Lab 1: Green Synthesis of Camphor

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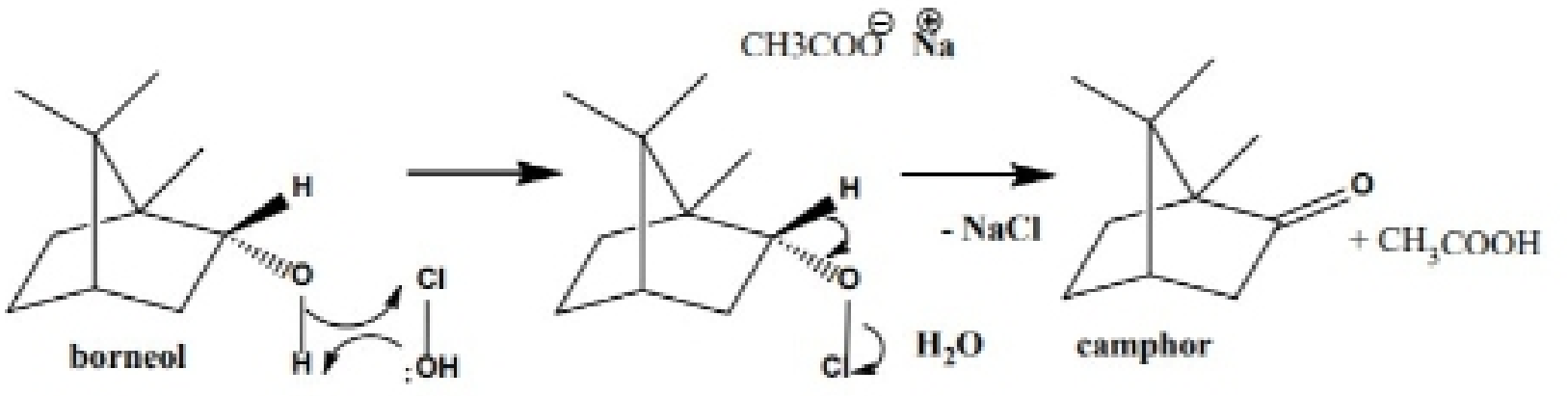
Abstract

The objective of the experiment is to oxidize a secondary alcohol, isoborneol to ketone (camphor) and determine the purity of the camphor to evaluate whether the synthesis is "green" or not. Bleach was used as a strong oxidizing agent to oxidize isoborneol into camphor, vacuum filtration was done to purify a crude sample, and sublimation was done to extract pure camphor from the crude sample. The amount of pure camphor obtained by the experiment is 0.013g and % yield, atom economy, reaction efficiency are 29.05%, 0.6655 and 0.1933 respectively.

Introduction

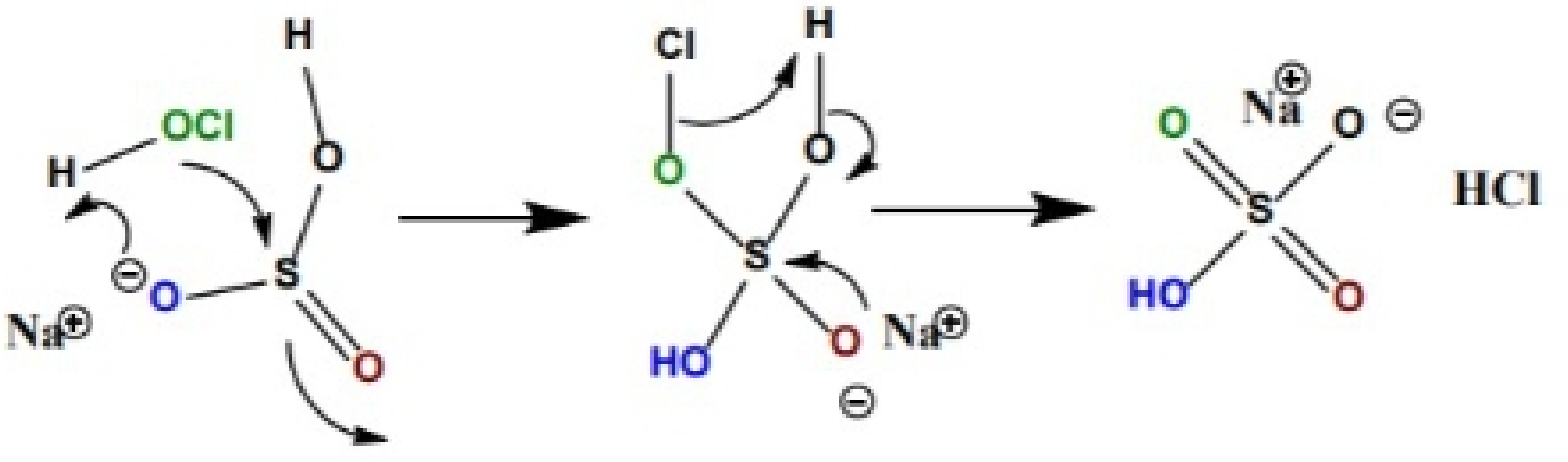
In this experiment, camphor was prepared by oxidizing a secondary alcohol, isoborneol to determine the purity of camphor. Oxidization is a chemical process in which a carbon atom gains bonds to more electronegative atom such as oxygen. Secondary alcohols can be converted to ketone using very strong oxidizing agents such as chromic acid. However, chromium oxidants are known to be corrosive and harmful to the environment. An environmentally friendly way to oxidize a secondary alcohol is by using bleach which contains sodium hypochlorite (NaOCl).



