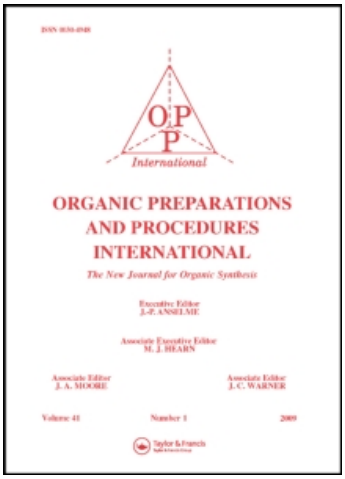

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11 H-11-OXO-5,10-DIPHENYLBENZO[b]FLUORENE FROM THE REACTION OF BENZYNE WITH INDANOCYCLONE

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11 H-11-OXO-5,10-DIPHENYLBENZO[b]FLUORENE

FROM THE REACTION OF BENZYNE WITH INDANOCYCLONE

Submitted (3/9/79)

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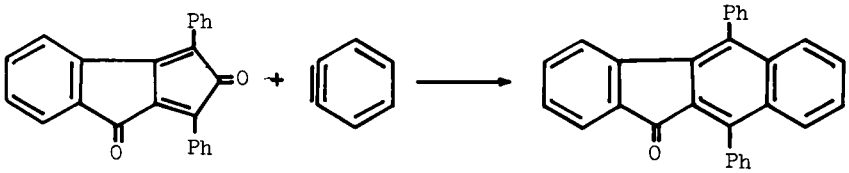
11 H-11-Oxo-5,10-diphenylbenzo[b]fluorene (II) has been reported as a minor product (8.7%) of the reaction of diphenylketene with 2-benzylidene-1,3-indanedione.[1] The same compound was also obtained in 48% overall yield from the reaction of 1,3-diphenylisobenzofuran with ethyl cinnamate, followed by treatment with HI in phenol and subsequent oxidation of the resulting 5,10-diphenyl-11H-benzo[b]fluorene.[2] The reaction of benzyne with tetracyclones is a well documented reaction[3] and with indanocyclone (I), gave II in 75% yield.



I II

EXPERIMENTAL

Propylene oxide (5 ml) was added as an acid scavenger to a refluxing solution of 1,3-diphenylcyclopenta[a]indene-2,8-dione[4] (I) (835 mg, 2.5 mmol) and benzenediazonium-2-carboxylatehydrochloride (560 mg, 3.0 mmol) in ethylene chloride (20 ml). The mixture was refluxed for 3 hrs. and solvent was removed. The residue was extracted with methylene chloride and the organic layer was washed with water and dried over anhydrous magnesium sulfate. The solvent was removed and the crude product was recrystallized (ethanol) to afford 708 mg. (75%) of II, mp. 202-205°, lit.[2] mp. 208°.

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STUDIES OF COMPOUNDS RELATED TO AZINES. 11. THE REACTION OF DIETHYL PHENACYLPHOSPHATE KETAZINES WITH 2,2'-DIFORMYLBIPHENYL

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The reaction of I with II gives polyaza cyclic compounds IIIb (1.4%) and IVa (7.3%) and IVb (29.9%).