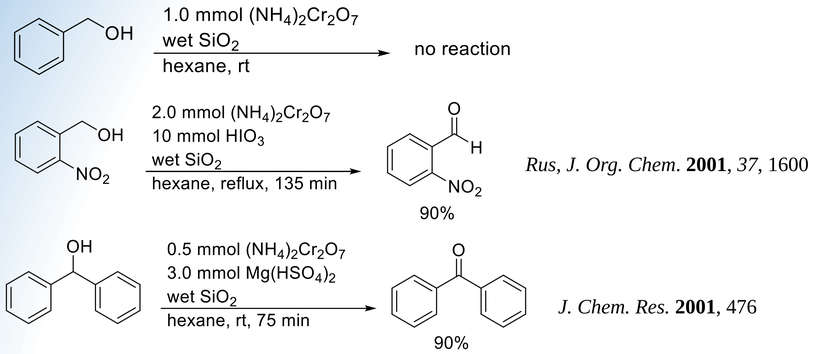
Developing Oxidation of Alcohols in Sand

John Lepore, Emily Toy, David Keeley, Elizabeth Merges, Jasmine Uralil, Stephanie Alvarez, Katelynn Dux, Theresa Lam, Dr. Robert Torregrosa

Department of Chemistry, State University of New York at Geneseo, Geneseo, NY 14454

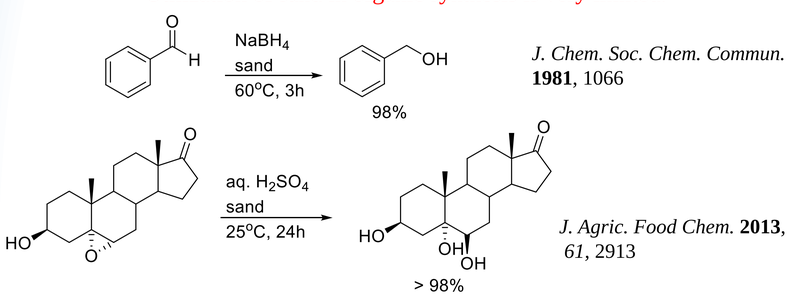
Introduction

The use of $(NH_4)_2Cr_2O_7$ in wet silica gel affords products when stoichiometric additives are used. (Torregrosa, R.R.P.; Lepore, J.A. "Ammonium Dichromate" *electronic-Encyclopedia of Reagents for Organic Synthesis, e-EROS,* **2020**.)

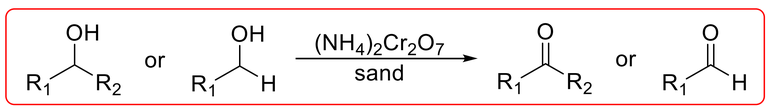


Silica Gel and Sand are both SiO_2

Utilization of sand in organic synthesis is very limited.

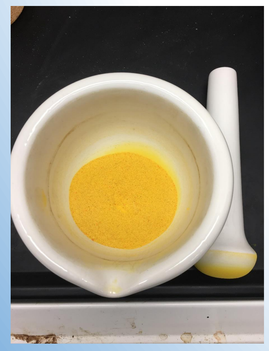


Advantages of Sand in Cr-based Oxidation of Alcohols

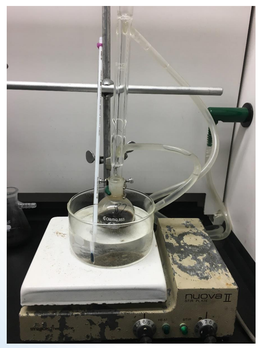


- Sand is cheaper ($32.00/500 g – Fisher) than silica gel ($698.00/500g - Fisher).
- Sand is less harmful than silica gel (known irritant).
- Sand is abundant in nature and has many practical uses such as geology and construction.
- Sand does not affect separation of mixtures in column chromatography unlike silica gel.
- Sand is not difficult to handle and easily accessible.
- Sand is also SiO_2 and should possess similar characteristic profile and activity just like silica gel.

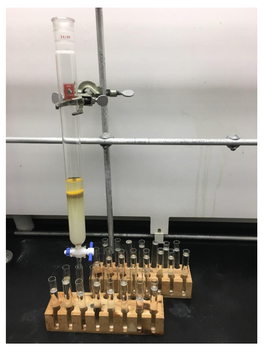
Reaction Design



1
Grind $(NH_4)_2Cr_2O_7$ (orange) and washed sand to produce yellow mixture for 15 min



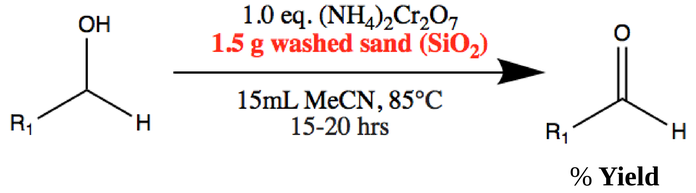
2
Add the alcohol to the ground Cr-SiO_2 in a flame-dried flask with solvent for reflux

