

# A Bleach Oxidation of a Secondary Alcohol to a Ketone

Stephanie Okerson  
CHE 498

## Outline

- ⌘ What is Green Chemistry
- ⌘ Oxidation reactions
- ⌘ The Hypochlorite Oxidation
- ⌘ Problems
- ⌘ My research
- ⌘ What's next?

## What Is Green Chemistry?

⌘ "The utilization of a set of principals that reduces or eliminates the use or generation of hazardous substances in the design, manufacture and application of chemical product."

P.T. Anastas and J.C. Warner, "Green Chemistry: Theory and Practice;" Oxford University Press: Oxford, 1998.

## Problems with Oxidation Reactions

⌘ Oxidations are often performed with chromic acid or PCC

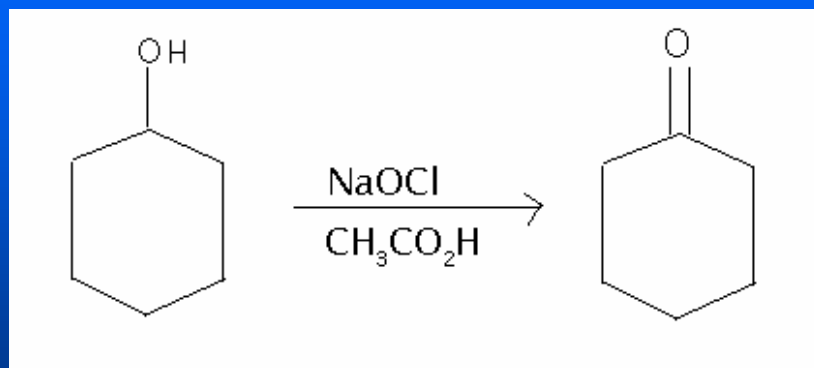
☒ these are carcinogenic

## Oxidation is good for Lab

- ⌘ This is a common and useful procedure
- ⌘ Other people are working on procedures to make this reaction more green.\*
- ⌘ High percent yield can be obtained by using bleach as the oxidizing agent.\*

\*Stevens R, Chapman K, Weller H. 1980. Convenient and Inexpensive Procedure for Oxidation of Secondary Alcohols to Ketones. *J. Org. Chem* 45:2030-2032.

## The Original reaction: Hypochlorite Oxidation



Stevens R, Chapman K, Weller H. 1980. Convenient and Inexpensive Procedure for Oxidation of Secondary Alcohols to Ketones. *J. Org. Chem* 45:2030-2032.

## What is non-green about this reaction:

- ⌘ It uses a large quantity of acetic acid
- ⌘ Waste products:
  - ☒ NaOH and water
  - ☒ Magnesium sulfate
  - ☒ Diethyl ether

## Other Problems with this reaction:

- ⌘ Acetic acid and Diethyl ether smell bad.
- ⌘ It takes an hour for the reaction to go to completion.

## The purpose of this project:

To make the reaction as green as possible, as safe as possible and as appropriate for an undergraduate lab as possible.

## Efforts to improve the reaction

- ⌘ Diethyl ether was replaced with ethyl acetate
- ⌘ Acetic acid, which was used as a solvent was replaced with a catalytic amount of hydrochloric acid

## What else was tried?

- ⌘ Changing the concentration of the bleach
- ⌘ Using more acid
  - ☒ These caused side reactions to occur
- ⌘ Heating the reaction
  - ☒ In the process of obtaining results

## Current Procedure:

- ⌘ Cyclohexanol and HCl are mixed in a round bottom flask
- ⌘ The flask is then placed in a ice bath and the bleach is dripped in slowly
- ⌘ The reaction proceeds under reflux for a half hour while being stirred.
- ⌘ Sodium bisulfite is added to neutralize the bleach
- ⌘ The solution is washed with ethyl acetate.
- ⌘ The ethyl acetate layer is dried using magnesium sulfate.
- ⌘ The magnesium sulfate is removed by vacuum filtration.
- ⌘ The ethyl acetate is evaporated off by using the rotovac.

## Results as of Today

The procedure is working but not is not going to completion. There is still starting material in the final product.

## What next?

- ⌘ Produce a more pure product
  - ☒ side reactions are a problem
- ⌘ Obtain result to determine if heating the reaction is useful

## Future Projects

⌘ Explore other starting materials

☑ could product precipitate out of solution

☑ no extraction would be needed

## Acknowledgments

⌘ Department of Natural and Behavioral Sciences

⌘ The CHE 498 Class

⌘ Dr. Lecher