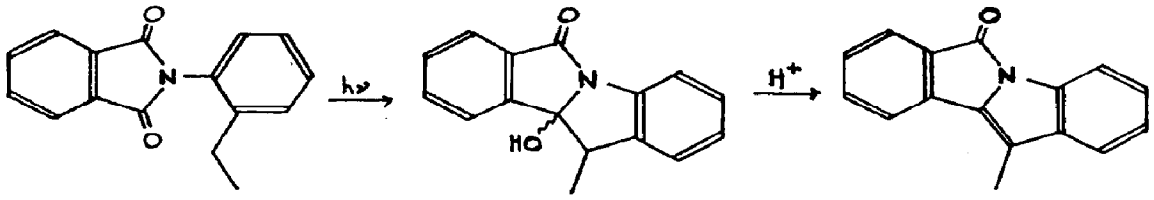
Organic Photochemical Synthesis

11-Methyl-6*H*-isoindolo[2,1-*a*]indol-6-one



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1. Procedure

A solution of *N*-(2-ethylphenyl)phthalimide (6.28 g, 25 mmol) (note 1) in reagent grade acetone (500 cm^3) is irradiated in a water-cooled quartz immersion well (note 2) under nitrogen for 10 h (note 3). The solvent is removed on a rotary evaporator under reduced pressure at approximately 30 °C and the residual yellow solid is recrystallized from ethanol (250 cm^3) containing concentrated hydrochloric acid (5 cm^3) (note 4) to provide 11-methyl-6*H*-isoindolo[2,1-*a*]indol-6-one as yellow needles (melting point, 174 - 177 °C; 3.75 g, 64%).

2. Notes

(1) The *N*-(2-ethylphenyl)phthalimide is prepared from phthalic anhydride and 2-ethylaniline by adaptation of the procedure for synthesizing phthalimide [1].

(2) An Eikosha 500 W high pressure mercury lamp, Eikosha Co., Osaka, Japan was used. The use of a Pyrex immersion well prolonged considerably the reaction time.

(3) Disappearance of the imide is monitored by thin-layer chromatography using silica gel (Merck GF 254) with benzene–ethanol (10:1) as eluant. The reaction becomes slow after 8 h, though some of the imide still remains. *t*-Butanol or acetonitrile can also be used as solvent.

(4) Addition of the acid causes a reddish brown colour to develop.

3. Merits of the preparation

This procedure is based on that originally published by Kanaoka and Koyama [2], and is perhaps the most convenient method for the synthesis of this ring system [3].

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1 W. A. Noyes and P. K. Porter, *Org. Synth., Coll., 1* (1941) 457.
2 Y. Kanaoka and K. Koyama, *Tetrahedron Lett.*, (1972) 4517.
3 Y. Kanaoka, C. Nagasawa, H. Nakai, Y. Sato, H. Ogiwara and T. Mizoguchi, *Heterocycles, 3* (1975) 553.

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