SYNTHESIS OF THE 1H,6H-INDOLO[6,7-g]INDOLE SYSTEM

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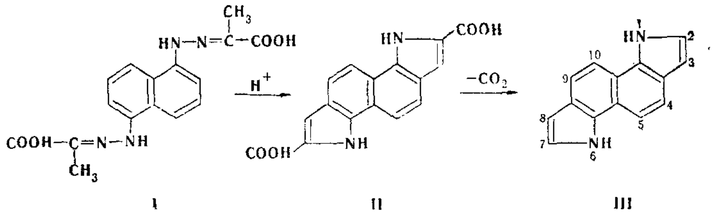
Except for the structural analog of isatin [1], heterocyclic structures formed by condensation of two indole rings through the benzene rings have hitherto been unknown. We have shown that compounds of this series (II and III) can be obtained by Fischer indolization of pyruvic acid 1,5-naphthyldihydrazone (I).



A mixture of concentrated sulfuric and acetic acids was used as the cyclizing agent. The dihydrazone underwent cyclization to give a mixture of geometrical isomers with mp 172-173°C. Thus 1H,6H-indolo[6,7-g]-indole-2,7-dicarboxylic acid (II), with mp 220°C (dec., purified with a column filled with silica gel), was obtained in 30% yield. PMR spectrum [in dimethyl sulfoxide (DMSO)]: ~12.3 (1H), 7.2 (3H), and 7.68 and 8.38 ppm (4-H and 5-H protons). IR spectrum (in mineral oil): 3460 (NH) and 1675 cm^{-1} (C=O). UV spectrum (in ethanol), λ_{max} (log ε): 210.5 (4.30), 232.5 (4.23), 270 (4.55), 282 (4.60), 328 (4.20), 354 (4.11), and 371 nm (4.15). Decarboxylation of II gave 1H,6H-indolo[6,7-g]indole (III), which sublimes satisfactorily at 300-310°C and gives white flocculent crystals with R_f 0.55 [on Silufol in a benzene—acetone system (10:1)]. PMR spectrum (in DMSO): 11.85 (1H), 7.39 (2H), 6.60 (3H), 7.72 (4H), and 8.0 ppm (5H) (the assignments for the 4-H and 5-H protons may possibly be reversed). IR spectrum (in mineral oil): 3410 cm^{-1} (NH). UV spectrum (in ethanol), λ_{max} (log ε): 205 (3.99), 256 (4.68), 265 (4.99), 268 (4.81) shoulder, 284 (3.99), 295 (4.12), 336 (3.52), and 353 nm (3.61). Found: M 206 (by mass spectrometry). Calculated: M 206. The character of the subsequent fragmentation, which is confirmed by metastable transitions, does not contradict the proposed structure. The results of elementary analysis of I-III are in agreement with the calculated values.

LITERATURE CITED

1. British Patent No. 1251082 (1971); Chem. Abstr., 77, 151907 (1972).

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