Cyclisation of Phenacylisoquinolinium Bromide and Phenacylquinolinium Bromide with Ammonium Acetate in Acetic Acid: a Reinvestigation

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Summary Treatment of the phenacyl quaternary salts of isoquinoline and quinoline with ammonium acetate in acetic acid gave the products (**5**) and (**7**) respectively, and not those previously reported.

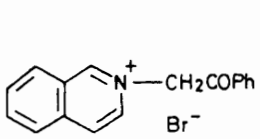
KROEHNKE and ZECHER[1] reported that refluxing the quaternary salts (**1**) and (**2**) in AcOH with an excess of NH_4OAc gave the dihydroimidazo-compounds (**3**) and (**4**) respectively. Re-examination of these reactions has shown that the formulation of these products is incorrect.

The ^1H n.m.r. spectrum ($CDCl_3$) of the product from phenacylisoquinolinium bromide (**1**) displays two 2H triplets (δ 4·15 and 5·04; J 2 Hz) in a pattern characteristic of 1,4-coupling.[2] A singlet (1H, δ 7·15) in the spectrum is lost on bromination. These observations indicate that the product is the imidazo[1,2-*b*]isoquinoline (**5**), formed by cyclisation at the 3-, rather than the 1-position of isoquinoline. Moreover, they account for the failure[1] of attempts to dehydrogenate the putative 2,3-dihydro-compound (**3**) and to dehydrobrominate its supposed 2-bromo-derivative, which must now be reformulated as compound (**6**). The ^{13}C n.m.r. spectrum of compound (**5**) is consistent with its assigned structure.

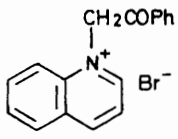
Phenacylquinolinium bromide (**2**) cyclises to give the 4,5-dihydro-compound (**7**) rather than the previously assumed 1,2-dihydro-derivative (**4**). Thus the ^1H n.m.r. spectrum ($CDCl_3$) of the product exhibits a singlet (1H, δ 7·62) which is lost on bromination, and a broad apparent singlet (4H, δ 3·07) which is resolved in C_6D_6 into a symmetrical AA'BB' multiplet.

We thank Dr. A. C. Kende for the determination of the ^{13}C n.m.r. spectrum.

Added in proof: Kroehnke and his co-workers[3] independently agree with our formulation for compound (**7**).



(1)



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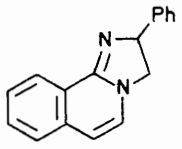

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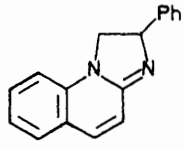
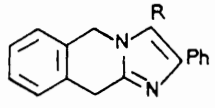
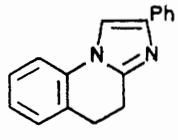


(4)



(5); R = H
(6); R = Br



(7)

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[1] F. Kroehnke and W. Zecher, *Chem. Ber.*, 1962, **95**, 1128.
[2] See for example A. M. Abd-Elfattah, S. M. Hussain, and M. I. Ali, *Tetrahedron*, 1974, **30**, 987.
[3] F. Kroehnke, personal communication.