A New Synthesis of Dihydroimidazo-compounds

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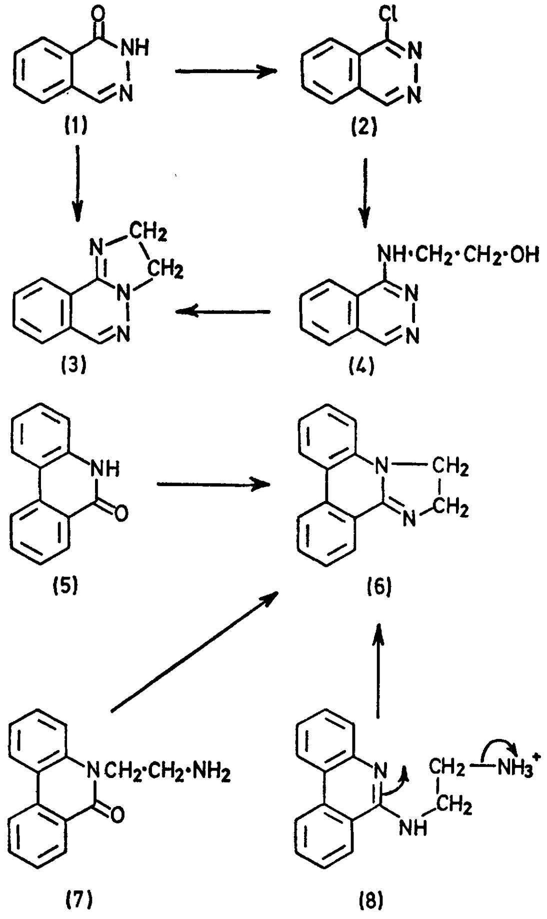
Summary Phenanthridone and phthalazone react with ethylenediamine monotosylate at 200—250° to give dihydroimidazo-fused systems.



In view of the recent report[1] of the reaction of ethylenediamine with a homophthalimide derivative to give a tetrahydroimidazo[2,1-*a*]isoquinoline we report a new method for the preparation of certain dihydroimidazo-compounds. Bremer's preparation[2] of 2,3-dihydroimidazo[1,2-*a*]pyridine has been extended to the quinoline,[3] isoquinoline,[4] phthalazine,[5] and more recently the quinoxaline[6] systems. This procedure involves reaction of an aromatic heterocyclic amide such as phthalazone (**1**) with phosphoryl chloride to give the chloro-heterocycle (**2**), followed by reaction with ethanolamine and sequential treatment of the hydroxyalkylamine (**4**) with thionyl chloride and base to give the cyclised compound (**3**).

During our work,[7] on phenanthridone derivatives we found that treatment of phenanthridone (**5**) with ethylenediamine monotosylate at 200—250° gave high yields of the phenanthridine (**6**), m.p. 129—130°, hydrochloride, m.p. *ca.* 355° (decomp.), which was identical (mixed m.p. and i.r. spectra) with the product obtained on thermal cyclisation of the ethylamine (**7**).[7] As with other phenanthridine derivatives,[7,8] the ^{1}H n.m.r. spectrum of (**6**) [in $(CD_3)_2SO$] shows the aromatic protons as a complex multiplet split into two regions: at δ 8·25—8·55 (3H), presumably due to deshielding, by the C=N group, of 12-H and van der Waals deshielding of 8- and 9-H, and 6·90—8·00 (5H) p.p.m.; the four aliphatic protons appear as a singlet at δ 4·10 p.p.m.

The mechanism of this annelation may involve elimination of ammonia from the intermediate (**8**). 6-(2-Aminoethylamino)phenanthridine (dihydrochloride, decomp. *ca.*

330°) was prepared from 6-chlorophenanthridine and ethylenediamine; thermal decomposition of its monohydrochloride gave the dihydroimidazo-compound (**6**).

We have also shown that treatment of the phthalazone (**1**) with ethylenediamine monotosylate gives (**3**) in one step.

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