Sodium Hypochlorite Oxidation of Alcohols

The attached article will serve as a starting point for this experiment.

"The design of laboratory experiments in the 1980's: A case study on the oxidation of alcohols with household bleach" by Jerry R. Mohrig , David M. Nienhuis , Catherine F. Linck , Carol Van Zoeren , Brian G. Fox and Peter G. Mahaffy in the Journal of Chemical Eduation, 1985, Vol. 62 (number 6), p 519-512

Maybe in the 1980s, students could sit around and wait an hour for the their reaction mixture to "brew," but in the twenty first century college students have places to be and things to do (besides Organic Chemistry, that is). To this end we will explore the procedure for the addition of bleach to a mixture of cyclohexanol and acetic acid. The authors of this JCE article describe several "improvements" they have made to previous procedures, but do not mention if they attempted to optimize the addition of bleach part of the experiment.

We will attempt to recreate the published procedure as closely as possible with the following considerations.

Notes on published experimental procedure:

1. Dispose of all chemicals in waste disposal hood please.

2. We do not have a large quantity of three-necked flasks. They cost about $50 apiece. Sorry.

3. In reference to the situation mentioned in 2, devise a method to perform a controlled addition of bleach, monitoring temperature, without allowing noxious vapors to escape from the reaction mixture.

Orginal literature procedure for addition of bleach:

1) Add 25 mL of bleach and swirl
2) Add an additional 90 mL of bleach in a controlled manner over the course of about 15 minutes being careful to not allow the temperature to exceed 45° C.
3) Add an additional 115 mL of bleach in a controlled manner over the course of 20 - 30 minutes being careful to not allow the temperature to exceed 45° C.
4) Swirl the mixture from time to time for another 20 minutes.

N.B. In order to gather critical data – record temperature of reaction mixture before addition of bleach and then afterwards at 5 minute intervals.

Proposals for modification:
Proposed modification 1:

Follow the previous procedure but reduce the time intervals by half.
Try to keep the temperature to under 45° C.
Record temperature of reaction mixture before addition of bleach and then afterwards at 5 minute intervals.

Proposed modification 2;

Mix the bleach “straight away” and let sit for 30 minutes.
Try to keep the temperature to under 45° C.
Record temperature of reaction mixture before addition of bleach and then afterwards at 5 minute intervals.

In lab analysis:

Obtain the IR spectrum of the product.

Data entry:

Record the yield in grams of product and your temperature data points at time = 0, 5, 10, 15, 20, 30, 35 min, … on a spreadsheet before leaving lab.

Submission for GC-FID and NMR analysis.

Hand in a sample of your product in a properly labeled vial: Your name, date, vials contents, and your experimental procedure (original, #1, or #2).

Checklist for completing the "Prelab" section:

(refer to Laboratory Syllabus for complete directions)

Title and Purpose.

____*Physical constants*. (2 points) Create a table of physical constants, solubility and safety data for the chemical compounds referred to in the procedure.

____ *Structures and equations*. (1 point) Write (using chemical structures) the balanced equation for this reaction. Hint: water is a product.

___ *Flowchart*. (1 point) Refer to "Procedure" Your assigned procedures: orginal:
(1R,2R,3R,4R,5R,6R,7R,8R)
modification 1: (1L,2L,5L,7L,8L) or modification 2 (3L,4L,6L,9,10)

___ *Calculations*. (2 points)

1) Determine the number of moles of acetic acid used.
2) Determine the number of moles of NaOCl used. Use 5.25g/100 mL NaOCl in Clorox.
3) Calculate the theoretical yield (in grams) of cyclohexanone.

Article:
_____ (1 point) According to the authors, what are the three advantages of using (sodium) hypochlorite rather than Cr(IV)?

____ Safety Question: (1 point) Consult the MSDS for Clorox <http://www.biosci.ohio-state.edu/safety/MSDS/CLOROX%20LIQUID%20BLEACH.htm> What Emergency/First Aid Procedures are advised if the product is splashed into one’s eyes?

