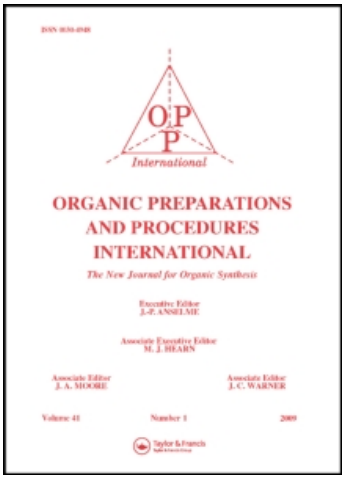

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STUDIES OF COMPOUNDS RELATED TO AZINES. 11. THE REACTION OF DIETHYL PHENACYLPHOSPHATE KETAZINES WITH 2,2-DIFORMYLBIPHENYL

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

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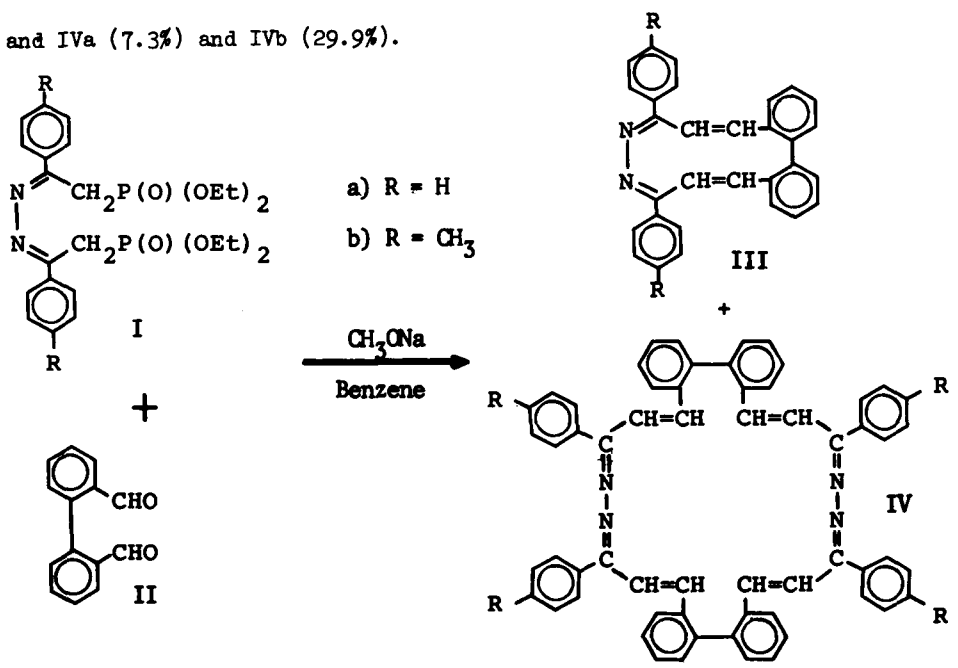
The reaction of I with II gives polyaza cyclic compounds IIIb (1.4%) and IVa (7.3%) and IVb (29.9%).



EXPERIMENTAL

All melting points are uncorrected. NMR spectra were determined at 60 MHz with a Hitachi R-20 NMR spectrometer with TMS as an internal reference. Mass spectra were obtained on Hitachi RMS-4 mass spectrometer with a direct inlet (ionization energy 70 eV).

The Reaction of Ia with II.- To a solution of 2.54 g (5 mmol) of Ia and 1.09 g (5 mmol) of II in 100 ml of benzene was gradually added at room temperature a solution of sodium methoxide (20 mmol) in 10 ml of methanol over a period of 15 min. After the reaction mixture was refluxed for 8 hrs, it was cooled to room temperature, washed with 100 ml (x 3) of water, dried over sodium sulfate, and evaporated in vacuo to leave the residue which was chromatographed on silica gel using a mixture of benzene and ethyl acetate (1:1) as an eluent to afford 0.15 g (7.3%) of IVa, as pale yellow crystalline powder, mp. 145-150° (dec). The purification of crude product was carried out by the continuous recrystallization from ether-petroleum ether to afford pale yellow crystals, mp. 156-160° (dec.), which were dried over P_2O_5 at 100° in vacuo for 24 hrs.

Anal. Calcd for $C_{60}H_{44}H_4 \cdot H_2O$: C, 85.89; H, 5.53; N, 6.68.

Found: C, 86.19; H, 5.35; N, 6.20.

Mass m/e: 820 (M^+); NMR ($CDCl_3$): δ 6.6-8.1 (m).

The Reaction of Ib with II.- Similarly 2.66 g of Ib and 1.09 g of II were treated and worked up as described above to afford 30 mg (1.4%) of IIIb and 0.65 g (29.9%) of IVb: IIIb, pale yellow needles ($CHCl_3$-EtOH), mp. 212-215° (dec.). Mass m/e: 438 (M^+). NMR ($CDCl_3$): δ 2.28 (6H, s, CH_3), 6.36 (2H, d, J = 16 Hz), 6.84-7.75 (18H, m).

Anal. Calcd for $C_{32}H_{26}N_2 \cdot 3/4\ H_2O$: C, 85.02; H, 6.13; N, 6.20.

Found: C, 85.05; H, 5.88; N, 6.23.

IVb, yellow crystalline powder (Et_2O-petroleum ether), mp. 145-150° (dec.) Mass m/e: 876 (M^+); NMR (CCl_4): δ 2.1-2.5 (12H, broad), 6.2-8.0 (40H, m,

broad).

Anal. Calcd for $C_{64}H_{52}N_4 \cdot 3/2\ H_2O$: C, 84.98; H, 6.12; N, 6.24.

Found: C, 85.03; H, 5.91; N, 5.79.

PREPARATION OF 3-(9,10-DIHYDRO-1-ANTHRYL)PENTANE-2,4-DIONE

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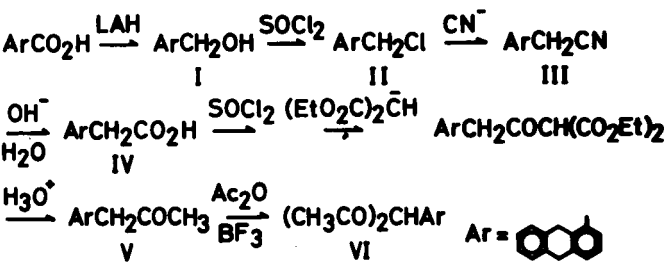
3-(9,10-Dihydro-1-anthryl)pentane-2,4-dione(VI) was obtained by the sequence shown below.



EXPERIMENTAL

Elemental analyses were performed with a Yanagimoto Model MT-1 CHN-corder. Infrared and ^1H NMR spectra were obtained with Hitachi Model EPI-S_2 and Hitachi Model R-20 spectrometers, respectively. Melting points are uncorrected.

1-Hydroxymethyl-9,10-dihydroanthracene(I).- Forty grams (0.19 mole) of 9,10-dihydro-1-anthroic acid[1] was added in small portions to a suspension of 12 g of $LiAlH_4$ in 1.2 l of ether under vigorous stirring. The reaction mixture was stirred and refluxed for 0.5 hr. After addition of water followed by 700 ml. of 10% H_2SO_4, a crystalline solid was obtained from the ether