DIHYDROFURO[3,4-b]-3-INDOLONES

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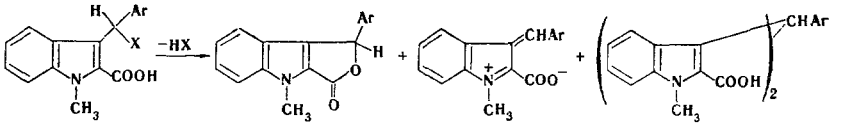
We have observed that the previously unknown lactones of 3-(α-hydroxybenzyl)indole-2-carboxylic acid i.e., 1-aryl-4-methyldihydrofuro[3,4-b]-3-indolones, can be obtained in 10-25% yields by fusion, without access to air, of 1-methyl-2-carbox-3-(α-X-benzyl)indoles (I). Indolenine salts (III) and diindolylphenylmethanes (IV) are formed simultaneously. Thus 0.003 mole of I (X=Cl) was heated in a stream of nitrogen to 130° and allowed to stand at this temperature for 5 min, after which it was cooled and dissolved in chloroform. Compounds IIIa and IVa were separated with a column filled with Al_2O_3. The colorless eluate was washed with 5% Na_2CO_3 solution and water, dried over Na_2SO_4, and evaporated to dryness to give a product with mp 159-160° (from ethanol) in 27% yield. Compound IIb (Ar=pClC_6H_4), with mp 156° and ν_{CO} 1785 cm^{-1}, was similarly obtained in 10% yield. The results of analysis for Cl and N of IIa, b were in agreement with the calculated values.



I IIa, b IIIa IVa

I X=Cl, Br, OH; I, II a Ar=o-ClC_6H_4; b Ar=p-ClC_6H_4

The IR spectra of II contain a band at 1780 cm^{-1}, which is characteristic for γ-lactones. The UV spectra of II coincide with the spectra of starting acid I and their esters and has λ_{max} 300±3 nm (log ε 4.16). The PMR spectrum (CCl_4) of IIa contains an N−CH_3 singlet (3H, 4.0 ppm), a singlet of benzyl protons (1H, 6.8 ppm), and a multiplet of aromatic protons (8H, 7.15 ppm). Lactones II are slowly hydrolyzed in alcoholic alkali to the starting carbinols (I, X=OH). Indolenine salt IIIa (mp 160°) is deeply colored in the crystalline form and in $CHCl_3$ solutions but is colorless in concentrated alkali solutions. It is decolorized on prolonged refluxing in ethanol, since it adds alcohol to give I (X=OC_2H_5). The IR spectrum of IIIa contains a band at 1540 cm^{-1} (carboxylate ion). We were unable to isolate IIIa in analytically pure form free of IVa. Diindolylphenylmethane IVa was found to be identical to a genuine sample.

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