 2.875g of solid A

0 g of B - 0.625g of B in solution= 9.375g of solid B

Calculating crystal composition

- Total amount of solid isolated (A+B): (2.875g of A + 9.375g of B)= 12.25g of solid

%A= (2.875/12.25)(100)= 23.47%

%B= (9.375/12.25)(100)

= 76.53%

Calculating the yield % process

- 12.25 g of solid was isolated

%yield= ((12.25g of solid)/ (3.5g of A + 10g of B))(100)

= 90.741% yield

Composition of the mother liquor

- The mother liquor contains 0.625g of A and 0.625g of B

2.

There are 2.875g of A and 9.375g of B dissolved in 100mL of solvent

1g of A/ 100 = (X)g of A/100= 1g

1g of B/100= (x)g of A/100= 1g

2.875g of A - 1g = 1.875

9.375g of B - 1g = 8.375g

Percent Yield= ((1.875g+8.375g)/ (12.25))(100)

= 83.67%

3. Explanation of the students poor yield

- Student needed to start at a higher temperature instead of starting at 25 degrees. This will allow recrystallization to occur since high temperatures increases solutes solubility in solvents.
- In order for crystallization to occur, polarities have to be close or similar in order to be miscible(like dissolves like). Using methanol which is a polar solvent instead of a non polar solvent would cause crystallization to be less effective and have a low interaction.

4.

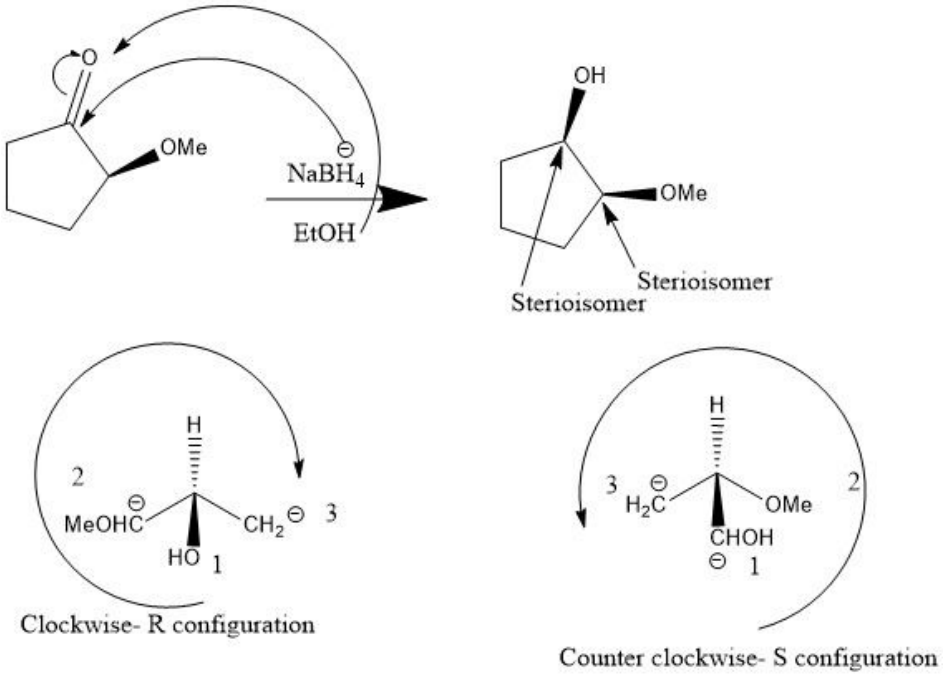
a)

- Since butanoic acid is a carboxylic acid and NaBH4 is not strong enough to reduce it, 1-butanol is not obtained. This is also due to the carbonyl carbon on the carboxylic acid being electrophilic.
- there is also acidic proton from the carboxylic acid that can react with hydride reagents. This is the cause for which sodium borohydride does not reduce a carboxylic acid. A stronger reducing agent should be used.

b)

- The product of this reaction will be hydrogen gas as carboxylic acid will undergo a deprotonation process.

5.



- For configurations, atoms were numbers from highest priority to lowest. This helps identify whether the stereoisomer has an R or S nomenclature.
- OH stereocenter can be anti or syn to the OMe
- Since Sodium Borohydride only reacted with the double bonded oxygen, the configuration for OMe stereocenter stays the same

References

Organic Chemistry Laboratory Manual 2017. By Prof. William Ogilvie and Prof.Tony Durst.

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Experiment 4. Stereo Chemical Analysis

Part A.

Observations

1g Benzil, w/ 10mL ethanol:

- When on stir plate and in ice bath it is slowly dissolving
- Color of Benzil is yellow; solution is yellow
- color yellow did not dissapear, did not dissolve

when 0.1 g of sodium borohydride w/ice bath

- solution isn't yellow anymore, Benzil still yellow, little pieces, not dissolved

When 0.2 g added w/ice bath

- looks more cloudy, Benzil still not dissolved

When 0.3 g added w/ ice bath

- even more cloudy
- benzil seamed to dissolve a little more
- yellow color slowly dissapearing
- by 2 minutes

In 10 minutes in ice bath

- completly dissolved, yellow color still there

10 min out of ice bath

- Color still yellow, light clear

>10 min

- cloudy white yellow, tinted white. like milk

TLC Part A

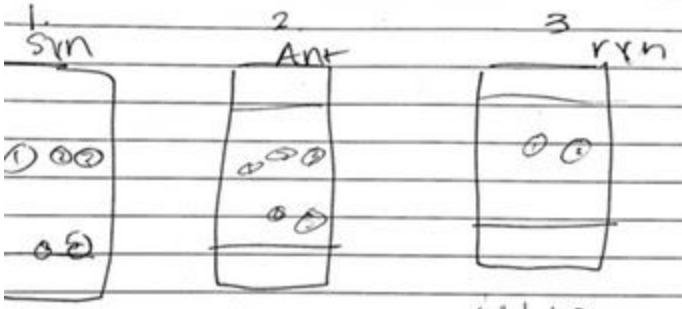
3rd

Td: 3.5 cm

reference: 1.1 cm SM: 1.2 cm Rxn 0.4 cm

0.4 cm

TLC Part B:



~~Total = 4.5~~

1 = 1.5

2 = 1.6

3 = 1.7

4 = 0.1

5 = 0.2

~~Total = 4.2~~

1 = 1cm

2 = 1.5cm

3 = 1.8cm

4 = 0.3cm

5 = 0.2cm

Total = 4.5

1 = 2cm

2 = 2cm.

TLC Plates
- Refence is benzyl dissaved in dichloro methane
- co spot is ↑ and reaction mixture
- 3rd is rxn mixture
- 1st TLC plate the rxn mixture had a little bit of benzyl
- 2nd rxn complete

when 10ml not water added & stirred
- pale yellow.

when 20ml HT added to rxn
- colorless
- bubbles

suction filtration
- crystals are formed really big
- solution slowly dripping

watch glass: 46.19 g
have a lot of product
final mass: 46.88 g
with crystals/product.
mass product: 0.69 g
Benzyl Et
yield. in moles

Moles = $\frac{m}{}$

sodium borohydride
Catalyst.