Investigating Oxidation of Alcohols with (NH₄)₂Cr₂O₇ in Washed Sand

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Investigating Oxidation of Alcohols with (NH4)2Cr2O7 in Washed Sand

Abstract
The oxidation of alcohols to aldehydes and ketones is among the important class of reactions in organic chemistry. Classical methods used to oxidize primary and secondary alcohols into aldehydes and ketones utilize chromium-based reagents such as pyridinium chlorochromate (PCC) and sodium dichromate (Na2Cr2O7). There has been an increasing interest throughout the years to use supported reagents in tandem with these reactions, particularly with silica gel. Despite the abundance of reported reactions carried out in silica gel in the literature, it is surprising that little is known about using sand as the medium for solid support, despite both have the same chemical formula (SiO2), degree of crystallinity, and porosity but different price. Herein, we report some of our results regarding the oxidation of selected primary and secondary alcohols by co-grinding (NH4)2Cr2O7 with washed sand. Using toluene or acetonitrile, primary and secondary alcohols are oxidized cleanly into aldehydes and ketones respectively based on TLC monitoring and 1H and 13C-NMR of the crude material. Current progress focuses on preparing aldehydes and ketones using the developed reaction and comparison to silica gel-supported oxidation reactions.

Disciplines
Organic Chemistry
Investigating Oxidation of Alcohols with (NH₄)₂Cr₂O₇ in Washed Sand
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Oxidation of primary and secondary alcohols produces aldehydes and ketones for functionalization and application to medicinal chemistry and pharmacology.

Background

Method Development: Data and Results

PART ONE: Oxidation of primary alcohols
To identify the best reaction conditions needed to oxidize primary alcohols to aldehydes, anisyl alcohol was chosen. Experiments were performed in 100 mg scale and reaction monitoring were done using Thin Layer Chromatography and ¹H-NMR Spectroscopy.

Why Sand? SiO₂ : silica gel vs. sand
- Sand is cheaper ($32.00/500 g – Fisher) than silica gel ($698.00/500g).
- Sand is less harmful than silica gel (irritant).
- Sand is abundant in nature and has many practical uses (geology and construction).
- Sand does not affect separation of mixtures in column chromatography unlike silica gel.
- Sand is not known in organic synthesis unlike silica gel which makes it very attractive to exploit and utilize in various chemistries.

Reaction Design

Grind (NH₄)₂Cr₂O₇ (orange) and washed sand to produce yellow mixture for 15 min
Add the alcohol to the ground Cr-SiO₂ in a flame-dried flask with solvent for reflux
Purify crude material from reflux by column chromatography

Current Progress

Oxidation of alcohols that are relevant in medicinal chemistry and biochemistry – results pending (NMR and percent yield)

Future Work
- Creation of small library of aldehydes and ketones for further use (organometallic chemistry)
- Investigating the role of sand towards interaction with another metal at the surface

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PREVIOUS WORK: Cr-based oxidation of alcohols in the presence of silica gel

CURRENT WORK: Explore Cr-based oxidation of alcohols using washed sand

PART TWO: Oxidation of secondary alcohols
Benzhydrol was used to react with (NH₄)₂Cr₂O₇ and sand to give the product, benzophenone. Experiments were also performed in 100 mg scale and reaction monitoring were done using Thin Layer Chromatography and ¹H and ¹³C-NMR Spectroscopy to check the disappearance of methine peak and the formation of carbonyl peak.