

Borohydride Reduction of Vanillin to Vanillyl Alcohol

The Design of a Greener Undergraduate Lab

Brandie Davis
Marian College
Directed Research 498
Spring 2005

Outline

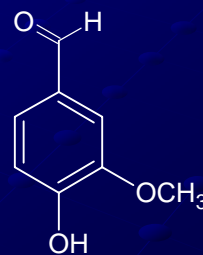
- My Research Goals
- Background
- Reduction Reactions
- My Research
- Green Results

My Research Goals

- To devise a lab that:
 - Appropriate for undergraduates
 - Safe
 - Relevant to lecture material
 - Appropriate length of time
 - Utilizes green principles
 - Reduce or eliminate use of solvents
 - Use knowledge of the substrates, reagents, and products to maximize efficiency
 - Solubilities

Background Information

- Natural vanilla flavoring comes from the fruit of the vanilla plant, which is native of Mexico
- Main component of vanilla flavoring is vanillin
- Natural vanillin is rather expensive



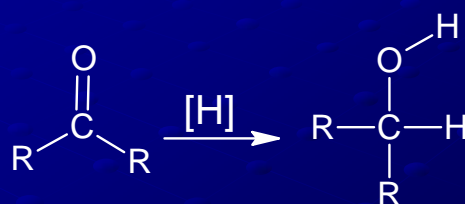
4-hydroxy-3-methoxybenzaldehyde

Background Information (cont.)

- Most vanillin is synthesized by breaking down lignin from wood pulp (far less expensive than natural vanilla)
- Vanillin is used in flavorings, perfumes, and starting material for the synthesis of drugs like L-dopa, which is used to treat Parkinson's disease
- Safe to work with and cheap (\$34/lb)

Reduction Reactions

- The addition of hydrogen and/or the removal of oxygen

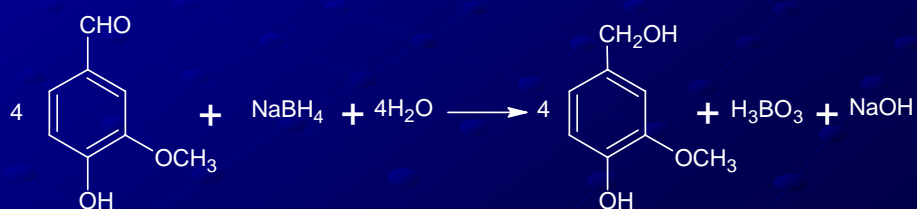


Reduction Reaction (cont.)

- In the case of this reduction reaction, vanillin is reduced to an alcohol when its carbonyl group gains 2 hydrogen atoms
- The hydrogens are provided by an appropriate reducing agent

Reducing Agent

- Sodium borohydride (NaBH_4)
 - Relatively safe to handle in its solid form
 - Sodium borohydride reductions involve a nucleophilic addition of hydride ion (H^-) to the carbonyl carbon



Sodium Borohydride (cont.)

- Sodium borohydride reductions are usually carried out in dilute 1M aqueous NaOH solutions or in an alcohol, such as methanol, ethanol, or 2-propanol
- Temperatures above 25°C can decompose the hydride

How is Reaction Typically Performed?

- Aldehyde or ketone is dissolved in a solvent
- Add NaBH₄ solution
- Organic extraction to isolate product

How to Isolate the Product?

- Vanillyl alcohol is soluble in:
 - Hot water
 - Hot and cold ethanol
 - Hot and cold ether
 - Hot benzene
- Vanillyl alcohol is insoluble in:
 - Cold water
 - Cold benzene
 - Forms supersaturated solutions in water

What to use?

- Dissolved NaBH_4 in NaOH
- Dissolved vanillin in small amount of ethanol

Now what?

- Added the NaBH_4 drop wise to the vanillin solution
 - Cool water bath
 - Precipitate formed
- Added 3M HCl to acidify reaction mixture
- Ice bath for 20 minutes
- Product and salts precipitate
- Isolated solids by vacuum filtration

Purification

- Need to separate product from salts
 - Product – soluble in ethanol
 - Salts – insoluble in ethanol
- Procedure:
 - Dissolve product (only) in minimal amount of ethanol
 - Filter off salts (impurities)
 - Allow ethanol to evaporate, leaving purified product

Identification

- I have product!
- mp and NMR confirmed it was vanillyl alcohol

Green Chemistry in this Undergraduate Lab

- Safe
- Inexpensive
- Fits time frame
- Minimal energy is required
- No organic extraction
- No recrystallization
- Safer solvent (ethanol)

Acknowledgements

- Dr. Carl Lecher
- Marian College Department of Natural Science and Behavioral Science

Questions?

??