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SYNTHESIS OF 4-ARYL-5-THIOXO-4,5-DIHYDROINDENO[1,2-b]PYRIDINES

V. K. Lusis, D. Kh. Mutsenietse, and G. Ya. Dubur

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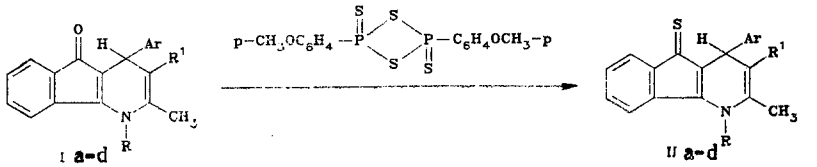
We have found that 4-aryl-5-oxo-4,5-dihydroindeno[1,2-b]pyridines Ia-d react easily with Lawessons's reagent (dimeric sulfide of p-methoxyphenylthionophosphine) to form the hitherto unknown 5-thioxo-derivatives IIa-d; they are not accessible via cyclocondensation [1] since the corresponding thioketones of the indane series are not known so far.



I, II a—c R=H, d R=CH_3; a, b, d R'=$COOC_2H_5$, c R'=CN; a, c, d Ar=C_6H_5, b Ar=$C_6H_4NO_2$-4

5 mmoles of ketone I and 1.01 g (2.5 mmoles) of Lawesson's reagent are refluxed in 250 ml dry benzene for 15-20 min (Ic for 40 min). The solvent is evaporated and the thioketones II isolated by preparative TLC (L 40/100 silica gel, eluent chloroform—hexane—acetone 9:7:1) or column chromatography [2] (L 100/160 silica gel, eluent first benzene then benzene—acetonitrile 10:1). All compounds obtained are dark blue crystalline substances which absorb in the visible region of the spectrum around 370 and 580 nm.

2-Methyl-3-ethoxycarbonyl-4-phenyl-5-thioxo-1H-4,5-dihydroindeno[1,2-b]pyridine (IIa). Yield 55%, mp 158-160° (from ethanol); IR spectrum (nujol): 3295 (NH), 1680 (C=O), 1218 cm^{-1} (C=S). PMR spectrum ($CDCl_3$): 5.11 (s, 1H, 4-H), 6.77 ppm (s, 1H, NH). ^{13}C NMR spectrum (DMSO): 167.9 (C=O), 217.1 ppm (C=S). Mass spectrum: M^+ 361.1139. $C_{22}H_{19}NO_2S$. Calculated: M 361.1136.

In the same way were prepared the thioketones IIb (mp 171-173° from ethanol), IIc (mp 182-184° from chloroform, CN 2203 cm^{-1}), and IId (mp 145° from ethanol; signal of the N—CH_3 group in the PMR spectrum ($CDCl_3$) at 373 ppm).

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