Experiment 4 – Stereochemical Analysis of the Reduction of Benzyl

Student Name:

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Chemistry 1321

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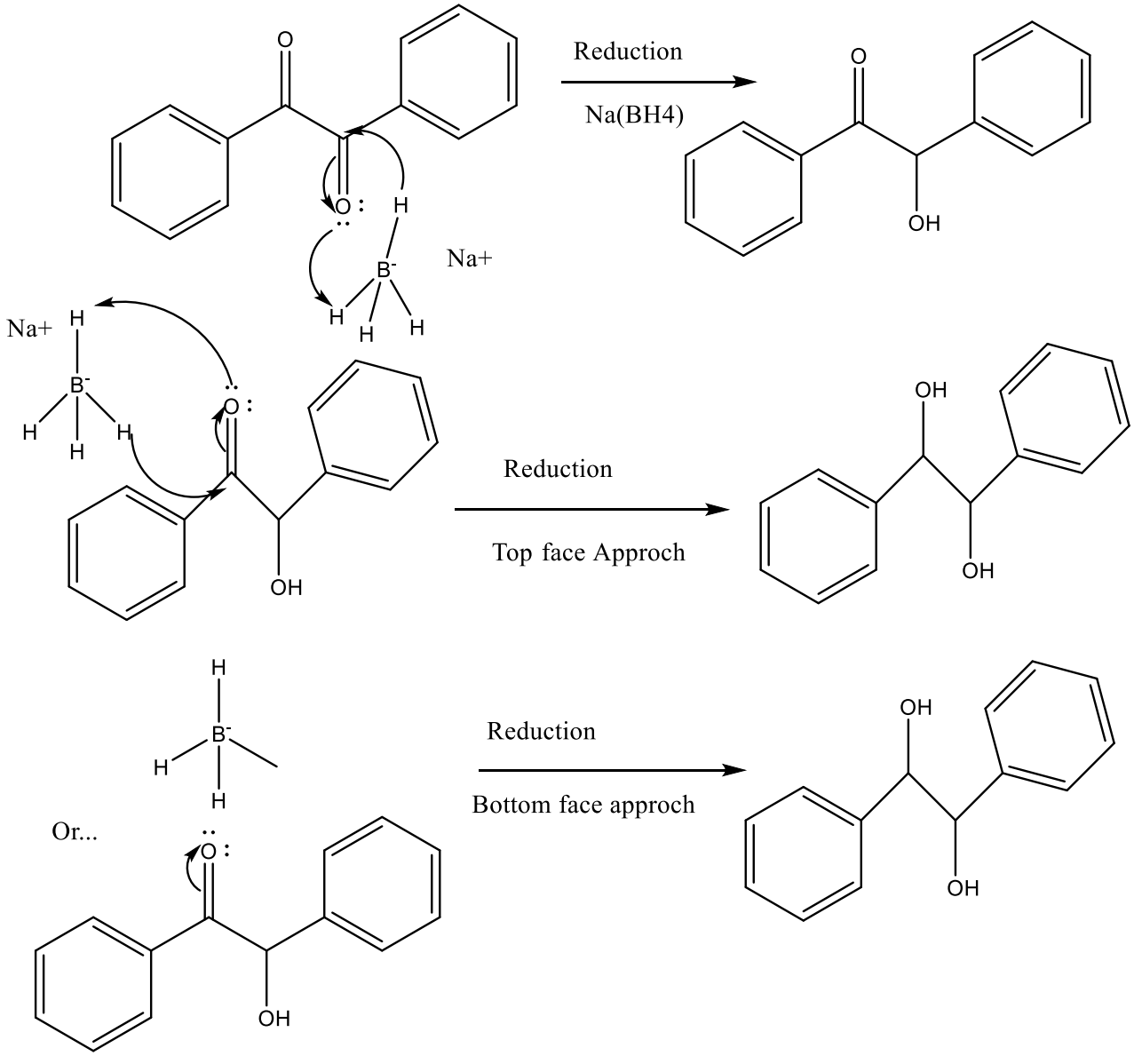
Experiment 4 – Stereochemical Analysis of the Reduction of Benzyl

Procedure: Reference to lab manual

Materials: Reference to lab manual

Safety Precautions: Reference to lab Manual

Observation:



