The Development of Green Chemistry Labs for a High School Setting

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Abstract

We have developed a series of green experiments for the high school laboratory setting, some of which are already in use in schools around New York State. One of our experiments involves the reduction of cinnamaldehyde (from cinnamon) into 3-phenyl-1-propanol (which smells of hyacinth). This experiment is designed to demonstrate to students the drastic effect the simple reduction of unsaturated bonds can have on both the chemical and physical properties of an organic compound. Our main goal for perfecting this experiment is to find a reagent that is safe for high school students to use to reduce the cinnamaldehyde, as traditional reducing agents like lithium aluminum hydride are not safe for a classroom setting. This quest led us to use sodium hydroxymethanesulfinate, followed by sodium borohydride. The product has been tested using TLC, IR, GC-MS, and NMR, and shows promising results.

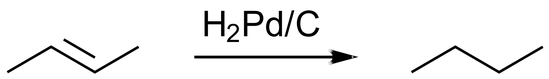
Introduction

In today's public educational environment, teachers tend to feel overworked and underappreciated, and have to deal with mandates from both the state and government, as well as curriculum changes. On top of that, schools are losing money, and departments are often underfunded. This, along with a general want to keep school children away from "dangerous" substances has led some to believe that hands-on work with chemicals in the science lab is not the best option, instead having students watch videos or do worksheets instead of actual labs. This can lead to students who are unmotivated and even scared to use these types of materials in the future because they were never taught how to use them properly.

A better solution would be to design safe and environmentally benign, yet active and engaging labs to allow students to participate in hands-on experiments pertaining to the curriculum. This way, teachers can spend less time worrying about dealing with mandates and disposal laws, and spend more time encouraging students the experience of working with chemicals, helping them to become comfortable using such substances, see clear examples of the material being taught, and hopefully learn to love the subject matter.

Examples of Organic Red/Ox Reactions

Reduction of an organic molecule:



Oxidation of an organic molecule:

Our Goals

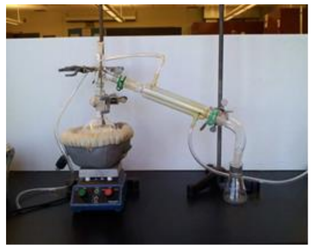
- Design an experiment to demonstrate the effect red/ox reactions have on physical properties.
- Have the experiment go to completion with minimal side products and minimal steps to show how easy it is to change the properties of a common compound without the use of hazardous material.
- Minimize the use or dangerous chemicals and hazardous waste
- Make an experiment that has a pleasant odor and fun to do!

Why the Green Approach?

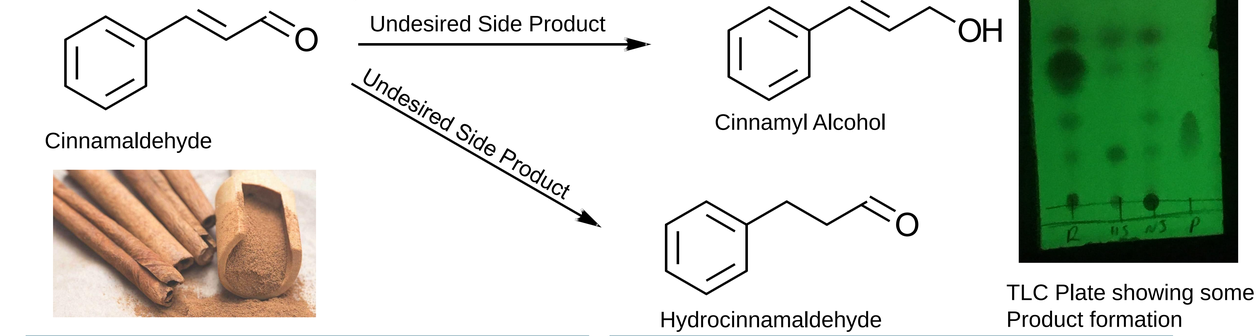
Many students may have environmental concerns and care about pollution and recycling. This experiment is designed to use a natural substance and convert it into a substance commonly found in perfume.

