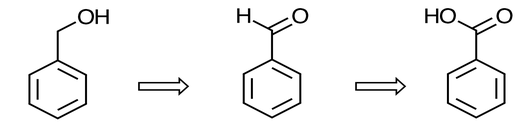
Oxidation of Alcohol to Produce Benzaldehyde

Katie Dietrich, Tia M. Hoisington, Wyatt Marcy, and Jered McGivern, Ph.D.

Department of Chemistry, Lakeland University, Sheboygan WI

Introduction

The oxidation of alcohols is a common and important reaction in chemistry. Primary alcohols can easily be oxidized into aldehydes then carboxylic acids, while secondary alcohols can be oxidized into ketones. Tertiary alcohols are rarely oxidized, but the reaction can occur after breaking one of the carbon-carbon bonds[2]. Traditional chemistry techniques involve the use of hazardous and expensive reagents and solvents that pose many health and environmental concerns in addition to producing large amounts of toxic waste. In addition, many of these reagents are not selective enough and are not effective when attempting to synthesize aldehydes because they will continue to fully oxidize the molecule until a carboxylic acid group is formed. This lab explores an alternative to the traditional methods. By utilizing less hazardous reagents, lower amounts of reagents, and producing lower amounts of toxic waste, this method is expected to be more cost efficient and environmentally friendly, as well as being selective to producing an aldehyde as opposed to a carboxylic acid. In this lab, an attempt be made to synthesize benzaldehyde from benzyl alcohol, without synthesizing benzoic acid[1].



Benzyl Alcohol — Benzyl Aldehyde — Benzoic Acid

Materials & Methods

Preparation of Tetrakis (benzyltriethylammonium) Octamolybdate Catalyst[1]

1. Add 0.30 g sodium molybdate dihydrate and 0.5 mL 4 M HCl to a vial.
2. Add 1 mL of water to complete the dissolution.
3. To a second vial, stir 0.525 g benzyl triethyl ammonium chloride (BTEAC) and approximately 3 mL water until dissolved. Heat the BTEAC solution to 70 °C with stirring.
4. Add the molybdate solution, dropwise, to the BTEAC solution, and stir for an additional 5 minutes.
5. Remove from heat and obtain the solid product by vacuum filtration. Wash the solid with approximately 5 mL water while on the filter under vacuum.

Preparation of Benzaldehyde[1]

1. Add 5 mL benzyl alcohol to a 50 mL round bottom flask containing 0.25 g dry catalyst (from Part 1).
2. Add 12 mL 15 wt% hydrogen peroxide to the flask. Heat the mixture to reflux for 60 minutes.
3. Allow the reaction flask to cool to room temperature. Isolate the product by simple distillation to yield benzaldehyde and water in the distillate.
4. Remove water with a pipet and record FTIR spectrum.

References

1. Selective Oxidation of Benzyl Alcohol to Benzaldehyde, Levy, I., Assor, K., Thames, E., and Walker, R., Gordon College Organic Chemistry Laboratory Experiment, laboratory exercise inspired by Guo, Ming-Lin; Li, Hui-Zhen Li., Selective oxidation of benzyl alcohol to benzaldehyde with hydrogen peroxide over tetraalkylpyridinium octamolybdate catalysts, Green Chem., 2007, 9, 421-423.
2. Smith, J. G. (2020). *Organic chemistry*. New York, NY: McGraw-Hill Education.

Results

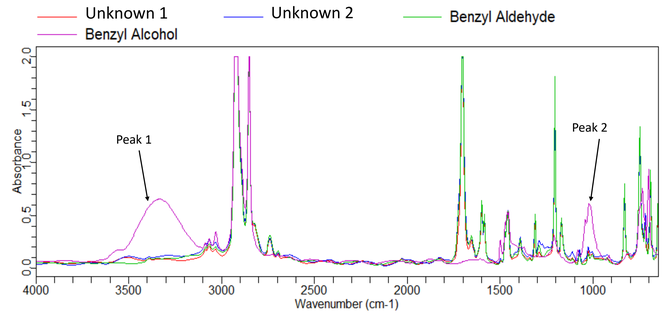


Figure 1: FTIR spectrum of pure benzaldehyde, pure benzyl alcohol, unknown sample 1 and unknown sample 2 overlayed. Peak #1 and Peak #2 within benzyl alcohol sample are different from benzaldehyde, unknown 1, and unknown 2.

