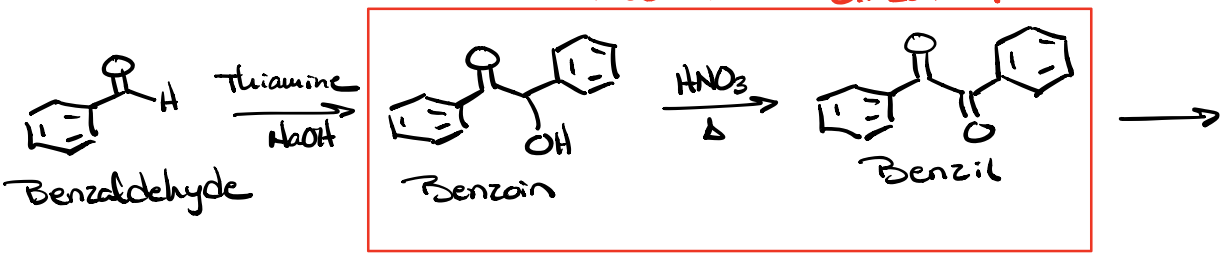
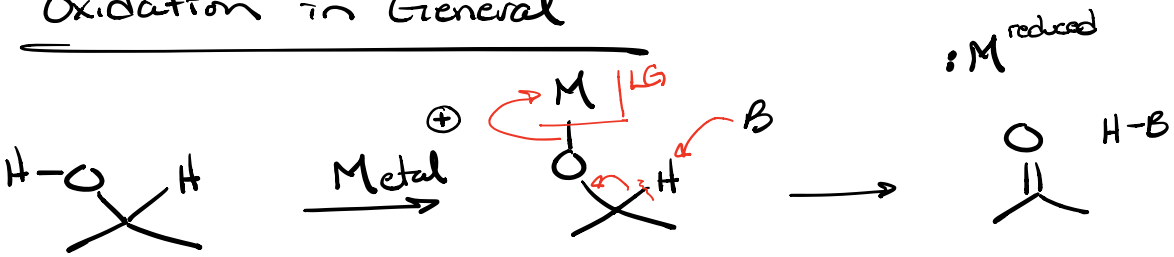
Reaction #2 Multistep Synthesis of Benzilic Acid

