



Figure 1. Field photo showing the development of unusually large-sized bedding fault. Note the development of bluish-green pseudotachylyte, the remnants of which are seen in the form of patches (outlined). Striations in two different directions are also developed, though they are obscured. View looking north-west. Location: 2 km west of Basidoni village.

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REACTION OF DIBENZOYLMETHANE WITH FORMALDEHYDE: A REVISED STRUCTURE FOR THE PRODUCT

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RECENTLY, Joglekar and Samant¹ reported that 1,3-diphenyl-1,3-propanedione and formaldehyde in

ethanol in presence of different amines gave 2-benzoyl-1,5-diphenyl-1,5-pentanedione (**1**) through base-catalysed cleavage. Spectroscopic data were interpreted and a suitable mechanism for the formation of **1** has also been proposed. However, a critical examination of the PMR spectroscopic data of **1** points out that (i) the methylene (4-CH₂) adjacent to carbonyl appears at considerably downfield (5.70 ppm), which is unusual for this type of protons², and (ii) the absence of a signal corresponding to a methine (2-CH) connected to two benzoyl groups, although such protons were located earlier³.

A literature survey reveals that the PMR data reported for **1** by Joglekar and Samant agree well with those recorded for 2,4-dibenzoyl-1,5-diphenyl-1,5-pentanedione (**2**)^{3,4}.



1 R = H

2 R = COPh

The mass spectrum of **1** was reported¹ to have a peak at *m/z* 356, attributed to the molecular ion. It

is known that compounds of type **2** fragment extensively² at 70 eV and the molecular ions may not be observed.

In view of the above, the reaction was repeated under typical conditions reported¹, using piperidine as the base. A product with the same m.p. and other data reported for **1** was obtained. However, the ¹³C NMR spectrum of the product showed two upfield signals at δ 29.1(*t*) and 54.2(*d*) only, suggesting that the structure of the product is **2** and not **1**. In fact, the mass spectrum recorded in CI mode using methane as ionizing gas gave the ion at *m/z* 461, corresponding to (M + H)⁺ of **2**.

From the foregoing, it is necessary to revise the structure of the product in the title reaction to **2**. It is interesting to note that the reaction of dibenzoylmethane and formaldehyde in ether in presence of diethylamine was reported to give 1,1-dibenzoyl-ethylene⁵, but revised latter to **2**^{3,4}. Formation of **2** was also favoured by Lieberman and Wagner⁶ in both acid- and base-catalysed reactions, but without any spectroscopic evidence.

Experimental

A mixture of 1,3-diphenyl-1,3-propanedione (1.12 g, 0.005 mol), formaldehyde (35% aq. solution, 1 ml, 0.01 mol), piperidine (0.5 ml) and ethanol (20 ml) was heated on a steam bath for 20 min and the reaction mixture was then kept overnight at room temperature. The precipitated solid was recrystallized from ethanol.

Yield: 0.69 g (60%), m.p. 178–180°C. IR (KBr): 1688 and 1668 cm⁻¹. PMR (CDCl₃, 90 MHz): δ 2.78 (*t*, *J* = 7.5 Hz, 2 H), 5.75 (*t*, *J* = 7.5 Hz, 2 H), 7.40–7.60 (*m*, 12 H), 8.05–8.20 (*m*, 4 H). CMR (CDCl₃, 22.5 MHz): δ 196.7 (*s*), 135.7 (*s*), 134.0 (*d*), 129.1 (*d*), 128.9 (*d*), 54.2 (*d*), 29.1 (*t*). Mass (CI/CH₄): *m/z* (%)—461 (22.2), 369 (22.2), 329 (33.0), 322 (28.7), 253 (29.2), 237 (100.0).

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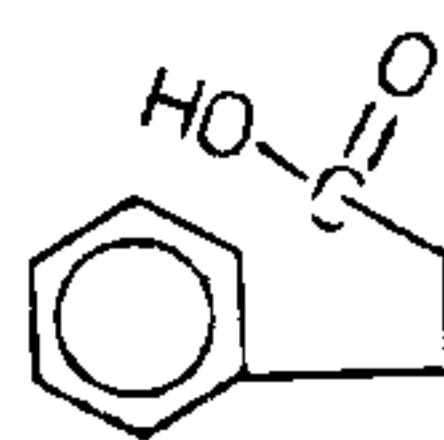
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CONDENSATION PRODUCTS OF β -PHENYLPROPIONIC ACID WITH COMMERCIAL PPA