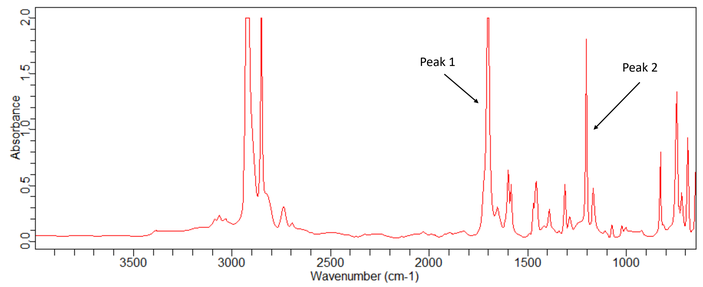


Figure 2: FTIR spectrum for pure benzaldehyde. Peaks #1 and #2 are the primary peaks that distinguish benzaldehyde from benzyl alcohol.

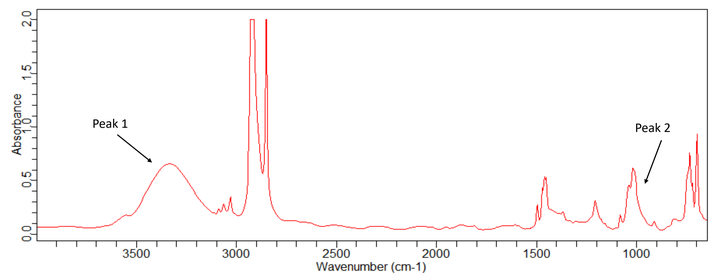


Figure 3: FTIR spectrum for pure benzyl alcohol. Peaks #1 and #2 are the primary peaks that distinguish benzaldehyde.

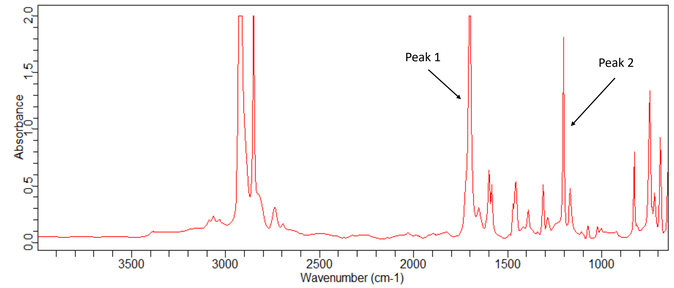


Figure 4: FTIR spectrum for unknown sample 1. Peak #1 and #2 match the corresponding peaks on Benzaldehyde FTIR spectrum (Figure 2).

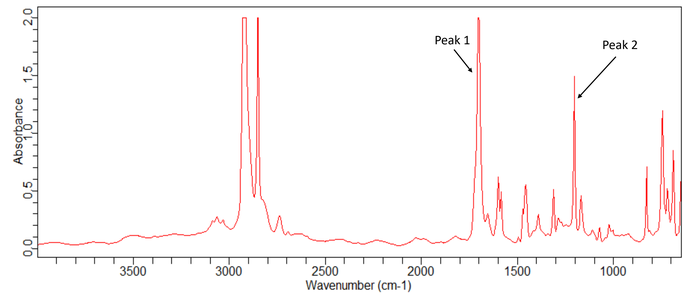


Figure 5: FTIR spectrum for unknown sample 2. Peak #1 and #2 match the corresponding peaks on benzaldehyde FTIR spectrum (Figure 2).

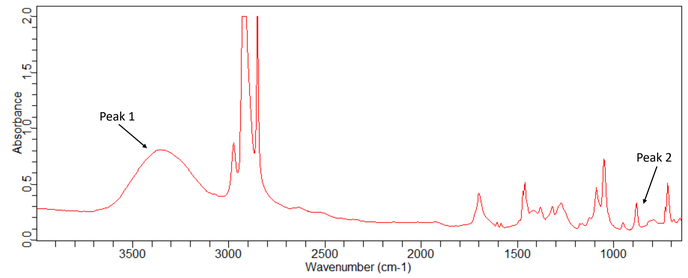


Figure 6: FTIR spectrum for benzoic acid. Peak #1 and #2 distinguish benzoic acid from benzaldehyde.

Discussion

Based on the FTIR spectra, it is concluded that benzaldehyde was successfully synthesized. The steep peaks found near 1700 and 1200 cm-1 on the spectrum for pure benzaldehyde (Figure 2) are also found on the spectra for the samples (Figures 4 and 5). Figure 1 overlays the spectra for pure benzyl alcohol (purple), pure benzaldehyde (green), and the samples (red and blue). This clearly illustrates the similar spectra of the samples with benzaldehyde, and the differing spectrum of benzyl alcohol. Additionally, the data shows that the reaction was successful in synthesizing benzaldehyde as opposed to benzoic acid as the peaks found around 3400 and 1100 cm-1 on the benzoic acid spectrum (Figure 6) are not found in the spectra for the samples.

Future Directions

- Run GC-MS of the unknown samples to verify that benzaldehyde was produced.
- Attempt oxidation of primary alcohols with additional organic compounds to determine the versatility of this method.
- Use other known methods of oxidizing primary alcohols and compare results with the results of this method to investigate the effectiveness and efficiency of methods.