Experimental Observations and Data:

Hand in a copy of your experimental observations and data before you leave lab.

Experimental Observations.

Refer to last semester and laboratory syllabus.

Lab Report Checklist:

Results.

____ Which experimental procedure did you use (original #1, or #2)?

____ (1 point) % yield of product → product mass x 100/theoretical yield. Show your calculations.

____ (1 point) Interpret the IR spectrum of your product. Did you obtain the desired product? How pure is it?

____ (1 point) Interpret the GC-FID chromatogram of your reaction mixture (GC-FID of standards may be obtained on the CHEM254 MyDU website.) Did you obtain the desired product? How pure is it?

Discussion and Conclusion.

Interpret the results of the class data. (2 points)
What are the issues that need to be addressed? (For example, is it sufficient to compare reported yields or are there other aspects of the experiment that should be addressed?)
What conclusions can be drawn?

What should next year's students do to continue to explore this intriguing reaction? (1 point)

Write a Journal of Organic Chemistry style abstract of this experiment including class data. (2 points)
JOC website: http://pubs.acs.org/journal/joceah
Sample article with abstract: http://pubs.acs.org/doi/pdf/10.1021/jo101791w

The Design of Laboratory Experiments in the 1980's

A Case Study on the Oxidation of Alcohols with Household Bleach

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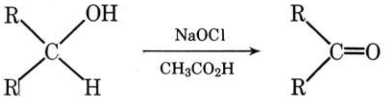
Chemists know that laboratory experiences are an important component of undergraduate education in chemistry. This is stated once again in the new Criteria and Evaluation Procedures booklet from the ACS Committee on Professional Training. However, teaching laboratories are expensive to operate and administrators may question the necessity of so many laboratory hours. We need to be clear on the goals and strategies in our laboratory courses.

Experiments must teach sound chemical principles, careful observation, and useful experimental techniques. They should be designed to catch students' interest, transporting them from passive spectators to active participants. Whenever possible, laboratory work should involve the commonplace and the fundamental. Experiments should relate to the common experience of students as well as to fundamental chemistry.

In the last few years undergraduate laboratory courses have faced new challenges—inflation and increased emphasis on laboratory safety and the disposal of hazardous wastes. An ideal experiment is inexpensive and safe and produces innocuous wastes. The use of some of the classic experiments in the organic chemistry laboratory must be re-evaluated in the light of these criteria.

One experiment, found in virtually all organic chemistry laboratory programs, is the oxidation of an alcohol with chromium(VI). The oxidation of alcohols to ketones relates two of the most important functional groups and is an important reaction in organic synthesis. Chromic acid has been used in introductory chemistry labs since the 1940's. Probably the most popular experiment is the oxidation of cyclohexanol to cyclohexanone, using sodium dichromate in an acidic medium. The oxidation occurs in reasonable yield and provides a good introduction to the use of infrared spectroscopy for functional group analysis (*1*).

In the summer of 1980 Stevens, Chapman, and Weller reported that "swimming pool chlorine" reacts with secondary alcohols to give ketones in good yield (*2*). Brief notes, adapting this method for use in the undergraduate laboratory, subsequently appeared in THIS JOURNAL (*3*).



What are the advantages of using hypochlorite rather than Cr(VI) oxidation of an alcohol? First, oxidations with hypochlorite present no hazardous waste disposal problem. The end products are chloride and the desired ketone; no toxic metal ions are present. Chromic acid oxidations pose a severe waste disposal problem, because chromium in any soluble form cannot be put down the drain in some states. Chromium(III), produced in the oxidation of a secondary alcohol, is toxic to fish and invertebrate species (*4*). Its acute toxicity depends upon water hardness. At 100 mg/L $CaCO_3$, the concentration of Cr(III) should not exceed 4.7 mg/L. The chronic toxicity of Cr(III) to freshwater aquatic life occurs at concentrations as low as 4.4 μg/L.