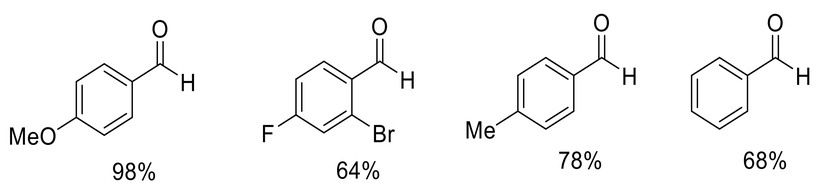


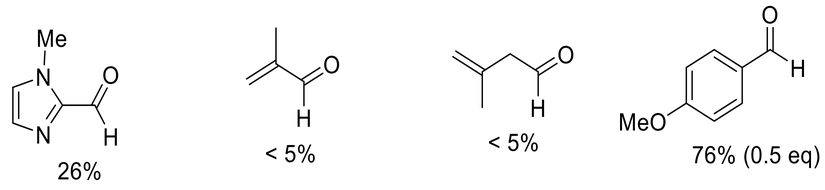
3
Purify crude material from reflux by column chromatography

Data and Results

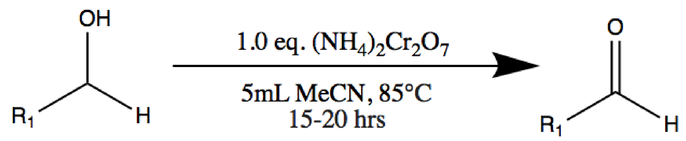
Part 1: Oxidation of primary alcohols into aldehydes in sand

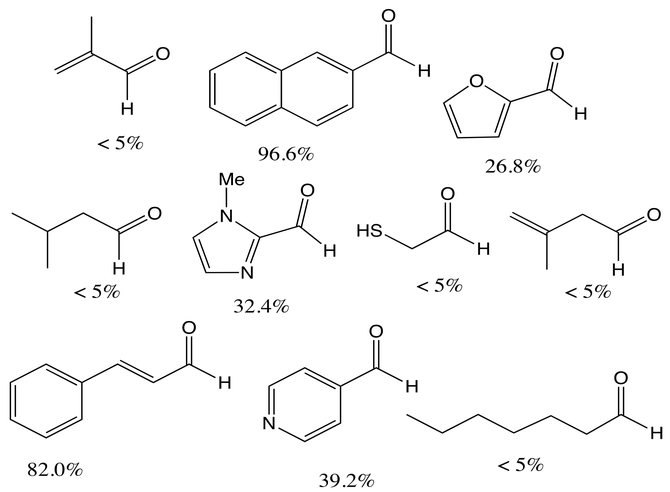




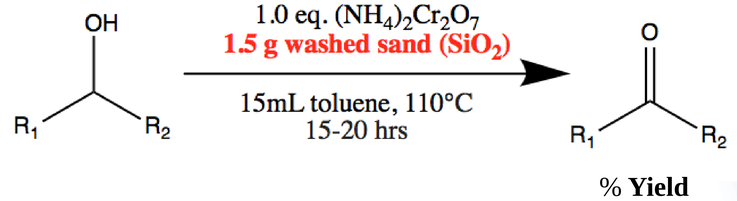


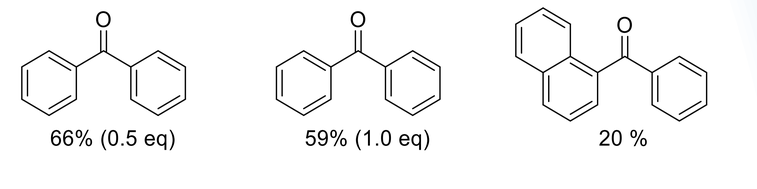
Part 2: Oxidation of primary alcohols into aldehydes without sand





Part 3: Oxidation of secondary alcohols into ketones in sand





Conclusions

1. Assuming percent conversion and percent yield are accurate representations of one another, there appears to be no major difference between oxidation of primary alcohols in sand and no sand conditions, except for 2-napthelenemethanol and furfuryl alcohol.
2. Simple, primary allyl alcohols of low molecular weight do not effectively oxidize into aldehyde forms under present conditions.
3. The substantial difference between percentages in furfuryl alcohol suggests that sand conditions are favored for Cr-based oxidation of furfuryl alcohol.
4. Secondary alcohols are tolerated towards oxidation.

Future Directions

1. Primary alcohols run in no sand conditions will be purified via column chromatography in order to determine percent yields.
2. Oxidation of primary alcohols in silica gel conditions will be done to allow for complete comparison of Cr-based oxidation of primary alcohols.
3. Investigation of various secondary alcohols in sand, no sand, and silica gel conditions will be carried out to determine if trends found among primary alcohols persist in secondary alcohols.

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