# Alkylation of an Enolate Anion

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## Aims

The experiment aims to synthesise Ethyl-2-benzyl-3-oxobutanoate via SN2 substitution.

## Introduction

The experiment to be carried out consists of the deprotonation of the β-ketoester 3-oxobutanoate by the base sodium ethoxide in ethanol. This generates an enolate anion (stabilised by keto-enol tautomerism) which is then alkylated by benzyl chloride.

## Experimental Method

Sodium ethoxide (3.4g, 0.04996moles) was added to ethanol(25ml) in a dry 100mL Quickfit flask. A magnetic stirrer bar was added and the solution was stirred until the sodium ethoxide had entirely dissolved in the ethanol. 3-oxobutanoate (6.5g, 6.4mL, 0.00499 moles) was added to the flask. A condenser was placed on the flask and then the flask was placed on an aluminium heating block on a hotplate stirrer. The contents were stirred until the solid had entirely dissolved. Heat was evolved with stirring. When the flask had cooled, benzyl chloride (6.3g, 5.7mL, 0.04977 moles) was added slowly drop wise down the condenser while not letting excess pressure build in the condenser and flask. The flask was then heated and refluxed for 40 minutes. It was the cooled and added to 100mL deionised water. The product was immediately extracted with ethyl acetate (2x50mL). The extracts were combined and washed with deionised water (2x50mL). The solution was then dried over anhydrous magnesium sulphate. The drying agent was filtered off. The solvent was removed using a rotary evaporator. The product was examined using thin layer chromatographic analysis, using ethyl 3-oxobutanoate as a reference and a 9:1 hexane-ethyl acetate solution as eluent. The mass of the product was recorded to be 4.57g.

## Results

The theoretical yield of the product was calculated to be 41.74%. A copy if the TLC analysis is included with the report.

## Discussion and Conclusions

The synthesis of the product was successfully carried out with a percentage yield of 41.74%. This low yield could possibly be contributed to the deprotonation step not being completed for all of the 3-oxobutanoate. This could have been amended by allowing more time for this step before beginning addition of the benzyl chloride. The TLC analysis was carried out and the results indicated that the product was successfully produced and was isolated from the reactants.

## Post Practical Questions

1. Mechanism included
2. Ethyl-2-benzyl-2-methyl-3-oxobutanoate could be synthesised by the same method used to synthesise Ethyl-2-benzyl-3-oxobutanoate (SN2 substitution) followed by a second step in which the Sn2 substitution is again carried out, this time using NaOEt and bromomethane.
3. The product is immediately extracted from the aqueous solution so as to prevent hydrolysis of the product.