Second, the hypochlorite reaction offers substantial safety advantages compared to chromic acid oxidation. Chromium trioxide and its salts have a corrosive action on the skin and mucous membranes. For 1981, the time-weighted average (TWA) concentration limit of Cr(VI) compounds in the air was 0.05 mg/m^3 (milligrams of compound per cubic meter of air) (*5*). Fortunately, neither chromium trioxide nor sodium dichromate dihydrate is very volatile. Within the last few years some Cr(VI) compounds have been indicted as carcinogens (*6*). Even though the atmospheric levels of Cr(VI) compounds in any teaching situation would be very low, this must be viewed as a disadvantage. Concentrated sulfuric acid, commonly used in Cr(VI) oxidations, is also hazardous. The only appreciable safety concern in the hypochlorite oxidation is chlorine gas which must be contained effectively; more about this later.

The rising cost of common chemicals has prompted most chemistry teachers to reconsider common practices. The costs of sodium dichromate and chromium trioxide have risen dramatically in recent years. At current prices one can estimate a cost of $0.25 per student for the sodium dichromate alone in the Cr(VI) oxidation of 16 g of cyclohexanol.

Waste disposal, safety, and cost considerations associated with the Cr(VI) oxidation procedures provided the impetus for our exploration of sodium hypochlorite as an alternative oxidant.

The Stevens article reports the hypochlorite oxidation of 10 alcohols in isolated yields of 85–96% with acetic acid as the solvent. We adapted this procedure for our introductory organic chemistry laboratory, using "swimming pool chlorine" (12.5% by weight) to oxidize cyclohexanol to cyclohexanone, but encountered several problems. First, the large volumes of solvent (3–6 mL AcOH/gram of alcohol) and the ether used in multiple extractions made the experiment too expensive for classroom use. Second, "swimming pool chlorine" had a number of drawbacks. Storage of "swimming pool chlorine" causes its concentration to decrease by about 20% per month; therefore, a titration was necessary before its use. Since we found it available only in 5-gal containers, there was substantial waste in small to medium-sized classes. A more severe disadvantage was the modest chlorine odor apparent in the laboratory from time to time. Although Cl_2 concentrations never exceeded the maximum recommended ACGIH short-term exposure level (3 ppm) (*5*), the odor was quite unpleasant at this level.

To address these problems, we significantly altered the oxidation procedure in three ways. We found household bleach, a 5.25% (0.74 *M*) solution of sodium hypochlorite available at most grocery stores, to function even better than "swimming pool chlorine" as the oxidant for cyclohexanol. Yields were comparable to slightly better and atmospheric Cl_2 concentrations were diminished considerably. The atmospheric Cl_2 level was less than 0.2 ppm and was not detectable physiologically in laboratories in which 20 undergraduates were working. Bleach is available inexpensively in half-gallon and gallon containers and standard brands do not require titration. Recently, the use of hypochlorite bleach has been reported for the oxidaton of secondary alcohols (*7*).

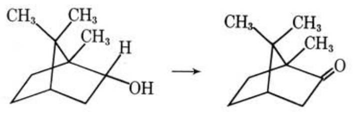
Second, we found no need for the large amounts of acetic acid reported by Stevens and others; 0.5 mL AcOH/g alcohol is sufficient. This point will be considered later in the discussion of the reaction pathway.

Third, we found ether extractions unnecessary when the acetic acid is neutralized with sodium hydroxide followed by a steam distillation of the cyclohexanone.

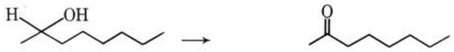
Class testing of this revised oxidation procedure by several hundred undergraduates in first-term organic chemistry laboratories showed that 50–60% yields of cyclohexanone are common. GLC analysis revealed that two-thirds of the students obtained cyclohexanone of >95% purity. Both Chlorox and Hilex brands of bleach worked equally well. The oxidation of 16 g of cyclohexanol by hypochlorite bleach works best; using smaller amounts produces unacceptable product losses on the semimicro 19/22 ₮ glassware. Higher yields (>75%) were obtained when extractions were used; dichloromethane is an effective extraction solvent. We chose not to use the extraction step in order to avoid any organic waste product. Neutralization of the acetic acid is necessary to avoid its codistillation in the steam distillation of the product. Addition of a few drops of an acid-base indicator makes the visualization of the neutralization step colorful and straightforward.

