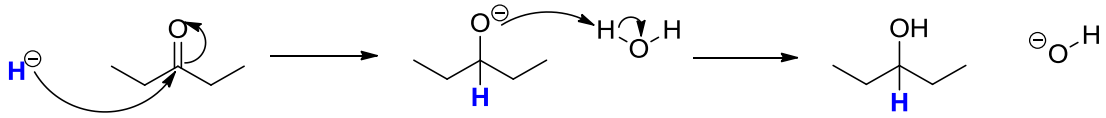
Reduction of Benzil

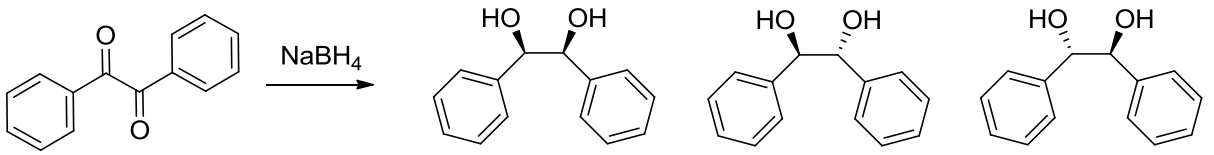
Background

Sodium borohydride is a common reducing agent in organic chemistry. It converts ketones to secondary alcohols and aldehydes to primary alcohols.

The effective nucleophile in the reaction is "H^-" which attacks the carbonyl carbon. Note for the stoichiometry, one equivalent of sodium borohydride can deliver up to 4 equivalents of "H^-". The mechanism below is a simplification. After the nucleophile attacks, the alkoxide formed is protonated by water to form the alcohol product.



When benzil reacts with sodium borohydride it can form three stereoisomers. You will use melting points to determine if you have the meso compound or the enanatiomeric pair.



meso hydrobenzoin
mpt 137-139°C

1R,2R hydrobenzoin
mpt 120°C

1S,2S hydrobenzoin
mpt 120°C

Procedure

Reaction Setup

Add 0.5 g of benzil and 5 mL of 95% ethanol to a 50 mL Erlenmeyer flask. Swirl flask and briefly cool in an ice bath. Add 0.1 g of sodium borohydride to the reaction flask. Allow to react for 10 minutes at room temperature, swirling occasionally.

Isolation and Purification

Add 5 mL of water to the flask and heat to just boiling. If the reaction is not clear, perform hot gravity filtration. Add an additional 10 mL of water to the flask allow to crystallize slowly, first at room temperature, then in an ice bath. Collect the crystals with vacuum filtration. Allow to dry until the next class before taking a melting point.

Characterization

Take an IR of both the starting material and product. Take a melting point of your product. Calculate percent yield.

Chemicals: Sodium borohydride, 95% ethanol, benzil.

Waste: Pour filtrate in the liquid waste jar for the lab and put solids into designated box.