Adding small amounts of acetic acid facilitates the reaction by converting sodium hypochlorite to hypochlorous acid (HOCl). This is the active oxidizing agent.

Sufficient amount of sodium hypochlorite is added to ensure that reaction has reached its completion. When hypochlorous acid (HOCl) is present in excess, it can be tested with starch and potassium iodide indicator paper. The paper should turn dark blue, which is positive. This happens because starch and

iodide paper oxidize iodide ions to iodine. Excess HOCl that remains after can be eliminated by using sodium bisulfite as a reducing agent.



Camphor has a symmetrical structure which gives it the property to change directly from a solid to a vapor when heated. Thus, camphor can easily be purified by sublimation technique. Sublimation is a phase change in which a solid phase passes directly into the vapor without going through an intermediate liquid phase. Solids which have vapor pressure below melting points can be purified by heating the solid on the hot plate to sublime it and condensing the vapor on a cold surface such as a funnel.

The purity of the camphor synthesis was determined via % yield, atom economy and reaction efficiency calculations. While % yield is only considering one reactant and one product, atom economy and reaction efficiency give a good indication of the greenness on the process. The greenness of the synthesis of camphor from isoborneol was evaluated using such calculations.

Safety

-Concentrated Acetic acid: Corrosive, can cause serious burns, irritate eyes and respiratory tract. Reacts violently with water and other chemicals

-Sodium Hypochlorite: Contains chlorine, which can irritate eyes and respiratory tract.

-Hypochlorous acid: May cause skin dryness, direct exposure to eyes could produce irritation.

-Camphor: Flammable solid; harmful if swallowed, causes skin irritation, causes serious eye irritation, may cause respiratory irritation

-When handling these chemicals, always work under the fume hood and wear goggles, gloves and aprons.

Experimental

-Combine 2.5mmol of isoborneol with 0.20mL of glacial acetic acid in Erlenmeyer flask and swirl the flask that is sealed with parafilm for 30 minutes

-While stirring, add 4.5mL of NaOCl to the solution using pipet

-Test the solution for every 5 minutes with starch-iodide paper and if it shows a negative result, add enough NaOCl to give a positive result (dark blue color).

-When the paper shows a positive result add saturated sodium bisulfite to give a negative result.

-Cool the mixture in ice bath for 5-10 minutes

-Collect a crude sample via vacuum filtration and dry it at room temperature.

-Pre-weigh watch glass, funnel and add 50mg of crude on watch glass that is covered with a funnel and being heated on hot plate for 5 minutes

-Weigh the funnel that has pure camphor deposit on the inner walls of funnel.

Results

Mass of isoborneol used	0.387g	Mass of watch glass	19.324g
Volume of glacial acetic acid used	0.25mL	Mass of crude	0.45g
Volume of NaOCl used	5mL	Mass of crude used	0.053g
Drops of sodium bisulfite used	1	Mass of pure camphor	0.013g
Mass of funnel	17.872g		

Actual yield = mass of crude x mass of pure camphor/mass of crude used =0.45g * 0.013g/0.053g = 0.1103773585

Theoretically, 0.0025 moles borneol yields 0.0025moles of camphor.

Theoretical yield of pure camphor= MW of camphor x 0.0025mol = 152.2g/mol x 0.0025mol = 0.38g

% yield = $\frac{Actual\ yield}{Theoretical\ yield}$ x 100%= (0.1103773585g/0.38g) x 100%=29.05%

Atom economy = MW of Product/ sum of MW of all reactants =MW of camphor/(MW of sodium hypochlorite + MW of isoborneol) = $\frac{152.2\,g/mol}{(74.4+154.3)\,g/mol} = i0.6655$

Reaction efficiency = atom economy x % yield = 0.6655 x 0.2905 = 0.1933