At current prices the chemicals for the entire experiment cost about $0.35 per student. Therefore, oxidation of a secondary alcohol with hypochlorite is somewhat less expensive than the Cr(VI) oxidation.

In addition to the cyclohexanol oxidation, we have used the same procedures for the synthesis of camphor from borneol and 2-octanone from 2-octanol. Both of these ketones are natural products. Camphor has many medical and industrial uses. Its fragrant and penetrating odor is familiar to most students. These two oxidation reactions can serve as excellent variants for adventuresome students and teachers.



borneol camphor



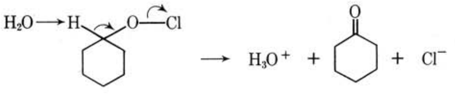
2-octaone (alarm pheromone of the ant, *Crematogaster peringueyi)*

Historical Interlude and Reaction Mechanism

With all of the advantages of positive chlorine as an inexpensive, safe oxidant, it is somewhat surprising that it has not been a traditional reagent for the transformation of secondary alcohols to ketones. The oxidation of methyl ketones by means of alkaline hypohalite, known to generations of organic chemistry students as the haloform reaction, was first reported by Lieben in 1822 (*8*). One can only speculate why it took until 1980 to appreciate the generality of the aqueous hypochlorite oxidation of secondary alcohols. In part, the delay may have resulted from the perceived lack of a convenient chlorine source, since the generation and handling of chlorine gas can be awkward. The synthesis and use of *tert*-butyl hypochlorite, another traditional oxidant, is both inconvenient and potentially unsafe. In the last decade Corey (*9*) and others (*10*) have developed the use of complexes of Cl_2 with dimethylsulfoxide, dialkyl sulfides, and hexamethylphosphorous triamide. These offer a number of advantages but are not suitable for large-scale use in the teaching laboratory.

Neglect of sodium hypochlorite for oxidizing alcohols may have also resulted from the lack of appreciation of the role of acid in the reaction. Many kinetic studies have been carried out on the oxidation of alcohols with Br_2/H_2O, but this reaction has a different acidity dependence than when chlorine is the oxidant (*11*). Alkaline aqueous solutions of sodium hypochlorite will not oxidize alcohols without additional catalysts; for example, $RuCl_3$ and phase-transfer catalysis have been reported (*12*, *13*). At low pH, molecular chlorine is a major component of the aqueous hypochlorite system. Alkali greatly increases the solubility of Cl_2 by converting it to chloride and hypochlorite; the pH of bleach is about 12.5. Disproportionation and evolution of oxygen are slow at room temperature, so one can produce stable solutions of hypochlorite.

Not a great deal is known about the mechanism of the hypochlorite oxidation of alcohols. Presumably, hypochlorous acid must be present for the reaction to proceed. Kudesia and Mukherjee have proposed that molecular chlorine is the actual oxidant and the alkyl hypochlorite ester is an intermediate (*14*). Their kinetic studies on the oxidation of 2-propanol with chlorine provide the most detailed mechanistic studies available. If an alkyl hypochlorite is an intermediate, one could envisage a simple E2 elimination leading to the ketone (*15*).



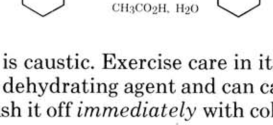
One expects that the mechanism of hypochlorite oxidation of alcohols will be unraveled more completely in time, through the use of some of the elegant experimental techniques that have been applied to Cr(VI) oxidations.

Our research has shown that the presence of molecular oxygen, hydroquinone, or galvanoxyl does not affect the hypochlorite oxidation. Therefore, free-radical chain reactions would seem to be unimportant in the oxidation.

