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CHEM 345
11/3/2019

$NaBH_4$ Reductions: Vanillin

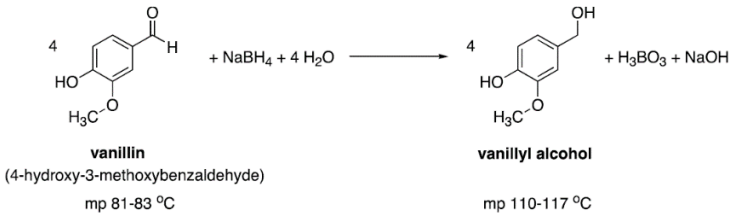
Reference: Handout; Chemistry lessons: Carbonyl chemistry, hydride reductions; Green lessons: renewable resources, benign solvents; Bruice p. 752-753

Purpose: To perform a sodium borohydride reduction of vanillin

Table of Reagents:

Reagents	MW	Amount	BP (°C)	MP (°C)	Density
Vanillin	152.5 g/mol	2.5 g	285 °C	82 °C	1.06 g/cm^3
$NaBH_4$	37.83 g/mol	5 mL	500 °C	400 °C	1.07 g/cm^3
Water	18.01 g/mol	Varies	100 °C	0 °C	1.00 g/cm^3
HCl	36.46 g/mol	Varies	-85.05 °C	-114.2 °C	1.20 g/cm^3
Ethanol	46.07 g/mol	5 mL	18.2 °C	-114.1 °C	0.79 g/cm^3

Balanced Chemical Equation:



Safety:

- Ethanol → flammable; Vanillin/$NaBH_4$ → irritant

Experimental Procedures	Data & Observations
1. Add 2.5 g vanillin, 5.0 mL ethanol, & stir bar to 25 mL round-bottom flask ➢ Warm flask in hand to drive dissolution of vanillin; clamp flask in ice bath	*Vanillin Used:* 2.4973 g
2. Add 5.0 mL $NaBH_4$ solution (3.42 M in 10 M NaOH) to separatory funnel and position over flask	

3. Add $NaBH_4$ dropwise over ~10 min period ➢ Work slowly & monitor temp below 25 °C ➢ Exothermic	
4. Remove from ice bath and allow to sit in RT for 10 min	
5. Place flask in ice bath and add 6 M HCl dropwise while stirring until H_2 gas is no longer evolved ➢ Work slowly; exothermic	*Observations:* Fumes coming from the top of flask
6. Check pH; Solution should be acidic. Stir in ice bath for 10 min and precipitate should form. ➢ Collect through vacuum filtration ➢ Dry product; Collect MP, mass, & IR	*Product Melting Point:* 98 °C *Product Mass:* 2.3961 g

Post-lab Questions:

1. **a)** We collected 2.3961 g of the crude product as well as the color of our product was styrofoam-white.

$$\%\ Yield = \frac{Product\ Obtained}{Theoretical\ Yield} \times 100$$

Obtained: 2.3961 g *Color and State:* Styrofoam-White

- First, we must obtain the theoretical value of our crude product in grams:

$$2.4973\ g\ Vanillin \times \frac{1\ mol\ Vanillin}{152.15\ g\ Vanillin} \times \frac{4\ mol\ Product}{4\ mol\ Vanillin} \times \frac{154.16\ g\ Product}{1\ mol\ Product}$$
$$= 2.5303\ g\ Vanillyl\ Alcohol$$

- Next, we must use the equation above to get our % Yield:

$$\%\ Yield = \frac{2.3961\ g}{2.5303\ g} \times 100 = 94.7\%\ yield$$

- *We received a 94.7% yield for our product*
- *We received a high percent yield!!!*

