Photochemical Reactions of Some Chalcones—Part I

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In the present work, the author reports the photochemical reactions of chalcone in alkaline medium.

INTRODUCTION

The photochemical reactions of chalcones are well studied¹⁻⁷. All these reactions have been carried out in neutral medium. Here we report the photochemical reaction of chalcones in alkaline medium. Chalcone, 4-chlorochalcone and 4'-hydroxychalcone were synthesized by the standard method⁸. These were then irradiated by light separately in alkaline medium. Ir-radiation in alkaline medium gave chalcone dimer II.

Ia:
$$X = H$$
, $Y = H$; Ib: $X = Cl$, $Y = H$; Ic: $X = H$, $Y = OH$

The following mechanism is proposed for dimerization:

or

The structure of the dimer has been confirmed by spectral and elemental analysis. However it is difficult to say whether it is a symmetrical dimer (II) or an usymmetrical dimer (III).

The hydrogen atom at α -carbon is acidic in nature; therefore this should be eliminated under the influence of NaOH rather than that of β -carbon.

The IR spectrum of the product (II) shows carbonyl absorption at 1640 cm $^{-1}$ (same as that of chalcone). UV shows λ_{max} at 320 nm and 240 nm. The R_f value is about 0.2. The mass spectrum gives molecular ion peak at m/e 414 which corresponds to the molecular weight of the dimer. This is a new type of chalcone dimer; the earlier reported dimers have cyclobutane structures $^{7,\,8,\,10}$. However, an analogous dimer IV has been reported by Stobbe and Hensel 9 .

EXPERIMENTAL

X = H, OMe or Cl.

Preparation of chalcones

The chalcones are prepared by the Claisen-Schmidt condensation of the corresponding benzaldehydes and acetophenones in an alkaline alcoholic solution.

Photochemical reactions of chalcones:

(i) Reaction of chalcone: The chalcone (1 gm) was taken in a beaker and dissolved in dried and distilled alcohol (25 ml). The solution was made alkaline by adding 1 ml of 10% NaOH solution. The solution was then put on a magnetic stirrer and irradiated by a 100 W tungsten lamp. The temperature of the solution was maintained by constant water circulation. The progress of the reaction was examined by the TLC of the reaction mixture in benzene after every hour. The reaction was complete in 14 hrs. The solution was then neutralized with HCl and the solid obtained was filtered, washed with water, dried and recrystallized from alcohol. M.pt. 92°C, yield ca. 0.6 g.

Results of elemental analysis: Found: C, 86.65%; H, 5.60%. $C_3H_{22}O_2$ requires C, 86.95%; H, 5.31%.

(ii) Reaction of 4-chlorochalcone: The reaction was carried out in the same manner as in the previous experiment. The reaction completed in 8 hrs. The product obtained was recrystallized from alcohol. M.pt. 120°C, yield ca. 0.75 g.

Results of elemental analysis: Found: C, 74.15% H, 4.24% Cl, 14.96%. $C_{30}H_{20}O_2Cl_2$ requires C, 74.53%; H, 4.14%; Cl, 14.70%.

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(iii) Reaction of 4-hydroxychalcone: The reaction was performed in the same way as in the previous experiment. The reaction completed in 12 hrs. The product obtained was recrystallized from alcohol. M.pt. 178°C, yield ca. 0.70 g. Results of elemental analysis: Found: C, 80.61%; H, 4.85%. C₃₀H₂₂O₄ requires C, 80.71%; H, 4.93%.

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