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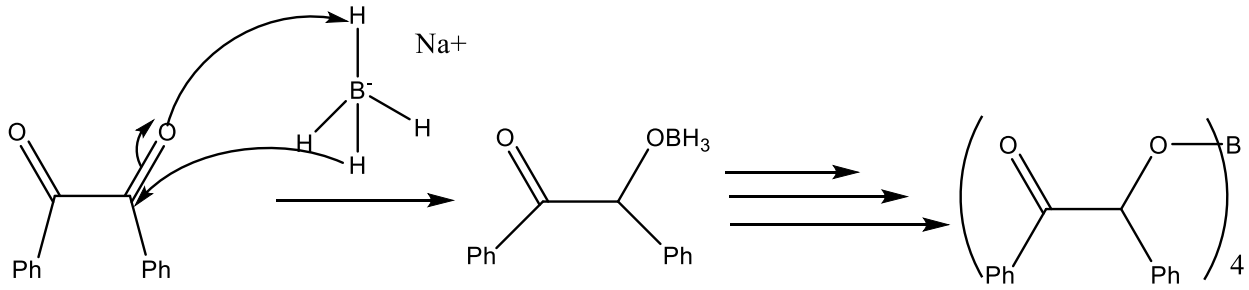
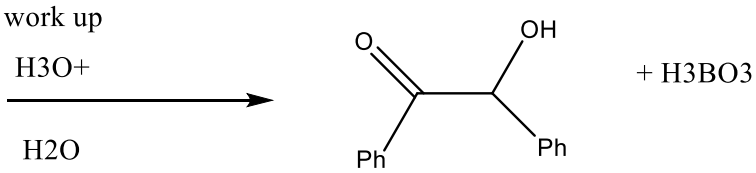
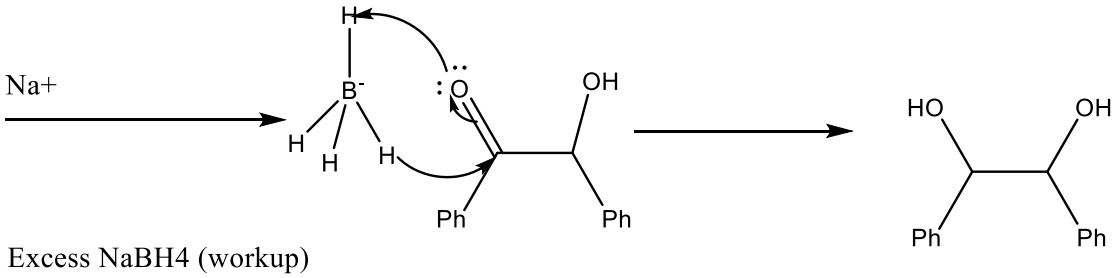
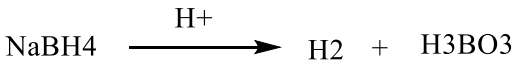
Whole mechanism: Work up included



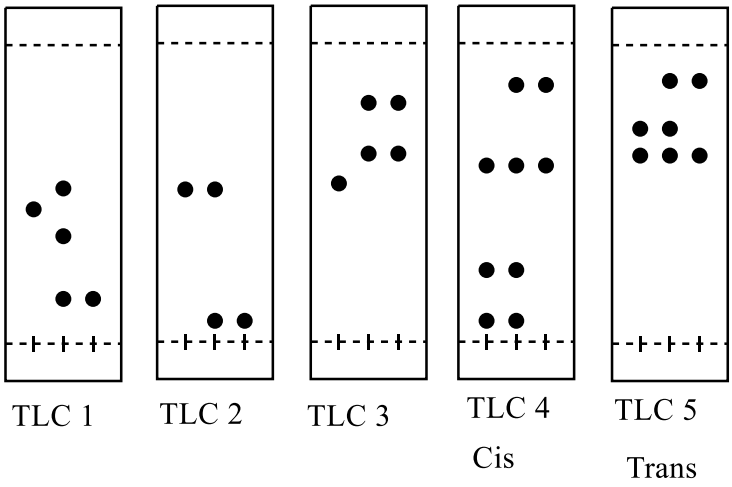




Excess NaBH4 (workup)



TLC Plates:



In this case there was some possible contamination with the Cis and Trans TLC plates, it is very hard to determine which one it could be. It is believed that Trans was obtained since there is more of an abundance of dots.

