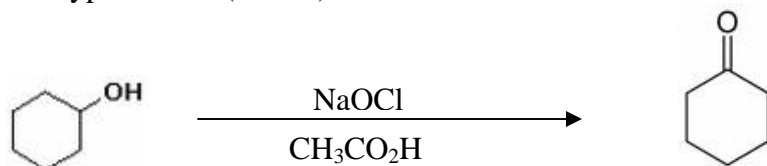


I. Purpose

In this experiment, the student will perform an oxidation reaction involving the interconversion of cyclohexanol to cyclohexanone using sodium hypochlorite (bleach).



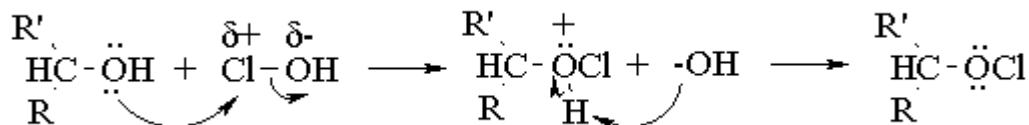
II. Introduction

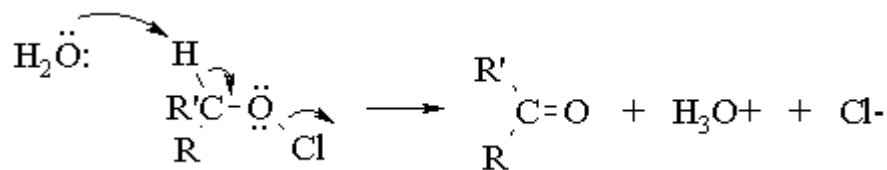
Alcohols that have a hydrogen adjacent to a hydroxyl (OH) group can easily be oxidized to a carbonyl compound.

If the alcohol is primary, then upon oxidation, an aldehyde results, which can be further oxidized to the carboxylic acid. Oxidation of a secondary alcohol will give rise to a ketone, and further oxidation presents no problem.

Wide varieties of oxidizing agents are known and are readily available to an organic chemist. Some of these oxidizing agents contain transition metals which are often expensive and toxic, presenting disposal problems. An excellent alternative is the use of commercially available sodium hypochlorite (bleach) since this reagent possesses lower toxicity and is more environmentally friendly.

Aqueous sodium hypochlorite (NaOCl), or common household bleach, can be used to oxidize secondary alcohols to ketones. The reaction occurs more rapidly under acidic conditions, so it is thought that the actual oxidizing agent is hypochlorous acid (HOCl), generated by the acid-base reaction between sodium hypochlorite and acetic acid. You have seen this reagent before, as that employed for the synthesis of α -chloro alcohols from alkenes (the chlorine analog of the reaction of alkenes with bromine in water). In alcohol oxidations, the reaction probably proceeds via E2 elimination of the alkyl hypochlorite produced by reaction of the initial alcohol with HOCl:





To ensure that complete oxidation of the alcohol occurs, it is necessary to employ an excess of sodium hypochlorite. Since the concentration of sodium hypochlorite in bleach can vary, a test for excess hypochlorite is performed by using starch-iodide solution. In acidic solution, iodide is oxidized by hypochlorite to iodine:



The iodine then reacts with the starch to produce the characteristic purple color of the starch-iodine complex.

III. *Experimental Procedure*

1. In a reaction vial containing, place 0.400 grams of cyclohexanol, 1.0 mL of acetone (0.5 mL), and 0.50 mL of glacial acetic acid.
2. Place the reaction vial in a water bath, set at 45 °C and mounted on a hot plate/magnetic stirrer. Gently flick or shaker the mixture for 5 minutes.
3. While the reaction mixture is in the water bath, add 4.0 mL of a 5% aqueous sodium hypochlorite solution over a total period of about 30 minutes. Add the bleach solution each time you flick the vial. Make sure the vial is cool enough to open. You may want to have a cool water bath to place your vial in.
4. When the addition is complete, remove a drop or two of the bottom aqueous layer with a clean pipet. Transfer this liquid onto a spot plate well. Add two drops of 1% starch solution and stir. Add two drops of potassium iodate solution to your spot plate well and stir. The purple color of the starch-iodine complex indicates that an excess of hypochlorite is present. If there is no color change, add an additional 0.2 mL of bleach to the reaction mixture, shake for 10 minutes, and repeat the starch-iodide test. Continue this process until the solution turns blue.
5. When the reaction time is complete, allow the mixture to cool to room temperature. Place ~7 mL of dichloromethane in a clean reaction vial, cap it, and bring it to your bench for use in the next few steps.
6. Use 2.0 mL of dichloromethane to rinse, into the reaction vial and the pipette you used to monitor the reaction above. Cap the vial, and shake it well with venting. Using a filter tip pipet, transfer the lower (dichloromethane) layer into another 5-mL conical vial.
7. Extract the aqueous layer twice more with 2-mL portions of dichloromethane, and combine them with the first dichloromethane extract.
8. Wash the combined organic extracts with 2.0 mL of 10% aqueous sodium bicarbonate solution. Stir the liquid until bubbling due to the formation of CO₂

- ceases. Cap the vial and shake with frequent venting to release any pressure produced.
9. Transfer the lower methylene chloride layer to another container and remove the aqueous layer. Return the dichloromethane layer to the vial and wash the solution successively with 2.0 mL of saturated sodium bisulfite and 1.0 mL of water.
 10. Using a dry filter-tip pipet, transfer the dichloromethane layer to a dry test tube or conical vial. Add three to four microspatulafuls of anhydrous sodium sulfate and let sit for 4-5 minutes with occasional shaking. This product should be your synthesized ketone.
 11. Develop your own analysis on how to determine your synthesized compound is cyclohexanone.

IV. Experiment Observations

V. Post-Lab Report

- Include the mechanism of the reaction to produce cyclohexanone starting with cyclohexanol.
- Side reactions that can occur in this experiment.
- Different oxidizing agents that can be used for this transformation other than using sodium hypochlorite.
- Clearly convey that the oxidation in the above reaction did in fact occur in your discussion section based on the results.
- Discuss any errors that were developed during the lab.