Borohydride reduction

ASSIGN BUSTER

John Heger CMY 211 Lab Section 13 11-30-2011 Borohydride Reduction of Vanillin to Vanillyl Alcohol Introduction: The purpose of this lab was to reduce vanillin to vanillyl alcohol. This lab report recaps the procedure and results of the lab. The chemical process studied in this lab was reduction, the process of reducing the number of bonds to oxygen and increasing the number of bonds to hydrogen. Other chemical processes included in the lab were recrystallization, melting point, and extraction.

Procedure: Preparation of Vanillyl Alcohol 2. 5 mmol of vanillin were dissolved in 2. 5 mL of 1 M NaOH solution in a 25 mL Erlenmeyer flask. The flask was swirled to produce a homogeneous yellow solution. The flask was swirled in an ice-water bath for 1-2 minutes and the solution was cooled to approximately 10°C. 1. 95 mmol of NaBH4 was added while the solution was constantly swirled. It was added in three to four portions over a period of 3 minutes. The solution was allowed to stand undisturbed for 30 minutes at room temperature.

After the time period, the flask was cooled in an ice-water bath and 3 M HCl was added dropwise with swirling. HCl was added until the pH of the solution was distinctly acidic to pH paper. The solution was gently cooled and the side of the flask was gently scratched with a glass rod to induce recrystallization. The crude product was suction filtered using a Buchner or Hirsch funnel and was washed with three small portions of cold water. The crystals were allowed to air dry for several minutes while suction continued to eliminate excess water.

The crude product was recrystallized from ethyl acetate. The dried crystals were weighed and a melting point was obtained. Results: Table 1:

Experimental Data Table Initial Mass of Vanillin| 0. 3843 g| Final Mass of Vanillyl Alcohol| 0. 2467 g| Melting Point Range of Vanillyl Alcohol| 75-90°C| Percent Yield| 63. 99 % yield| Discussion: The purpose of this lab was to reduce vanillin to vanillyl alcohol. The mechanism of the lab involved reduction. Sodium borohydride was used to reduce the aldehyde on the vanillin molecule, which left the oxygen with a negative charge.

Feature Article Relative Rates: Free-Radical Bromination

HCl then reacted with the negative oxygen on vanillin, and the oxygen then became protonated, which made another alcohol on the benzene ring. The melting point range obtained for vanillyl alcohol was between 75-90°C. The actual melting point was 115°C, which was approximately 25 degrees higher than the experimental values. The reason for this melting point difference can be attributed to impurities that developed throughout the procedure. The percent yield from the original sample was 63. 9%. A decent percentage of the original sample was lost throughout the reduction process of vanillin to vanillyl alcohol. Conclusion: Vanillin was reduced to vanillyl alcohol which obtained a melting point of 75-90°C. The percent yield from the original sample was 63. 99%. The mechanism involved the reduction of vanillin to vanillyl alcohol, which increased the number of bonds to hydrogen and decreased the number of bonds to oxygen by the reduction of a carbonyl.