

Comparison of green oxidation methods in organic chemistry lab

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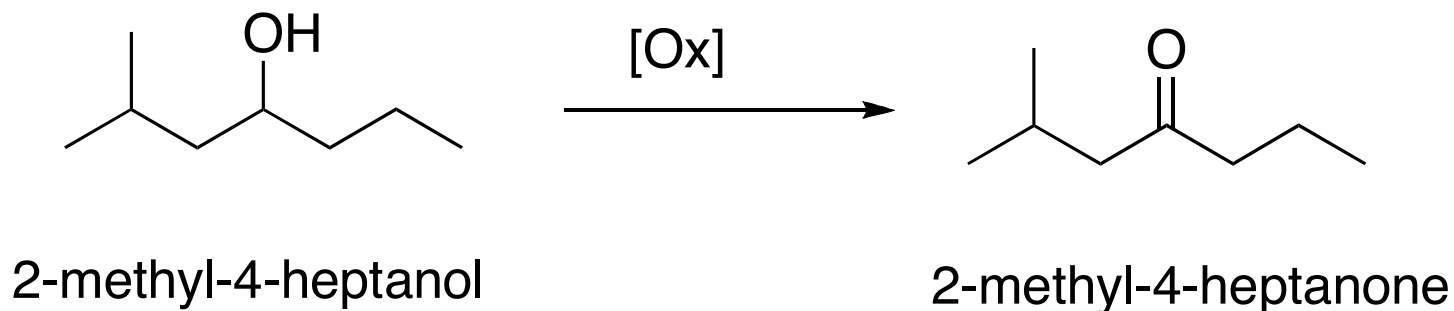


Substrate



As part of a natural product synthesis, students in second semester organic chemistry lab prepare 2-methyl-4-heptanol using a Grignard reaction.

The secondary alcohol is then oxidized to a ketone forming 2-methyl-4-heptanone, an ant pheromone.



Methods of oxidation



Oxidations of alcohols have traditionally been performed using Chromium VI reagents

- Greener methods developed in the last few years include:
 - Peroxide/Sodium Tungstate
 - $\text{CuSO}_4/\text{KMnO}_4$
 - Bleach/Acetic acid
 - Oxone/NaCl



Comparing 2 oxidation methods

- Our labs have been performing the oxidation using a solution of bleach (12%NaOCl) in acetic acid for 5 years.
- In 2 labs this semester, half of the students tried the oxidation with oxone (KHSO_5) and sodium chloride in ethyl acetate/water solvent. The workup procedure was modified between the labs.

Results of oxidations



Reaction products were analyzed by GC after extraction workup.
(Extraction procedure for oxone lab was modified for lab 2.)

	Method	Range % oxidized	Average % oxidized
Lab 1	NaOCl	38%-52%	47%
	Oxone	57-94%	88%
Lab2	NaOCl	42-72%	62%
	Oxone	80-94%	89%

Conclusions



- The reaction of 2-methyl-4-heptanol with oxone and sodium chloride showed overall more complete oxidation than reaction with bleach and acetic acid.
- Based on observations of bleach oxidation from past years, there was also less variability between students results using oxone.



Comparison of Some Green Attributes

Reaction	Workup solvent	By products
Oxone/NaCl	Ethyl acetate	K_2SO_4
Bleach/Acetic acid	Dichloro-methane	NaCl, CH_3CO_2H

References



Oxone Oxidation:

Lang, P.T. Harned, A.M., Wissinger, J.E. *J.Chem.Ed.*
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Bleach/Acetic Acid Oxidation

Pavia, D.L., Lampman, G.M., Kriz, G.S., Engel, R.;
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