Our kinetic studies on the acid dependence were done primarily with 2-propanol, which allows a homogeneous reaction mixture. Reaction aliquots were quenched with excess acidified iodide and the usual thiosulfate titration carried out. Reagent-grade sodium hypochlorite was the oxidant. The reactions were carried out under flooding conditions, where the concentration of 2-propanol was 20–30 times greater than that of the hypochlorite. The reaction was shown to be first-order in oxidant. The pH was held constant at 25° with sodium pyrophosphate (pH 9.9 and 7.8) and sodium acetate (pH 4.1) buffers. The reaction of hypochlorite with 2-propanol was very slow at pH 9.9; only 6% reaction occurred in 12 h. At pH 7.8 it was 83% complete in 15 min, whereas at pH 4.1 it was 83% complete in 5 min. Probably, any pH less than 7 will provide a facile oxidation pathway. The pH dependence may reflect the need for hypochlorous acid. At pH 8.0, 24% of the total chlorine is present as hypochlorous acid; at pH 6.5, 91% is present as hypochlorous acid (*8*). Since acid is produced in the oxidation itself, care must be taken in studying the acid/rate relationship. We also confirmed earlier reports that metal chlorides can catalyze the oxidation (*14*).

Oxidation of a secondary alcohol seems to proceed faster than chlorination at the α-carbon of the related ketone in a weakly acidic environment. Both "swimming pool chlorine" and hypochlorite bleach produce good yields of unchlorinated ketones (*2*, *3*, *16*). The ring of cyclohexanone can be cleaved and adipic acid produced with alkaline hypohalite solutions (*8*), although we observed no ring cleavage under our reaction conditions.

The Experiment



Caution: NaOH is caustic. Exercise care in its use. Glacial acetic acid is an excellent dehydrating agent and can cause burns. If either gets on the skin, wash it off *immediately* with cold water. Acetic acid fumes are also irritating, so the acid should be poured in the hood. The same is true for the bleach solution. Chlorine gas is a respiratory and eye irritant. Its acceptable short term atmospheric exposure level is 3 ppm. Cyclohexanol and cyclohexanone are moderate irritants; the acceptable 8-h average atmospheric exposure limit for cyclohexanol is 50 ppm; for cyclohexanone it is 25 ppm. The acceptable short-term exposure level for cyclohexanone is 100 ppm. Small quantities of excess sodium hypochlorite should be disposed of in a fume hood by slow addition to a large volume of concentrated reducing agent (sodium bisulfite or sodium thiosulfate). When reaction is complete, adjust the pH to 7 and flush down the drain with large amounts of water (*17*).

Pour 16 mL (0.15 mol) cyclohexanol into a three-necked 500-mL round-bottomed flask. Add 8 mL glacial acetic acid. Place a condenser in one neck of the flask, a 125 mL dropping funnel in another and a thermometer in the third opening. If the laboratory is well ventilated or the experiment is being carried out in a hood, the apparatus is complete. (Without good ventilation, put a piece of glass wool in the bottom of a drying tube and pour in ~2 cm of sodium bisulfite. This will react with any Cl_2 gas that might escape up the condenser. In the top of the condenser, placed a one-hole rubber stopper with the drying tube attached.)

Grease the stopcock in the 125 mL dropping funnel, and take it to the hood to put in 115 mL of the bleach solution. When pouring the bleach, use rubber gloves and dispense the solution carefully. Stopper the dropping funnel in the hood, then carry it back to the bench with a large beaker underneath it, and return it to your three-necked flask. Put a small piece of paper between the stopper and the neck of the dropping funnel so the solution will drop evenly. Make available an ice bath with about 250 mL of ice in case the reaction gets too hot.

Add about 25 mL of the bleach solution to the reaction vessel. Swirl the vessel to mix the reagents together. Then start adding the rest of the sodium hypochlorite solution, swirling the flask from time to time during the addition so that the reactants remain well mixed. The rate of addition will be determined by the temperature of the solution, but should take no longer than 15 min. Ideally, the maximum temperature should not exceed 45°; 40–50° is fine, however. Use the ice bath if the reaction temperature exceeds 45°. If the reaction is kept too cold (<40°), the product yield may be low.

