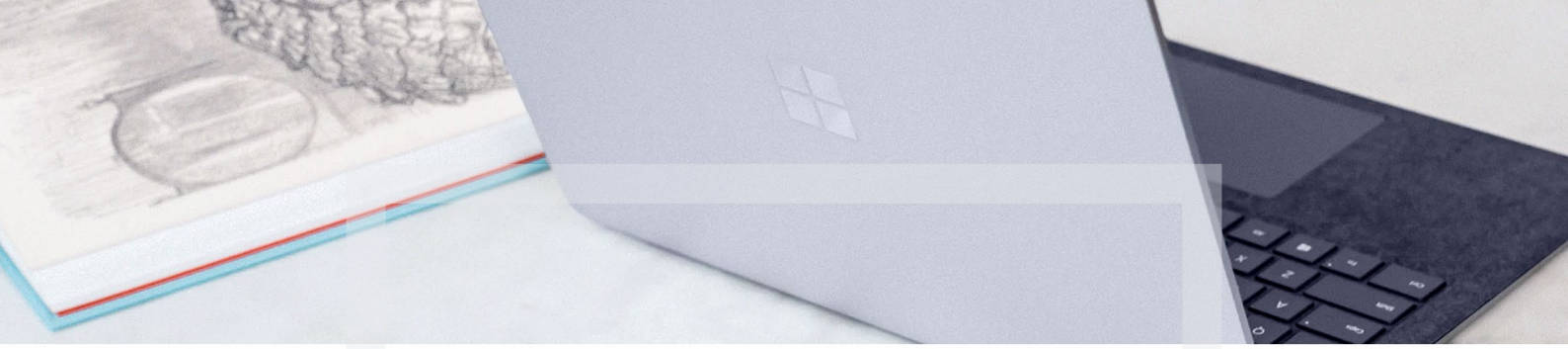
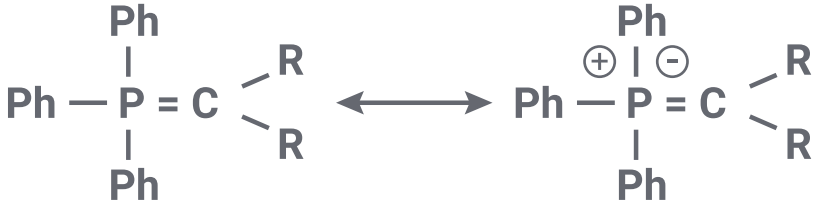


FREE LAB REPORT SAMPLE

Introduction

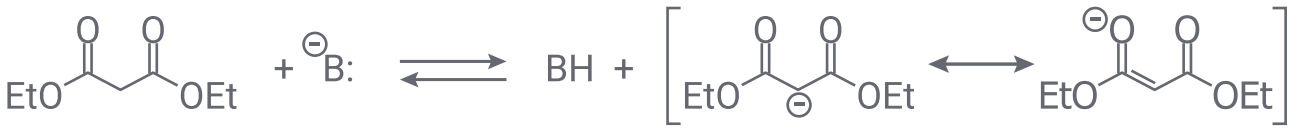
Wittig reaction is a chemical reaction whereby an aldehyde or a ketonethat has a triphenyl phosphonium ylide which acts as the witting reagent giving a product referred to as an alkene. Wittig reactions are mainly applied in coupling of aldehydes and ketones to form ylides (Gusar 1999). The phosphorane form and the ylide form of the wittig reagent can be written as.



phosphorane form ylide form

An Important element in wittig reactions is malonic acid which is a simple carboxylic acid and usually has two of the carboxylic groups held closely together. In the formation of diethyl malonate using malonic acid, during the chemical reaction the hydroxyl group is replaced by and ethoxy group on both the hydroxyl groups (Gusar 1999). Neighboring the two carbonyl groups in the element is a methylene group. According to Alegret et al, the acidity present in the acid can be attributed to the fact that the hydrogen atoms on the carbon that is positioned adjacent to the carbonyl group within the molecule is slightly more acidic as compared as compared to those lying next to the alkyl group. Sung et al in their publication Asian Journal of Organic Chemistry state that the stability of the carbanion is maintained by the carbonyl groups and this results in a proton being removed from the methylene group.

In this experiment, the Diethyl malonate which is a useful building block in the synthesis of organic substances. This can be attributed to the fact that the two hydrogen atoms that are contained within the methylene group are acidic in nature with a pKa 13 and hence make the alkylation of the corresponding anions easier.

