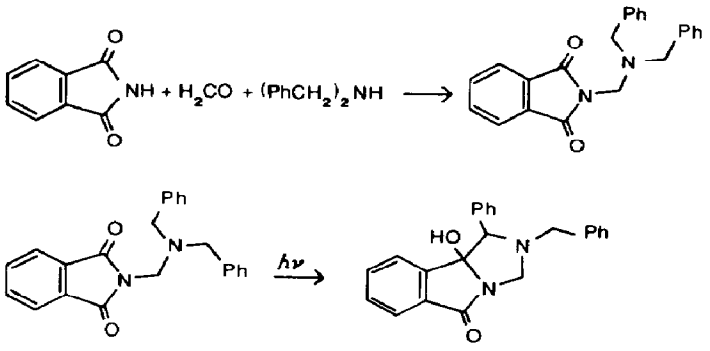
Organic Photochemical Synthesis

A 2,3,5,9b-tetrahydro-1*H*-imidazo[4,3-*a*]isoindol-5-one



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1. Procedure

1.1. N-(Dibenzylaminomethyl)phthalimide

Dibenzylamine (12.4 g, 0.063 mol) is added to a suspension of finely ground phthalimide (9.25 g, 0.063 mol) in aqueous formaldehyde (30 cm^3, 40%), and the resulting mixture is stirred for a few minutes. The filtered product is then recrystallized from ethanol (400 - 500 cm^3) to yield *N*-(dibenzylaminomethyl)phthalimide (19.0 g, 85%) as white crystals (melting point, 147 - 150 °C) (note 1).

1.2. Photocyclization

A solution of *N*-(dibenzylaminomethyl)phthalimide (5.0 g, 0.014 mol) in benzene (350 cm^3) (note 2) is stirred magnetically in a photochemical reactor vessel, and nitrogen is bubbled through the solution. Irradiation is carried out for up to 10 h (note 3) with a centrally immersed 450 W medium pressure mercury arc (Hanovia) which is cooled using a Pyrex water-cooling jacket. After irradiation the solvent is removed under vacuum (note 4). The pale yellow solid is recrystallized from a small volume (5 - 10 cm^3) of ethanol (note 5) to give the photocyclized product, 2-benzyl-9b-hydroxy-1-phenyl-2,3,5,9b-tetrahydro-1*H*-imidazo[4,3-*a*]isoindol-5-one (2.41 g, 48%) as a white powder (melting point, 176 - 178 °C) (note 6). (Spectral data for the photocyclized product are given in ref. 1.)

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2. Notes

(1) The checkers used dibenzylamine and phthalimide from Aldrich Chemical Company and obtained a 94% yield with a melting point of 146 - 147 °C.

(2) Toluene, acetone or acetonitrile can be used as the irradiation solvent, although acetonitrile leads to a crude product which is more difficult to purify.

(3) Overirradiation can lead to problems in the work-up because of increased amounts of a stereoisomer and other products. The lamps we used had been in operation for several hundred hours; with a brand new lamp irradiation for 4 h may be sufficient. The progress of the reaction can be monitored by silica gel thin layer chromatography, using ethyl acetate: petroleum ether (15:85) as eluent. The irradiation is stopped when the starting material spot becomes quite faint but is still just visible.

(4) An oil vacuum pump is needed to remove the final traces of benzene. Recrystallization can be successfully achieved using the semisolid which still contains these traces.

(5) The final recrystallization can be difficult, especially if nitrogen has not been bubbled through the solution during irradiation or if a much more concentrated solution is irradiated. Using the method described here, reasonable yields (29% - 36%) can be achieved by relatively inexperienced undergraduate students.

(6) The checkers obtained a 34% yield with a melting point of 178 - 180 °C.

3. Merits of the preparation

Photochemical cyclization as a result of hydrogen abstraction by a carbonyl group is a very characteristic transformation leading to carbocyclic or heterocyclic alcohols. The example here illustrates the formation of an imidazolidine from a readily synthesized phthalimide.

1 J. D. Coyle and G. L. Newport, *Tetrahedron Lett.*, (1977) 899.

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