After all the sodium hypochlorite has been added, remove the dropping funnel, temporarily stopper the reaction flask and obtain another 115 mL of bleach solution in the dropping funnel. Add this to the reaction mixture, keeping the temperature at 45°. Addition of the total 230 mL of bleach should take about 20–30 min. In the early stages of the reaction, one can see the color of the chlorine disappear as it reacts. Near the end of the addition,the yellow color should linger. After addition is complete, remove the dropping funnel and stopper the neck of the flask. Swirl the mixture from time to time for another 20 min.

Test for Excess Oxidant

$$\underset{\text{bisulfite}}{OCl^- + HSO_3^-} \rightarrow \underset{\text{bisulfate}}{Cl^- + HSO_4^-}$$

Wet a piece of starch-iodide indicator paper (if the bleach is too concentrated, colorless iodate may form) and put a drop of aqueous solution from the reaction vessel onto it. If excess oxidant remains, the blue color of the triiodide-starch complex will appear. If you get a blue color, add 2 mL saturated $NaHSO_3$ solution to the reaction vessel and stir. This should react with any remaining oxidant. Perform the starch-iodide test again. Continue the addition and testing until no excess oxidant is present.

Remove the thermometer and condenser. Rinse them, as well as the dropping funnel, immediately with water and then sodium bisulfite solution. Add 2 mL thymol blue indicator solution to the reaction mixture. Then over a 3-min period add 6 *M* NaOH (about 30–40 mL) through a short-stemmed funnel until the solution is neutral (color change to blue). Swirl the flask gently at times during the addition of base.

Distill the reaction mixture using a short-path column; cyclohexanone and water will codistill. Approximately 70–80 mL of distillate will have to be collected to recover all of the product. Since it is somewhat water soluble, do not collect too much distillate. Cyclohexanone forms the top layer in the receiving flask.

Add 10 g solid sodium chloride to the distillate in order to decrease the solubility of cyclohexanone in the aqueous layer. Stir the mixture until almost all of the salt dissolves. Separate the layers and purify the crude product by drying it for 20 min over 2 g anhydrous magnesium sulfate *or* potassium carbonate.

Options and Comments

If the bleach has been stored for many months, one may want to increase the quantity by 5%; it is present in just over 10% stoichiometric excess in our procedure. Extraction with 30 mL of CH_2Cl_2 before drying will increase product yield for two reasons—it will recover more of the ketone that is dissolved in the water and it will facilitate filtration of the drying agent. However, using CH_2Cl_2 adds a distillation step. Analysis of the product can be accomplished by GLC or infrared spectroscopy (*1*).

There are many interesting open-ended options possible with this experiment. Numerous secondary alcohols can be substituted for cyclohexanol. The procedure is simple enough that students can design their own experimental variations after looking up the physical properties of the secondary alcohol and the related ketone. The proper amount of sodium bisulfate can be substituted for acetic acid and the neutralization step omitted. Isopropyl alcohol can be used in place of sodium bisulfite to destroy the excess oxidant (*16*): any remaining acetone can be removed easily by distillation.

The oxidation of secondary alcohols with common hypochlorite bleach provides a safe, environmentally sound, and inexpensive modern synthetic method. It utilizes a variety of laboratory techniques and some fundamental oxidation-reduction chemistry. The experiment provides an opportunity for the student to review chemistry described in most introductory chemistry courses. It offers some pretty colors and uses an oxidant known to virtually everyone. Numerous experimental options are available within the experiment.

Acknowledgment

Our thanks go to Jean Mohrig and Richard Ramette for helpful suggestions and to the many Carleton undergraduates who performed variations of the experiment. A Curricular Research and Development Grant from Carleton College to JRM and PGM helped to support part of this work.

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