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Table of Reagents:

Reagent	Molecular Weight ($g*mol^{-1}$)	Amount (g)	Density (g/cm^3)	Moles (mol)
Benzyl	210.2	0.97	1.23	0.0046
$NaBH_4$	37.8	0.31	1.07	0.0082
p-toulenesulfonic acid	172.2	0.05	1.24	0.0003
2-methoxypropene	72.11	1.0	0.753	0.0139

Observations during the experiment:

- The yellow and clear fluid that mix for approximately 10 minutes heating to room temperature after ice bath changed colour. The compound became opaque and milky white, including some precipitate.
- Possible source of error; too much water was added at once, 50-60ml of water was added to the reaction mixture.
- After allowing reaction mixture to cool after heating it formed white precipitates with a transparent solution.
- The mass of the solid product obtained was 2.12g

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Questions:

Given 3.5 g of A, and 10g of B

B= $10g\,\frac{100ml}{16g}$

B= 62.5ml

A/B=3.5g at 20°C

at 20°C A/B = 62.5ml($\frac{1g}{100ml}$)

A/B composition= 0.625g is left in the solution.

A's composition: 3.5g – 0.625g

A= 2.875g

B= 10g- 0.625g

B=9.375g

Composition B% = ($\frac{9.375}{9.375+2.875}$) $*$ 100

Composition B% = 76.5%

Composition A%= 100%-76.5%

Composition A%= 23.5%

Yield = $\frac{Final\ mass\ A+B}{Initial\ mass\ A+B}$

Yield = ($\frac{2.875+9.375}{3.5+10}$)

Yield= 90%

Therefore you will obtain a final product of 12.25g of A and B and a percent yield of 90%

2.) 2.875g of A, 9.375 of B dissolved in 100ml

A: 2.875g-1g =1.875g

B: 9.375g-1g= 8.375g

Amount of crystals made= 1.875+8.375

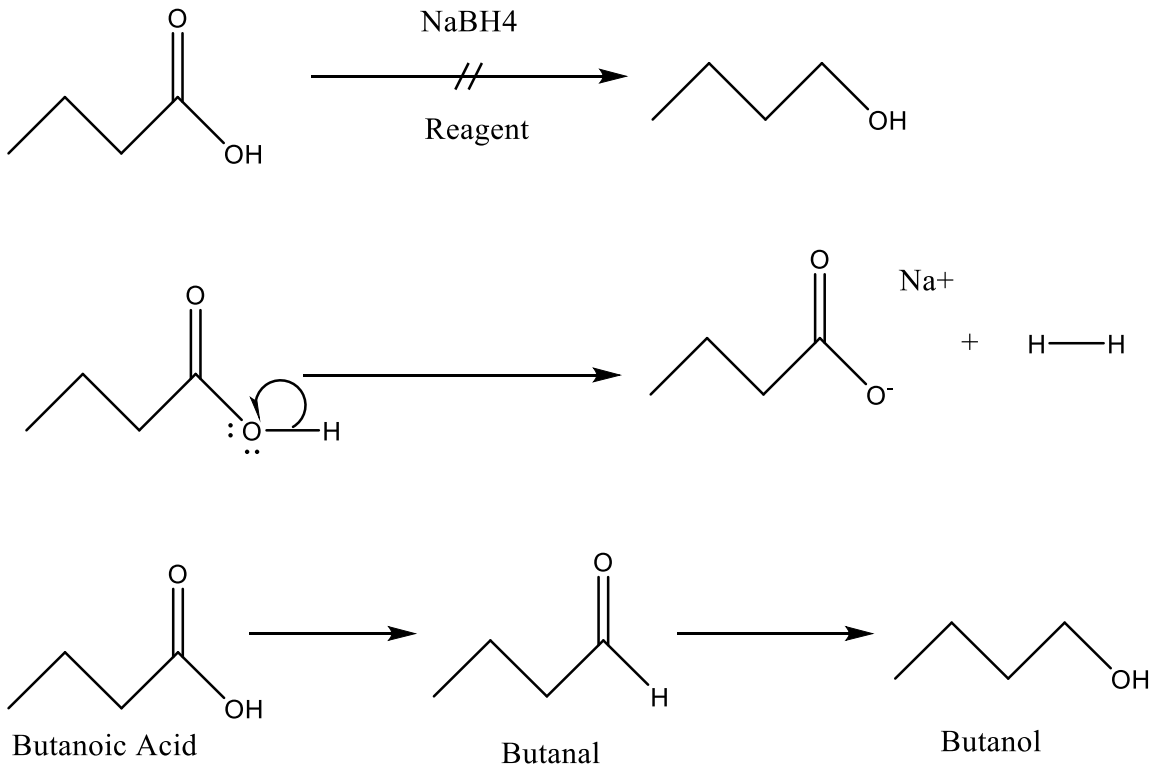
Amount of crystals made=10.24g

Yield Percent= (10.24g/12.25g)*100

Yield Percent = 83.6%

3.) During recrystallization if you induce a hot liquid you won't require much solvent. This will produce more product precipitating out thus reducing product yield. In order to improve this process; cool the product and this will increase the amount of product yield.

