# Crossed Aldol Condensation

**CHEM HELP *ASAP***

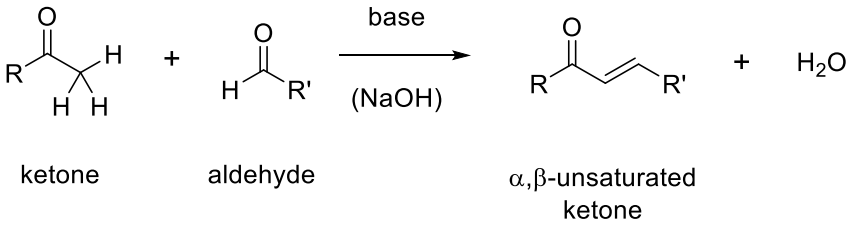
**experiment video:** https://youtu.be/c7dr3bLgZKQ

## Purpose

This experiment demonstrates the aldol condensation. This is a classic reaction that has been well understood for decades and is still very widely used today.

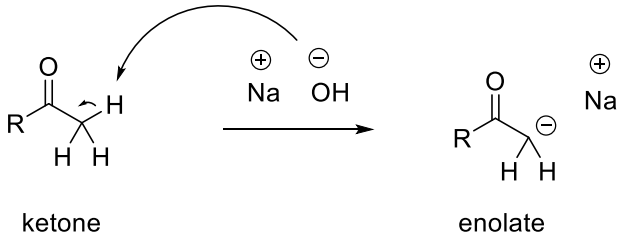
## Background

The *aldol condensation* is one of the most important reactions in organic chemistry. The reaction is also vital to the processing of fats in all living organisms. In the laboratory, the aldol condensation is often performed as a reaction between a *ketone* and an *aldehyde* in a strong base (Scheme 1). The reaction forms a product that is called an α,β-*unsaturated ketone* with water as a side product. Two hydrogens are lost from the ketone, and the carbonyl oxygen is lost from the aldehyde. The aldol condensation does have limitations and works best when the condensation product precipitates from the reaction as it forms.



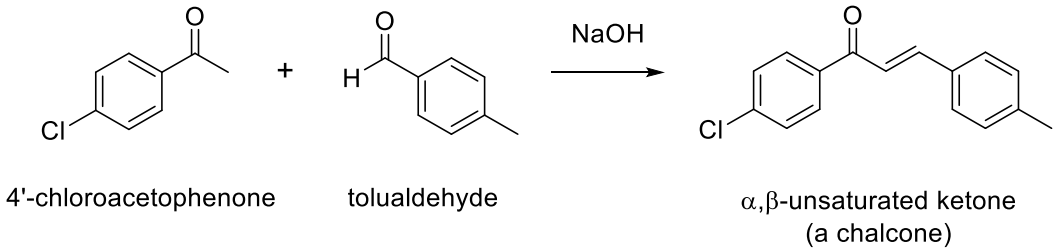

**Scheme 1.** The aldol condensation reaction

The enolate formed by deprotonation of an α-proton relative to the ketone is stabilized by resonance with the adjacent carbonyl pi-bond (Scheme 2). Enolates are very important intermediates and play a role in aldol reactions as well as many other reactions.




**Scheme 2.** Formation of an enolate

This aldol experiment involves 4'-chloroacetophenone (the ketone) and tolualdehyde (the aldehyde) (Scheme 3). The reagents form an α,β-unsaturated ketone product with benzene rings on opposite sides of the molecule. α,β-Unsaturated ketones with this structure are called *chalcones*. Chalcones readily precipitate from polar solvents. They appeared in the chemical literature all the way back into the 1800s.




**Scheme 3.** Today's reaction scheme

## Procedure - (*E*)-1-(4-chlorophenyl)-3-(4-methylphenyl)prop-2-en-1-one

Procedure adapted from … Kulp, S. S. "Knoevenagel Condensation to $\alpha$-Phenylcinnamonitriles: $NaBH_4$ Reduction to Propanenitriles" *J. Chem. Ed.* **1988**, *65*, 742.

Mix methanol (5 mL), 4-chloroacetophenone (1.5 mmol), and tolualdehyde (1.5 mmol) in a 20-mL scintillation vial. Add 40% NaOH (15 drops) with a pipet and immediately swirl the vial to mix the reaction. Heat the reaction in a bath of warm water for 30 minutes. [Get warm water from the sink. Do **not** use a hot plate to heat the water.] Within a few minutes, the reaction should contain precipitate. After about 5 minutes, use a microspatula to break up any solid if necessary, and let the reaction continuing standing. After 30 minutes, add water (10 mL), swirl the mixture, and let the reaction stand for another 5 minutes at room temperature. Filter the reaction with a Buchner funnel with a 50-mL side-arm Erlenmeyer flask. Be certain to seat the filter paper with water and have the vacuum on before you start your filtration. Use small amounts of water as needed to rinse the product out of the vial. Do not add enough water to over fill your side-arm beaker (and suck water into the house vacuum). While the filtration is continuing, tare a watch glass labeled with your initials. Record the tare weight in your notebook. Turn off the vacuum, and scrape your product from the funnel onto your watch glass. If the paper sticks to your product, transfer the paper too. Leave your product to air dry. The filtered liquid should be disposed as lab waste. Once the product is dry, determine the mass of the product. Remove the filter paper if needed. Determine your percent yield. Determine the melting point (melting range – both upper and lower limits) of the product. Take a TLC of the product using 15% ethyl acetate/85% hexane as the mobile phase. Once you finish your TLC, dispose your mobile phase in a waste jug. Interpret the provided NMR spectrum. Record all your observations in your notebook.