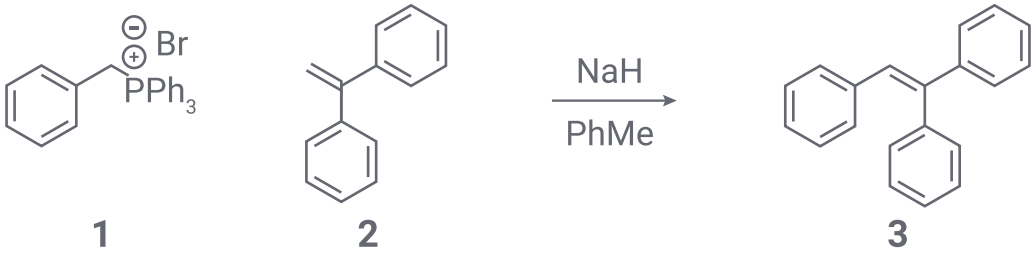


In order to accomplish the task at hand, we shall prepare diethyl butylmalonate using the 'sodiomalonic ester' route and then analyse

each product obtained to determine both the physical and chemical properties.

Aim

The aim of the experiment is to synthesise triphenylethylene (3) through the application of a Wittig reaction of benzophenone (2) and the ylide generated from benzyl triphenylphosphium bromide (2), as depicted in Scheme 1.



1 2 3

Scheme 1

Results and discounting

The experiment on the synthesis of triphenylethylene was conducted and each step followed as outlined in the lab manual. The results of each step were recorded subsequentially. From work done, the following results were obtained:

1. The product obtained was yellow in color and was a crude solid which weighed 2.53g after it was collected and weighed on an electronic scale.

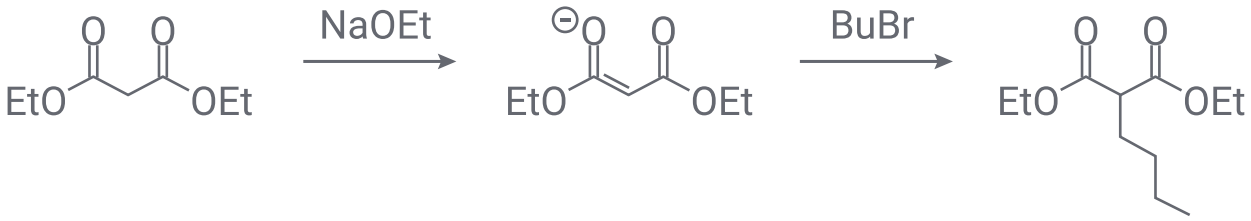
2. The yellow solid was found to crystallize from ethanol as colorless needles which had a weight of 1.87g and 56% of the total weight of the solid. It had a melting point of 71–72 °C.

3. The expected results as available in a written literature are [lit.172–73 °C for the boiling point of the solid obtained].

It was also observed that the boiling point of Diethyl butylmalonate was between 130-135° after running a small fore-run of about 2-3ml of the impure material. The mass of diethyl butlmalomatr is 17.5g. The Boiling point range of Diethyl butylmalonate 164-168. It is important to note that Diethyl malonate is a useful building block in the synthesis of organic substances. This can be attributed to the fact that the two hydrogen atoms that are contained within the methylene group are acidic in nature with a pKa 13 and hence make the alkylation of the corresponding anions easier (Nicolaou, Härter, Gunzner, & Nadin, 1997). When the synthesis of triphenylethylene (3) through the application of a Wittig reaction of benzophenone (2) and the ylide generated from benzyl triphenylphosphium bromide (2) is performed, first the keto carbonyl is converted into a double bond through the chemical reaction and it is then connected to the aromatic groups. Triphenylethylene has a high thermal stability and thus the new product is hence formed at a high temperature of 71–72°C.