4.)



This molecule will become deprotonated, also leaving BH3. The carboxylic acid converts to an aldehyde and then converts to an alcohol. Thus this reaction is not very reactive.

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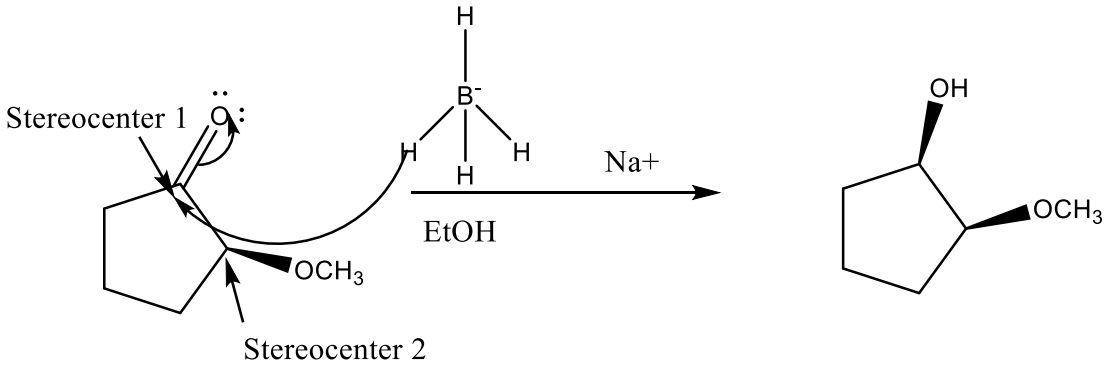
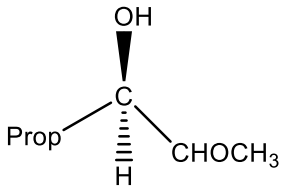
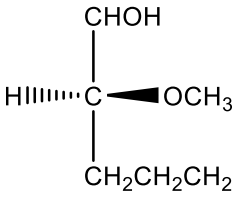
5.)





R-configuration

Stereocenter 1



S-configuration

Stereocenter 2

The large constituent group, methyl, and forces the hydrogen to attack the carbonyl using the bottom approach. Stereocenter 1 is an R-configuration, its lowest weight atom being the hydrogen and its highest the hydroxyl group.

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Discussion:

Part A:

Why do we add water to the reaction mixture? What is going on until intense bubbling stops?

Water is added in order to perform the "workup" with the weak acid. This will allow the hydrogen atom on the weak acid to attack 1 of the carbonyl groups of benzyl to form a hydroxide group instead. Therefore the water is added so we can break the opposing carbonyl group.

The intense bubbling will stop due to the release of hydrogen gas that is made by the "work up". Excess sodium borohydride will react will the H+ on the acid (H_3O) to produce hydrogen gas and boric acid. The hydrogen gas will be introduced into its surroundings, thus the intense boiling decreases.

Part B:

Do I have anti or Syn, give a reasonable hypothesis why?

It is hard to tell whether anti or syn had been obtained since the TLC plates has some contamination. Although the best guess would be syn, since it had an abundance of spots at the same Rf value.

How was it made?

- The benzyl underwent reduction using sodium borohydride, and the 1 of the carbonyl groups broke its double bond forming a single bond between the carbon and oxygen.
- Depending on the way the carbon was attacked can determine whether the molecule will become anti or syn. If carbonyl is attacked from the bottom it can produce an anti. If the carbonyl is attacked from the top (which it did) it can produce a syn molecule.
- Water and acid was added as a "workup" to separate the newly formed hydroxyl group on benzyl from boric acid. Any excess sodium borohydride reacted with the acid and produced hydrogen gas and boric acid.
- Again sodium borohydride was added to break the other carbonyl double bond between the carbon and the oxygen. The electrons on the oxygen attracted the H atom to form a hydroxyl bond.

Raw Data

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