The Adducts of Dimethylketen and 9-Methylacridine

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A RECENT communication[1] describes the evidence for assigning the structure (Ia) to Staudinger's adduct of dimethylketen with acridine.[2] We now wish to report the preparation of a second type of adduct formed by 9-methylacridine and dimethylketen.

Dimethylketen and 9-methylacridine reacted in ether at room temperature to give two isomeric compounds $C_{22}H_{23}NO_2$. One of these [m.p. 117°; ν_{max} 1736, 1699 cm.$^{-1}$ λ_{max} (EtOH) 207, 277 mμ, log ϵ 4·69, 4·20] was identified as (Ib) by the very close similarity of its n.m.r. spectrum to that of (Ia),[1] and the conversion of its dihydro-derivative (Ic) into 9,10-dihydro-10-isobutyryl-9-methylacridine and *N*-benzylisobutyramide on heating with benzylamine. The second adduct [m.p. 183° (decomp.); ν_{max} 1754, 1710 cm.$^{-1}$; λ_{max} (EtOH) 239, 273 mμ, log ϵ 3·92, 3·44] has a completely different n.m.r. spectrum consisting of singlets at τ 8.90 (6 protons), 8·26 (6 protons), and 7·71 (3 protons), with a complex absorption at τ 2·5—3·0 (8 protons). Reduction with lithium aluminium hydride gave a compound (IIIa) $C_{22}H_{27}NO_2$ [m.p. 170°; ν_{max} 3450, 1659 cm^{-1}; n.m.r. spectrum: τ 9·11 (singlet, 6 protons), 8·77 (doublet, $J = 6·5$ c./sec. 6 protons), 8·42 (broad singlet, 1 proton), 8·24 (singlet, 3 protons) 6·70 (septet, $J = 6·5$ c./sec. 1 proton), 6·63 (singlet, 2 protons), 2·3—3 (multiplet, 8 protons)] and heating with benzylamine gave $C_{29}H_{32}N_2O_2$ (IIIb)

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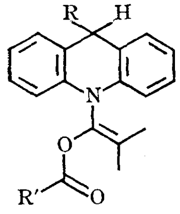
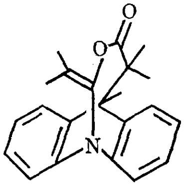
[1] S. A. Procter and G. A. Taylor, *J. Chem. Soc.*, in the press.

[2] H. Staudinger and H. W. Klever, *Ber.*, 1907, **40**, 1149; H. Staudinger, H. W. Klever, and P. Kober, *Annalen*, **1910**, **374**, 1.

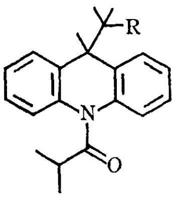
[m.p. 148°; ν_{max} 1679, 1625 cm.$^{-1}$ λ_{max} 251, 279 (sh.) mμ, log ϵ 4·05, 3·38; n.m.r. spectrum: τ 8·88 (singlet, 6 protons), 8·76 (doublet, J = 6·5 c./sec., 6 protons), 8·14 (singlet, 3 protons), 6·78 (septet, J = 6·5 c./sec. 1 proton), 5·73 (doublet, J = 5·5 c./sec. 2 protons), 4·56 (broad triplet, 1 proton), 2·3—3·1 (multiplet, 13 protons).] On the basis of this evidence we assign structure (II) to the second adduct.



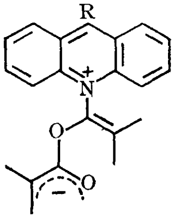
(I) a; R=H, R′=CH_2:CMe
b; R=Me, R′=CH_2:CMe
c; R=Me, R′=Me_2CH



(II)



(III) a; R=CH_2OH
b; R=CONHBz



(IV) a; R=H
b; R=Me

The formation of (Ia) has been suggested[1] as occurring *via* the intermediate (IVa), and it would seem that (Ib) and (II) arise from alternative reactions of the intermediate (IVb), hydride abstraction from the enolate anion giving (Ib) and nucleophilic attack of the anion on the aromatic system giving (II). The competition between hydride abstraction from, and electrophilic attack on the enolate anion has an interesting similarity to the well known competition between nucleophilic attack on and proton abstraction from carbonium ions.

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