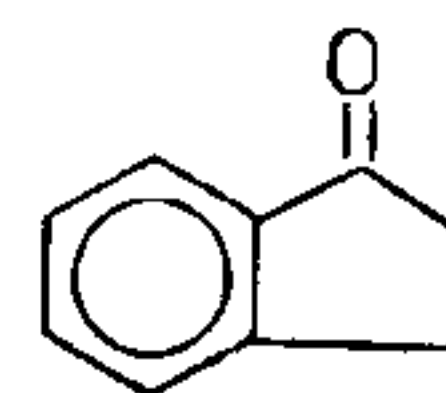
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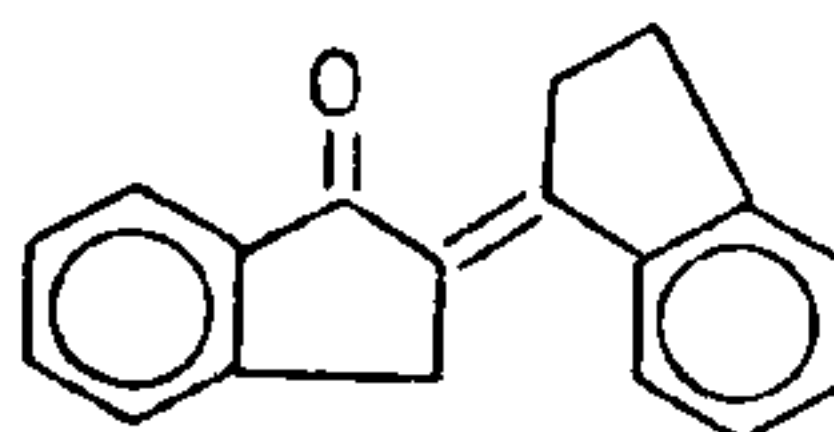
β -PHENYLPROPIONIC acid (**I**) is cyclized to indan-1-one (**II**) when the former is heated with commercial polyphosphoric acid at 70°C for 1.5 h. Indan-1-one is one of the starting materials for the synthesis of coumarins¹ and isocoumarins^{2,3}, two medically important class of compounds. During the preparation of indan-1-one by the literature method¹ it was found that when the temperature is raised to 140°C keeping the reaction mixture well stirred for 3.5 h, a product characterized as anhydrobis-indan-1-one (**III**), is obtained. However, carrying out the



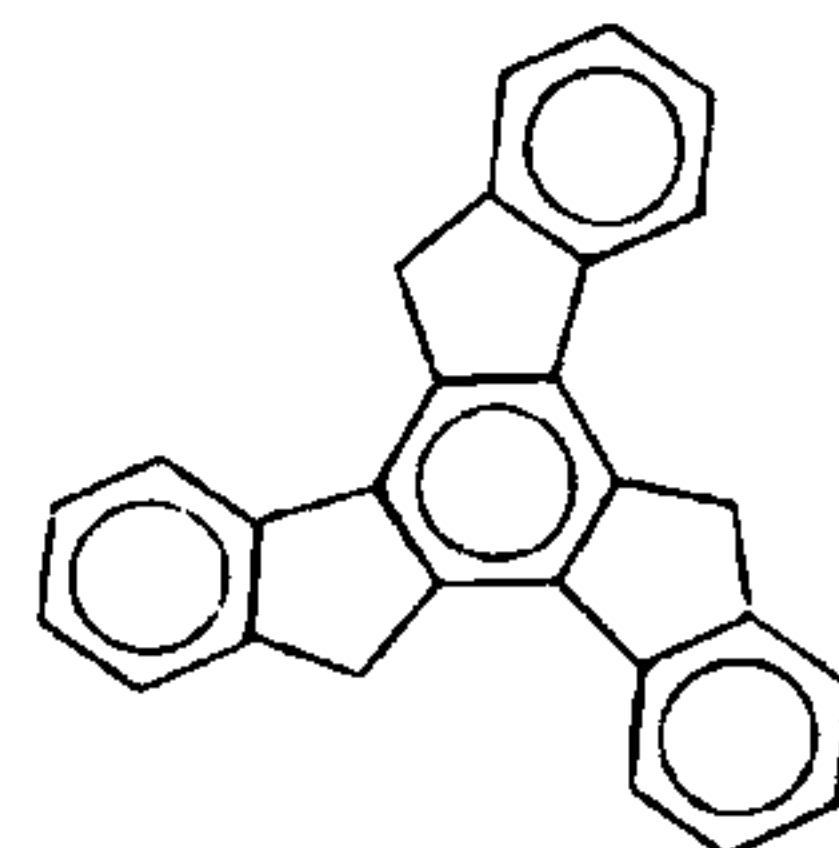
I



II



III



IV