Questions

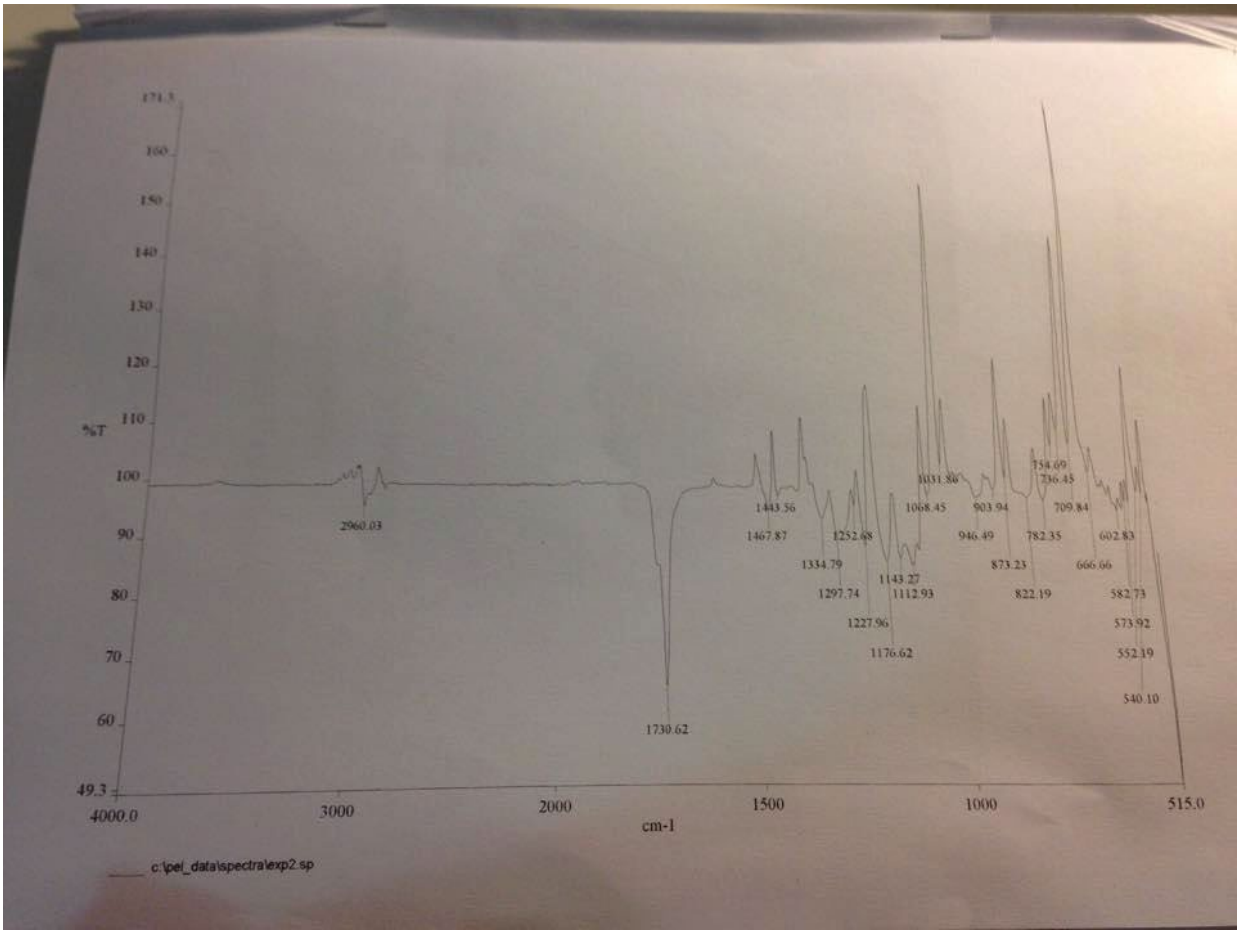
Q1. **Write the mechanism of the reaction.**



Q2. **A possible side-product in the reaction above is diethyl dibutyl malonate. Why is this product formed in insignificant amounts?**

Dibutylmalonate is formed as a side product from the reaction above and the product is formed in some insignificant amounts due to the fact that the temperatures reached are low and below the required temperatures for the formation of Dibutylmalonate. The temperatures reached in the formation of the product obtained is 720C and hence the Dibutylmalonate by product is formed in scanty amounts which may not even be noticeable. Another reason for this occurrence can be attributed to the level of acidity in the reagents (Palacios, Alonso, Aparicio, Rubiales & Santos 2007).

Q3. **Acquire an infrared spectrum of the purified distilled diethyl butylmalonate and identify the carbonyl stretches by annotating the spectrum. Why are there two?**



The spectrum obtained is shown above and the reason why there are two carbons stretches on the spectrum can be attributed to the fact that the regions marked 4000 cm-1-1750 cm-1 and 1750 cm-1-550 cm-1 show absorption and this can be attributed to vibrations that occur in the aromatic ring formed by the carbon-hydrogen atoms forming a bond between them.

Conclusion

In conclusion, the main objective of the experiment which aimed to synthesise triphenylethylene (3) through the application of a Wittig reaction of benzophenone (2) and the ylide generated from benzyl triphenylphosphium bromide (2) was achieved and the physical and chemical properties of the products obtained determined. It was also noted that the level of acidity and the temperatures reached during the experiment determine whether Dibutylmalonate will be formed as a byproduct in significant amounts or not.