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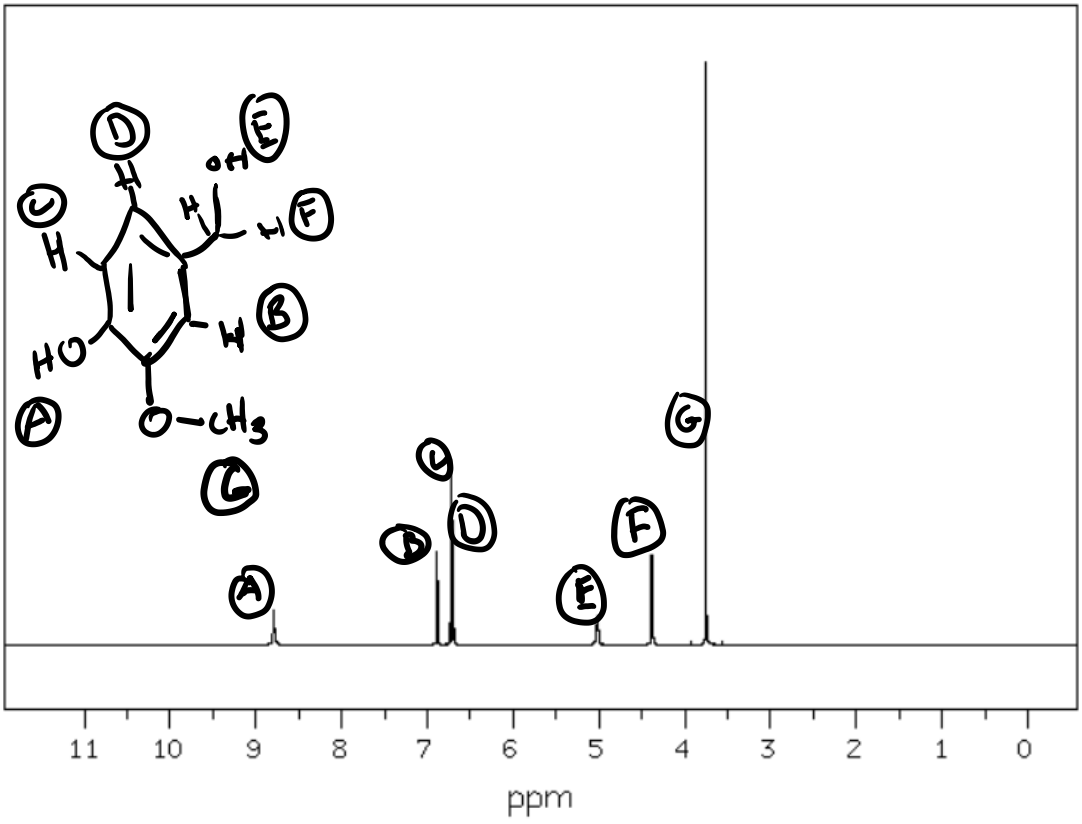
b) Our crude product melted at 98 °C, which concludes that it contains impurites as it has experienced melting point depression. The true melting point range of the pure product was recorded to be between 110 – 117 °C, whereas our product melted below the range. On the same note, this proves that our product's crystalline structure was disrupted by impuritues, which lowered our thought to be pure product's melting point. In addition, some of the reageants might not have fully reacted such as the vanillin, which might have resulted in the lower melting point.

2. The melting point for vanillyl alcohol is so much higher than that of vanillin because of the intermolecular hydrogen bonding. In vanillyl alcohol, the presence of the extra -OH group results in the increase of hydrogens to bond with one another, and as we all know, hydrogen bonding is one of the strongest intermolecular forces next to ionic bonds. This strong intermolecular force constitutes to the molecules to bind more tightly, hence the higher melting point.

3. IR and NMR of Vanillyl Alcohol

a) ON THE PAPER

b) H^1 NMR Vanillyl Alcohol



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c) We could use IR and NMR as tools to indicate a successful reaction by looking at the reaction before and after anything has occurred. In our experiment, we started with an aldehyde in our vanillin, which changes to an alcohol by the end of the reaction to make vanillyl alcohol. Throughout the reaction, we could monitor when this aldehyde turns into an alcohol by looking through an IR. We would see the C=O stretch vanish as we proceed to make more products (1720-1740 cm^{-1} would disappear). In addition, this would facilitate the appearance of an OH stretch around 3500-3700 cm^{-1} mark as seen with our IR spectra. Furthermore, the H^1 NMR would change as with the addition of the OH group, the overall conditions for the Hs would also change. Similar to the IR spectra, the hydrogen peaks in our NMR will change due to the switch from an aldehyde to an alcohol group. In vanillin, the aldehyde produced a peak around 10 ppm, whereas our alcohol appeared around 5 ppm.

4. Using excess $NaBH_4$ will increase the overall reaction rate, since the rate of the reaction is directly proportional to the concentration of $NaBH_4$. So if $NaBH_4$ is doubled, then the overall rate of reaction would be doubled as well.

$$Rate = k[aldehyde][NaBH4]$$

5. $NaBH_4$ (s) + HCl(aq) + 3 H_2O(l) → NaCl(aq) + H_3BO_2 (aq) + 4 H_2 (g)