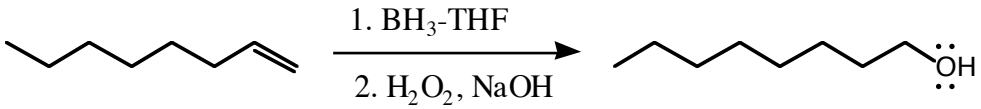
Hydroboration-Oxidation of 1-Octene

Introduction

In this reaction you will convert 1-octene to 1-octanol by using the hydroboration-oxidation reaction. This reaction is highly water sensitive and care must be taken during the first step of the reaction not to let air (which contains water vapor) into the reaction mixture.



Reagents:	volume	density	mass	MW/ molarity	mmoles	eq
1-octene	0.630 ml					1.00
BH_3-THF	1.5 ml	x	x	1 M		
3 M NaOH (aq)	1.0 ml	x	x	3 M		
30% H_2O_2 (aq)	1.0 ml	1.108 g/ml	soln: H_2O_2:			
1-octanol						1.00

Borane-THF is flammable and highly reactive with water, so care must be taken during the first step of the reaction not to let water or air (which contains water vapor) into the reaction mixture. The borane-THF solution will be dispensed with a syringe.

30% hydrogen peroxide is caustic to the skin (if you get any on you it will turn white and may blister); be careful when dispensing it (don't touch the part of your graduated pipet that was in the bottle) or when getting rid of a ruined reaction.

Please read "Water Sensitive Reactions" and review the following techniques before coming to lab: "Refluxing a reaction," "Extraction and washing," "Drying an Organic Solution," "Evaporating an Organic Solution," and "Characterizing Compounds by IR Spectroscopy." Write an introduction in your lab notebook, including the reaction table (filled out!).

Procedure

Set up and run the first step of the reaction:

- Remove your conical vial and spin vane from the oven. Connect the conical vial to a Claisen adapter with a yellow clip, capping the straight part of the adapter with a cap and septum.

- Pack a drying tube by placing a small piece of cotton close to the joint, then adding solid $CaCl_2$ pellets, and finally some more cotton to hold it in (see the white board for a drawing). Connect it to the bent part of the Claisen adapter with a yellow clip.

- Add 1-octene to the vial through the septum using the 1 ml plastic syringe and needle in the test tube connected to the bottle (estimate the volume as closely as possible).

- Cool the reaction in an ice/water bath in a small beaker on your stirrer/hot plate. After it has cooled for a minute or two, add the borane-THF dropwise with stirring over a period of 5 minutes. Put the uncapped needle in the needle waste container, and throw the empty syringe away.

- Remove the ice bath and stir at room temperature for 45 min.

Set up and run second step of the reaction:

- Remove the drying tube and Claisen adapter. Add a reflux condenser (no yellow clips).

- Carefully add two drops of water to destroy any remaining borane-THF.

- Add 1 ml of 3 M sodium hydroxide using the labeled graduated pipet. Make sure to read the labels and use the right pipet!

- Add 1 ml of 30% hydrogen peroxide to a 10 ml beaker using another labeled graduated pipet and then add it dropwise to the reaction mixture with a plastic pipet over 10 minutes.

- Reflux for 45 minutes.

Isolation of the product:

- Allow the mixture to cool; when it is near room temperature, cool it in ice.

- Add about 1 ml of diethyl ether, rinsing the spin vane as you do so.

- Transfer the lower (water) phase to a sep funnel with a pipet and extract it once with ether to remove any of the product which may be in the water layer.

- Add the original ether later from the reaction to the ether extracts in the sep funnel.

- Wash the ether solution once with 1% HCl to remove any sodium hydroxide.

- Then wash it once with distilled water to remove any trace of acid.

- Dry the ether layers over sodium sulfate.

- Tare a round bottom flask and transfer the dry ether solution to it, rinsing the sodium sulfate with a few ml of fresh solvent.

- Evaporate the solution to obtain the product.

Characterization of the product:

- The isolated product will be characterized without further purification. Observe the appearance of your product. Pure, authentic 1-octanol is a clear, colorless liquid.

- Measure the mass of the product and calculate the percent yield.

- Obtain an IR spectra of your product. Compare it to the spectrum of the alkene. Is there any alkene left? Do you see evidence that the alcohol was formed? Label any important bands and turn in a copy with your lab (print two copies, one for each student).

- Your conclusion should include the appearance of the product, the mass and % yield, and a discussion of the IR including peaks that show the desired product was formed and the starting material was not present.

Questions

1) What kind of reaction is this, substitution, addition, or elimination? Why?

2) What is the structure of the intermediate formed in the first step of this reaction?

3) BH_3 is a gas, but it can be dissolved in THF. Show how BH_3 and THF react reversibly to form a charged complex that stabilizes the BH_3.

4) Less than 1 molar equivalent of BH_3 was used in this reaction. Why isn't it the limiting reagent?

5) Why was the first step of this reaction cooled in an ice bath, while the second step was heated at reflux?

6) What reagent in this reaction would be destroyed by water? What precautions did we take to prevent this? Why were they no longer necessary during the second step of the reaction?