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Green Experiment Oxidation of Borneol Summary Sheet

Overview:

This experiment analyzed the topic of green chemical synthesis through an oxidation reaction from borneol to camphor with Oxone. Camphor is a natural terpene product found in the camphor tree and has many applications including medicinal, pyrotechnics, and a preservative in pharmaceuticals and cosmetics. As the world is heading on an environmentally friendly path, green chemistry has been gaining traction in the scientific community. Green chemistry, also known as sustainable chemistry, is the concept of creating a greener chemical, process or product. Previous oxidation reactions creating camphor from borneol, such as the popular bleach conditions, in undergraduate organic teaching laboratories produced toxic and environmentally harmful by-products. In comparison, using Oxone with sodium chloride as the conditions yielded a reliable and efficient method that adhered to a few principles of green chemistry including, environmentally friendly solvents, no hazardous waste created and minimal energy requirements.

Reaction:

Reaction 1 below outlines the reaction observed in the experiment, the (1*S*)-borneol starting material was treated with 0.6 equivalent of Oxone and 0.3 equivalent of sodium chloride to produce the product (1*S*)-camphor. Due to the ratio of Oxone to borneol being 0.6:1, the amount of reagents were able to be reduced by half. The procedure is as follows:

- 0.500 g of (1*S*)-borneol placed into a 50 mL round-bottomed flask and dissolved in 2 mL ethyl acetate. A magnetic stir bar was added.
- While being stirred, 1.2788 g of Oxone and 0.0682 g of NaCl was added to the flask. The solution appeared to be clear with white undissolved precipitate at the bottom.
- Once the solution was stirred for 50 min at room temperature, 0.015 g of NaCl was added and stirred for another 10 min.
- Then, 7.5 mL of deionized water and a spatula tip of $NaHSO_3$ was added to the flask.
- The solution was then separated using a separatory funnel and the aqueous layer was tested with starch-iodine paper which indicated with a blue-black color that more $NaHSO_3$ was needed, so a spatula more was added.
- With the addition of 2.5 mL of ethyl acetate, the aqueous layer was again filtered out with the organic layer being added to an Erlenmeyer flask. The aqueous layer was then extracted two more times with the addition of 2.5 mL of ethyl acetate each time.
- The organic phases were then returned to the separatory funnel and washed three times with 2.5 mL portions of saturated aqueous sodium chloride solution, pouring the organic phase into a clean Erlenmeyer flask.
- Then the combined organic phases were dried with magnesium sulfate which formed white clumps at the bottom of the flask and was filtered via gravity filtration with fluted filter paper and transferred to another clean and pre-weighed Erlenmeyer flask.
- The solution was then heated gently on a hot plate while flowing a light stream of air through it until crystallization appeared.
- The Erlenmeyer flask was then re-weighed and the solid product obtained appeared to be crystallized on the edges of the Erlenmeyer flask and was white.

Conclusion:

Overall the product obtained had a crude mass of 0.4808 g producing a percent yield of 37.85. The low percent yield could be partially due to the missed steps involving desiccation and sublimation due to time constraints. Further, possible improvements within this experiment for the future could be a more gradual heating or a lighter stream of air over top of the crude product, during the concentration step. With proper preparation the oxidation with Oxone of borneol to camphor is a straightforward reaction and is able to be understood by beginner organic chemists.