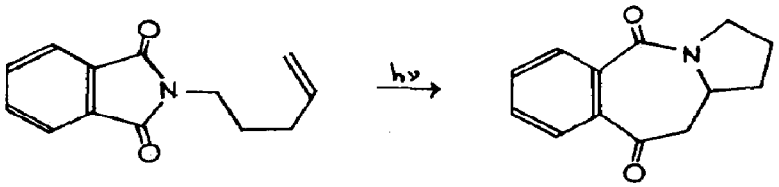
Journal of Photochemistry, *28* (1985) 571 - 572

Organic Photochemical Synthesis

1,2,3,10,11,11b-Hexahydro-5*H*-pyrrolo[1,2-*b*][2]benzazepine-5,10-dione



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1. Procedure

A solution of *N*-(4-pentenyl)phthalimide (3.0 g, 0.014 mol) (note 1) in acetonitrile (400 cm^3) is placed in a photolysis cell equipped with a gas inlet tube, reflux condenser and a water-cooled Pyrex immersion well. The solution is deoxygenated by passing a stream of nitrogen for 30 min, after which the nitrogen flow is decreased to a slow stream. The solution is irradiated with a 300 W high pressure Eikosha mercury arc lamp. After 12 h analysis by thin-layer chromatography (TLC) shows that the starting material has nearly disappeared (note 2). Removal of the solvent on a rotary evaporator leaves a yellowish semisolid residue which is dissolved in chloroform (10 cm^3) and chromatographed over silica gel (20 g, 200 mesh) (note 3). Elution with chloroform (200 cm^3) gives 280 mg (9%) of recovered starting material. Subsequent elution with 5% methanol–ether (360 cm^3) gives 2.39 g of 1,2,3,-10,11,11b-hexahydro-5*H*-pyrrolo[1,2-*b*][2]benzazepine-5,10-dione (88%, based on consumed imide) (note 4). Recrystallization of the product from ethyl acetate–hexane gives white crystals (melting point, 129.5 - 130 °C) (note 5).

2. Notes

(1) *N*-(4-Pentenyl)phthalimide was prepared by the method of Kirmse and Grassmann [1].

(2) The reaction can be monitored by TLC (Merck silica gel HF_{254}-type 60). With ether as the developing solvent, the R_f value of the starting material is 0.8 and that of the product is 0.4.

(3) Silica gel (Wakogel C-200) is supplied by Wako Jyunyaku Kogyo Company, Osaka, Japan.

(4) Twelve fractions each containing 30 cm^3 of the eluant are collected and examined by TLC. Four fractions containing the product are combined

0047-2670/85/$3.30

and evaporated. Soon after the solvent is removed a mixture of ether–hexane (5 - 10 cm^3) is added to the residue and the product crystallizes. The product obtained is in an almost pure form, and the yield is 78 - 92% from several runs, based on consumed imide.

(5) The melting point is measured with a Yanagimoto micromelting point apparatus and is uncorrected. The IR, nuclear magnetic resonance and mass spectra of the product have been reported [2].

1 W. Kirmse and D. Grassmann, *Chem. Ber.*, *99* (1966) 1746.
2 K. Maruyama and Y. Kubo, *Chem. Lett.*, (1978) 769.

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