There are also no hazardous wastes produced in this experiment. However, because these compounds are organic in nature, they can not be put down the drain.

Main Project



Distillation of cinnamaldehyde from cinnamon, in earlier work from John Proetta



TLC Plate showing some Product formation

Red/Ox Reactions in Organic Chemistry

Organic molecules tend to be composed to long chains of carbon atoms with hydrogen atoms coming off of the carbons, with the occasional function group present. Sometimes in these carbon chains, a double or triple bond will form between two carbons, or between a carbon and a functional group. When these extra bonds are present in an organic molecule, it means that hydrogens have been removed from the carbon chain in order to make room for the new bond. On the other side of this, the double or triple bond can usually be removed simply by adding hydrogens to the carbon chain. These are the main types of reduction/oxidation (red/ox) reactions in an organic field. In this experiment, cinnamaldehyde is reduced by the addition of hydrogens to double bonds in the carbon chain, forming 3-phenyl-1-propanol.

What We've Done

Experiment #	Reducing Agent	Heated (Y/N)	Outcome
77A/77B	Aluminum powder	N	No reaction occurred. Aluminum did not dissolve in solution
77C	Sodium borohydride	N	Large amount of cinnamyl alcohol and hydrocinnamaldehyde produced
78	Sodium hypophosphite	N	Large amounts of cinnamyl alcohol and hydrocinnamaldehyde produced
79A	Sodium hydroxymethane sulfinate	N	Mixture of cinnamyl alcohol, hydrocinnamaldehyde, and cinnamaldehyde produced
79B	Sodium hydroxymethane sulfinate + nickel (II) chloride catalyst	N	Produced high yield of cinnamaldehyde produced, with noticeable amounts of cinnamyl alcohol and hydrocinnamaldehyde present
80	Sodium hydroxymethane sulfinate + sodium borohydride + nickel (II) chloride catalyst	Y	Produced high yield of cinnamaldehyde produced, with small amounts of cinnamyl alcohol and hydrocinnamaldehyde present

Desired Outcome

The major difference between cinnamaldehyde and 3-phenyl-1-propanol is the addition of four hydrogen to the chain coming off the benzene ring. Despite the difference between these two compounds sounding like a minor change, there are drastic differences between the two compounds in terms of physical properties. Some of the changes are easily testable in a lab setting, such as drastic changes in boiling points and freezing points, but the fun part of this lab are the changes that can be noticed through the use of the five senses

The first noticeable change between the two compounds is color, as cinnamaldehyde tends to be a yellow color, but 3-phenyl-1-propanol is colorless. However, the most appealing part of this reaction is the change in smell. As the name suggests, cinnamaldehyde has a strong smell of cinnamon. However, when full reduced, the resulting 3-phenyl-1-propanol has a smell of flowers and is a commonly used compound in adding scent to perfume.

Issues with the Reaction

One of the major drawbacks to this reaction is the tendency for the cinnamaldehyde to only be partially reduced to either Hydrocinnamaldehyde or Cinnamyl alcohol. This creates two problems. Firstly, as the names suggest, both of the partially reduced compounds still smell like cinnamon, so the most appealing part of the project is ruined if enough of the side products exist to cover up the smell Tof the desired product. The second problem is that having an impure product will produce skewed results when testing for other changed properties, such as boiling or freezing point.

Some solutions to this issue are to use multiple reducing agents, use more powerful reducing agents, or use excess reducing agents. However, these tend to go against the principles of green chemistry. Using an excess of materials or using multiple reagents is only wasting resources. As for using stronger reducing agents, the goal of this experiment is to use high school appropriate materials. Really strong reducing agents can be considered dangerous, and as such would not work in the high school setting.

Further Research

Research this semester was cut short due to the COVID-19 pandemic. As such, research that would have taken place did not. Further testing that was planned was to test the purity of the products produced using new instrumentation that was purchased by the department, such as NMR spectroscopy or GC-MS testing. Column chromatography testing would also have taken place to find the purity of the products. It is also likely that other reducing agents might have been tested, as the current procedure is done in two step, and the desired outcome is to be able to do the whole reaction in one step.