# Formation and Use of an Organometallic Reagent

## Aims

This experiment aims to synthesise triphenylmethanol using phenylmagnesium bromide, a gringard reagent and methyl benzoate.

## Introduction

The first step of the experiment is to synthesise phenylmagnesium bromide. This is carried out via the reaction of bromobenzene, an organic halide with magnesium in diethyl ether. The diethyl ether solution serves to complex the magnesium(II) centre., ensuring magnesium is tetrahedral and obeys the octet rule. The Grignard reagent is both a strong base and a strong nucleophile. It will react with all protons more acidic than those found on alkenes and alkanes. It will react readily will water and therefore all apparatus must be kept toally dry for the experiment. The reaction of the phenylmagnesium bromide with methyl benzoate, an ester, is carried out in order to synthesise triphenylmethanol, a tertiary alcohol. Two moles of Grignard reagent is required for every 1 mole of ester. The first mole of Grignard reagent reacts with the ester to yield a ketone. On addition of aquoes acid, the ketone quickly reacts with the second mole of Grignard reagent to form the tertiary alcohol, triphenylmethanol.

## Experimental Method

## Synthesis of phenylmagnesium bromide

Magnesium turnings (0.6g, 0.0247moles) was placed in a dry 100ml round-bottomed flask. Sodium-dried diethyl ether(10ml, 0.0963 moles) was added to the flask and a reflux condenser was fitted to the flask. A portion of bromobenzene( 2.7ml, 4g, 0.0255 moles) was acquired. 1ml of the bromobenzene was added to the reaction flask through the condenser. A small area of bubbling was observed in the flask. The solution became cloudy and heat evolved. A further 10ml diethyl ether was added, followed by the remainder of the bromobenzene. The solution continued to react for 10 minutes, with continued stirring. When bubbling had finished progressing independently, the flask setup was placed on an aluminium heating block and refluxed on a hot plate for 15 minutes. Very little of the magnesium turnings remained unreacted in the flask.

## Synthesis of triphenylmethanol

A solution of methyl benzoate(3.3g, 3ml, 0.0242 moles) in 10ml diethyl ether was added dropwise to the solution without allow pressure to build in the flask/reflux condenser setup. The methyl benzoate solution was added at such a rate to ensure that a gentle reflux was maintained. When addition of methyl benzoate was complete the solution was again refluxed, for 20 minutes. When the solution had cooled, it was poured into dilute sulphuric acid solution(25ml of 10% solution). Some solid remained in the flask. This was dissolved using a mixture of 10ml diethyl ether and 10ml dilute sulphuric acid and added to the rest of the solution. 2 layers formed in the solution, an lower aqoues layer and an upper ether layer which had a light green pigment. The ether layer was isolated and washed using saturated sodium chloride solution(2x 10ml). The solution was dried over MgSO4 granules and the diethyl ether was evaporated using the rotary evaporator to yield asolid product lining the round bottomed flask. The product was recrytallised from ethyl acetate and hexane( roughly in the ratio 1 part ethyl acetate to 2 parts hexane). The recrystallisation yielded 0.26g of white microcrystalline triphenylmethanol. The melting point of the product was found to be in the range of 154-159OC.

## Results

The percentage yield of triphenylmethanol was calculated to be 7.8%.

## Discussion and Conclusions

The product was synthesised, albeit with a very low yield. This could potentially have been improved by more thoroughly refluxing the round bottomed flask, both in the stages before and after addition of the bromobenzene solution.

## Post Practical Questions

(B) Suggest an alternative synthetic route to triphenylmethanol.

Triphenylmethanol could be synthesised by substitution of benzophenone for methyl benzoate. In this scenario the reaction would take place in diethyl ether initially, followed by the addition of an aqoues acid.

(C)

(i)The reaction of phenylmagnesium bromide with benzaldehyde would yield diphenylmethanol.



(ii) The reaction of phenylmagnesium bromide and formaldehyde would yield benzyl alcohol .



(iii) The reaction of phenylmagnesium bromide with carbon dioxide would yield benzoic acid after the addition(and removal) of hydrochloric acid to protonate the benzoate salts and dissolve the magnesium salts.