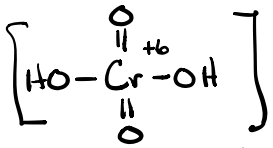
Reaction #2 Oxidation



Oxidation in General



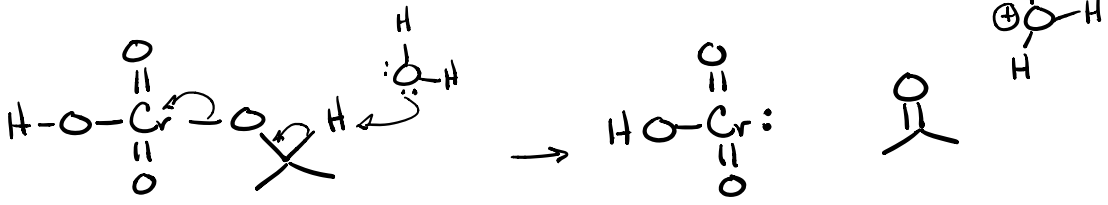
Jones Reagent



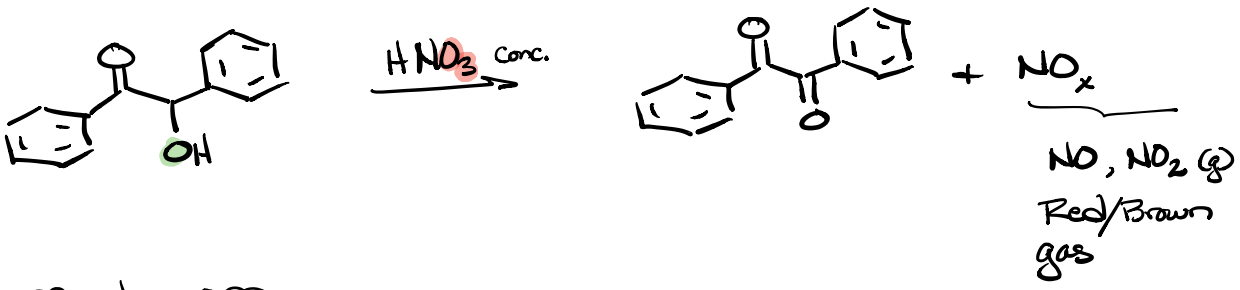
Chromic acid

Hexavalent Chromium

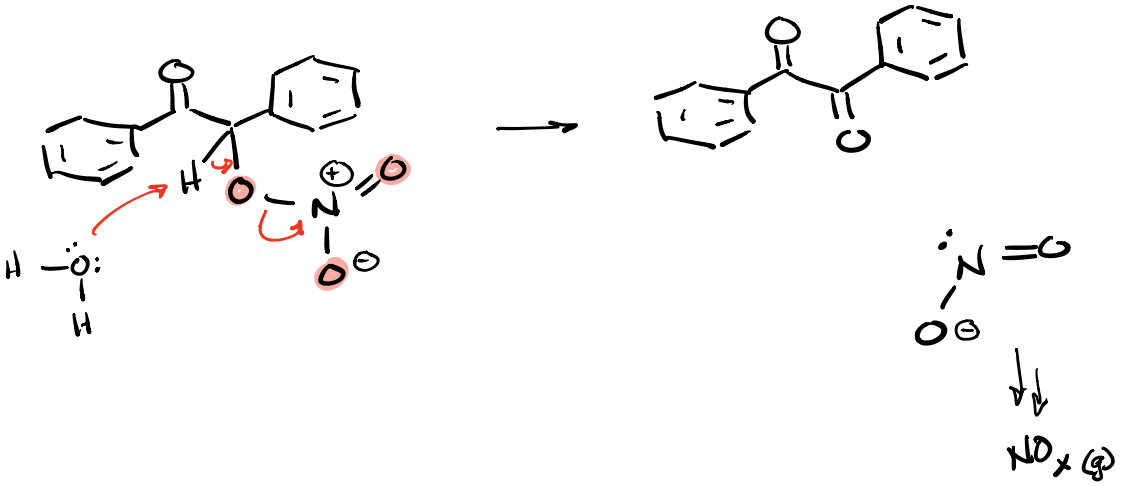
Cr^{6+} known Carcinogen



Our Reaction uses Nitric Acid

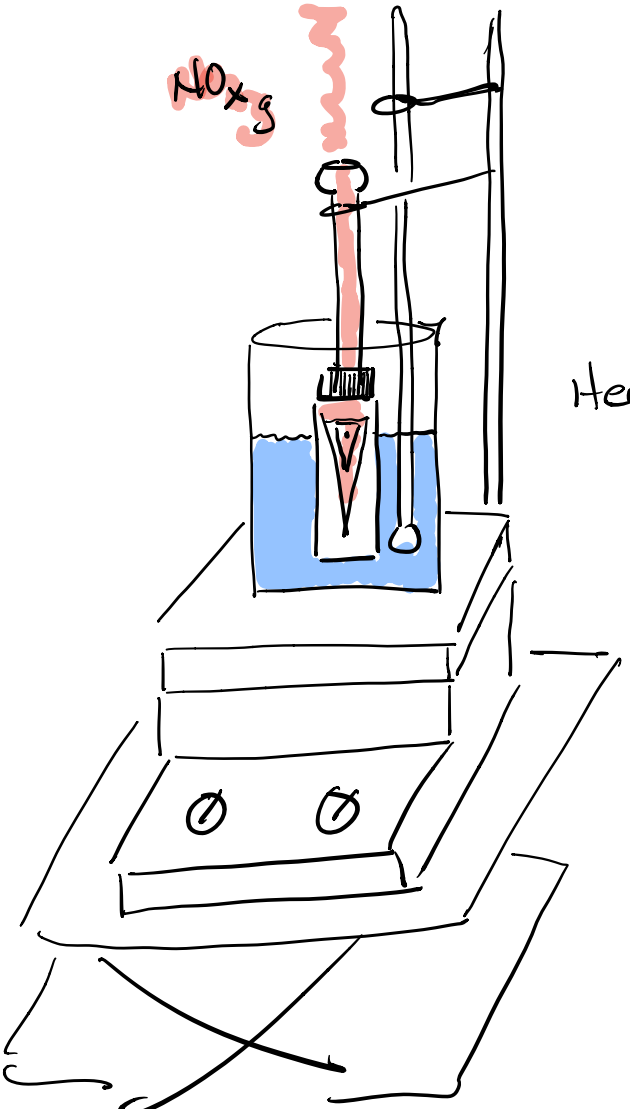


Mechanism



Procedure

- 0.3 g Benzoin in a 5-mL Conical Vial
- Add Spin vane & air Condenser
- Set-up hot water bath in fume hood @ 70°C ± 5°C
- Heat Reaction w/ Stirring for 1 hr 15 min



Work-up

- Cool rxn to room temp
- Disconnect air condenser in fume hood
- Transfer Reaction contents into a beaker w/ 4.0 mL 0°C DI H_2O.
- Rinse Spin Vane & Conical vial with DI & add to the beaker
- Ice bath the beaker for 15 min
- Filter on Hirsch funnel & Rinse with ~5mL 0°C DI H_2O
- Recrystallize from 95% EtOH
- % yield
- MP
- FTIR

