Alan Yu

Organic Lab 309:03

Santanu Malakar

Experiment 29: Borohydride Reduction of Vanillin to Vanillyl Alcohol

March 11, 2019

Purpose:

To synthesize vanillyl alcohol by reduction of vanillin by borohydride.

Equations:

Mechanisms:

Amounts and Properties:

Table 1: Important properties for chemicals

Chemical	**Mol Wt**	**MP**	**BP** (d = decomposes)
Vanillin	152.2	79	285
Sodium Borohydride	37.83	37	400 (d)
Vanillyl Alcohol	154.2	115	(d)

Hazards and Safety:

Sodium borohydride is corrosive and toxic; can react violently with concentrated acids, oxidizing agents and other substances. Aqueous sodium borohydride solutions with pH lower than 10.5 have been known to decompose violently. Make sure solution is sufficiently alkaline. Avoid contact, don't inhale fumes, and keep away from other chemicals. No open flames nearby. Dispose all in marked containers under the hood.

Procedure:

The beginning TLC after starting the reaction is to determine if the reaction has gone to completion converting vanillin to vanillyl alcohol. To be completed, the TLC plate should have a spot that would've moved up the plate in a large density. After the reaction was done, saturated ammonium chloride to make the acidic condition for the borohydride reduction go through and reduce the ketone group. Washing this reaction twice is to remove all the other compounds and the remainder that hasn't converted from the organic layer. The purpose of collecting in fraction is to save time and to determine which fraction begins to stop having any sample after eluting. Rotovapping the fractions allows the solution to dry and create the crystals that are needed for data acquisition.

Observations:

After adding the NaBH4, the beginning solution changed to a vibrant yellow liquid. When adding the 10 mL of NH4Cl, the solution turned clearish yellow. When this was added, there was gas that was created which was the Hydrogen gas created. For Day 1 of the process, the TLC was used to determine when the vanillyl alcohol was made. To decide what eluent was to be used, the same eluent that was used for the Day 1 TLC was sufficient enough. When doing the TLC, there were 8 fractions collected. One plate had the even fractions and the other plate had the odd fractions. Since only fractions 1 through 6 showed a spot, 7 and 8 were excluded from the rotovap.

Measurements:

Table 2: Measurements from experiment

Vanillin used: .156g	Flask Mass: 48.044g
Day 1 Mass of Crystals and Flask: 48.164g	Day 2 Mass: 48.144g
Rotovap Mass: 48.154g	Melting Plateau: 106 - 110 degrees C

Data and Calculations:

Mass acquired: $48.154\,g - 48.044\,g = .110\,g\,Vanillyl\ Alcohol$

% Yield: .156g ¿ $\frac{154.2\,g}{152.2\,g} = .158\,g\,theoretically$

$\frac{.110\,g}{.158\,g} * 100 = 69.60\,\%\,yield$

Discussions:

The melting plateau for the vanillyl alcohol was from 106 to 110 degrees C. The documented value for the melting point is 115 degrees C. Since there was a discrepancy, the sample that was undergoing the melting point test could have still been wet or the thermometer was not correctly calibrated. Since there was a 69.90% yield, there could have been an error with how the TLC plate was developed. Either the TLC plate was developed past the solvent front or the container was moved when the plate was developing.

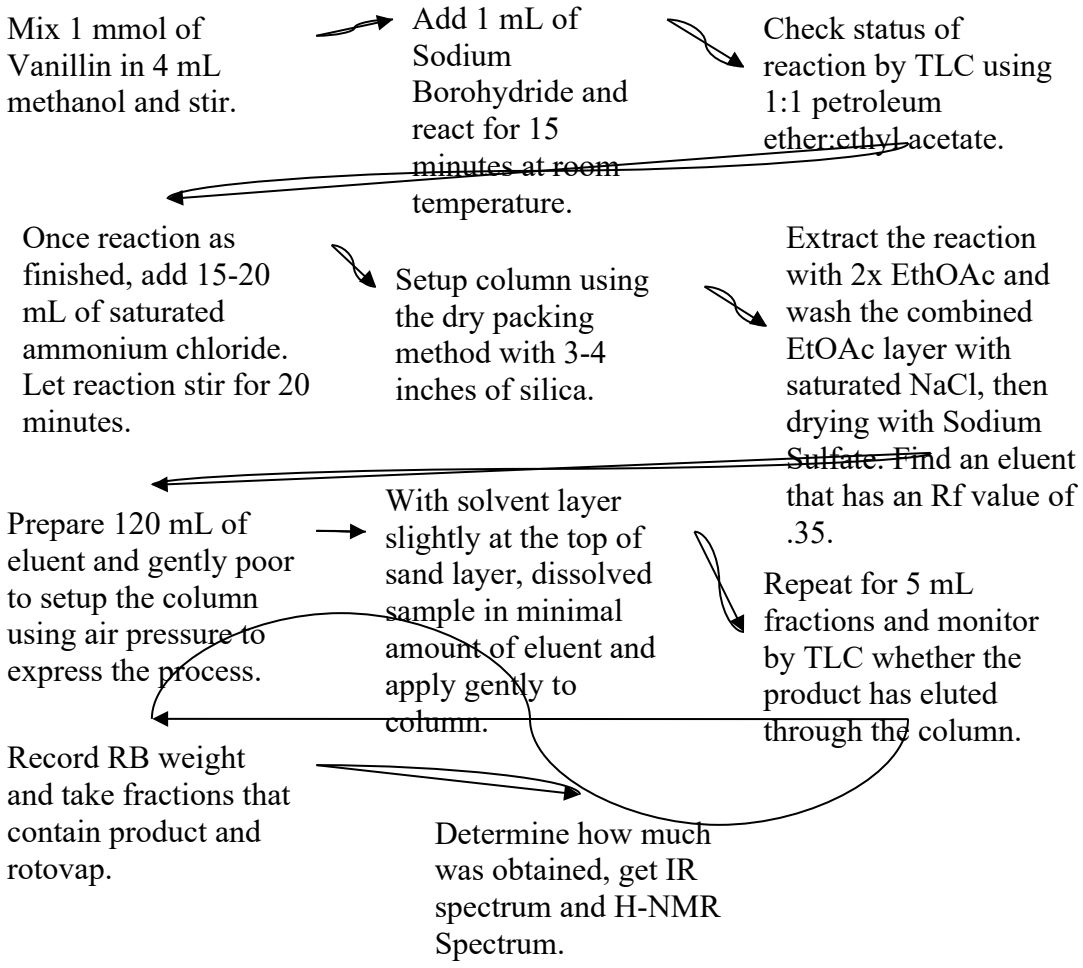
Conclusions:

From the IR spectrum of vanillyl alcohol compared to the vanillin, the difference in peaks would be the peaks in the 3200 region where it has the O-H stretch of the alcohol. The ones around the 1600 region stay there since there is still the benzene ring which applies to the aromatic C=C bonds within the compounds.

Exercises:

1. Mechanism: Same under the mechanism section of lab report.

5. Flow Chart:



7.