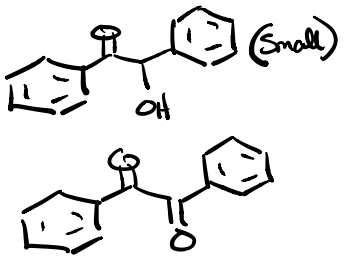
Separation Scheme

HNO_3 , HNO_x

H_2O

Solid
org

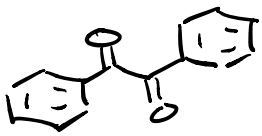
liquid
aq
H_2O
HNO_3
HNO_x

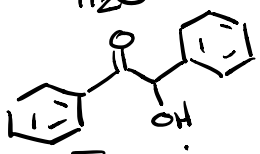


95% EtOH

Solid

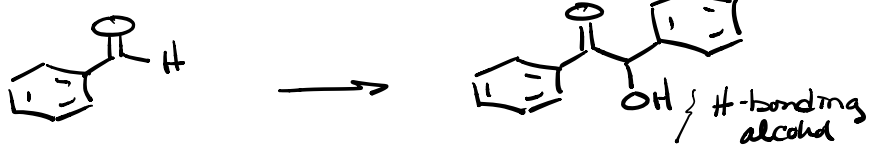
liquid
EtOH
H_2O





Benzoin

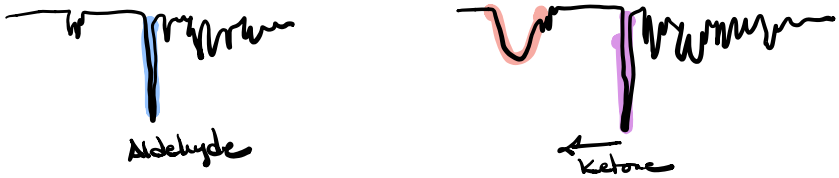
① Benzaldehyde → Benzoin



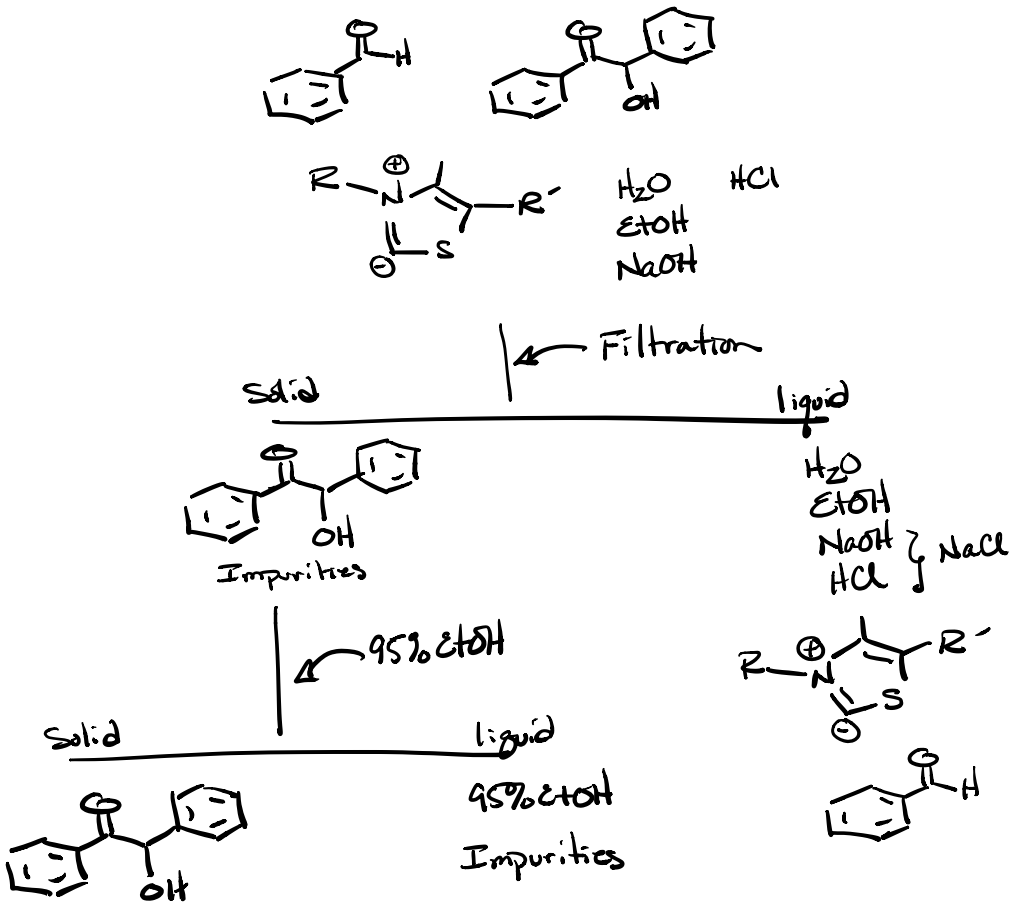
What do we expect to see:

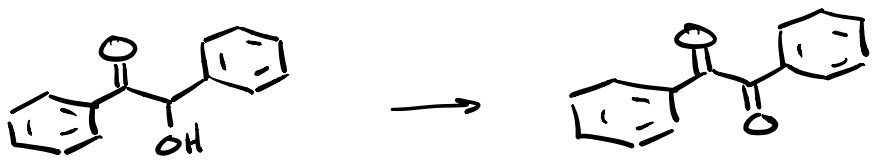
MP
- dipole-dipole, liquid @ RT
- 2x mass + H-bonding, solid w/ high MP (~137 °C)

FTIR
- Carbonyl ~1700 cm⁻¹, aldehyde
- Carbonyl + hydroxyl, R-OH



Separation Scheme for Benzoin Reaction





Benzoin → Benzil

What do we expect to see:

MP

Higher MP
137°C
H-Bonding

Low MP
98°C
dipole-dipole

FTIR Carbonyl + hydroxyl

R-OH

Loss of 3400 cm^{-1} alcohol & shift of the ketone