SYNTHESIS OF 1H-1,4,7,10,13,16-HEXAOXACYCLO-OCTADECAEN-2-[2,3-*f*]INDOLE, A NEW HETEROCYCLIC SYSTEM

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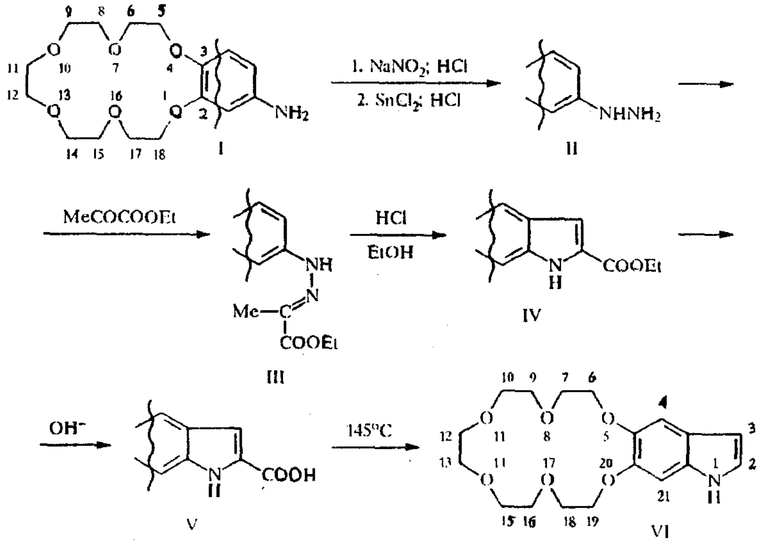
A synthesis is reported for VI, a new heterocyclic compound with crown ether and indole structural fragments.

Diazotization and subsequent reduction of amine I was carried out at −20°C. Cyclization of a mixture of geometrical isomers of hydrazone III obtained by the reaction of hydrazine II with ethyl pyruvate was carried out using 15% ethanolic hydrogen chloride. Ester IV was subjected to alkaline hydrolysis, while acid V was decarboxylated at 140-145°C over 30 min.

The key compounds obtained in carrying out this scheme have the following indices:



1H-2-Ethoxycarbonyl-1,4,7,10,13-hexaoxacyclooctadecaen-2-[2,3-*f*]indole (IV) was obtained in 47% yield, mp 114-115°C, R_f 0.41 (2:1 acetone—benzene). IR spectrum (vaseline oil): 3480 (NH), 1730 (CO, ester), 1130 cm^{-1} (CH_2—O—CH_2). UV spectrum (ethanol), λ_{max} (log ε): 212 (4.65), 323 nm (4.43). PMR spectrum in DMSO-d_6: 11.7 (1H, s, N—H), 6.42 (1H, d, J = 1.48 Hz, 3-H), 6.93 (1H, s, 4-H), 6.91 (1H, s, 21-H), 1.15 (3H, t, $C\underline{H}_3$—CH_2), 4.1 ppm (2H, q, CH_3—$C\underline{H}_2$). The methylene protons of the ether fragment of the ring appear at 3.8-4.06 ppm in the PMR spectrum.

1H-2-Carboxy-1,4,7,10,13,16-hexaoxacyclooctadecaen-2-[2,3-*f*]indole (V) was obtained in 56.8% yield, mp 131-132°C, R_f 0.66 (2:1 acetone—water). IR spectrum (vaseline oil): 3320 (N—H), 1700 (CO, acid), 1130 cm^{-1} (CH_2—O—CH_2). UV spectrum (ethanol), λ_{max} (log ε): 211 (4.40), 309.5 (4.17), 320 (shoulder) nm (4.07).

1H-1,4,7,10,13,16-hexaoxacyclooctadecaen-2-[2,3-*f*]indole (VI) was obtained in 42.8% yield, mp 88-89°C, R_f 0.79 (2:1 acetone—water). IR spectrum (KBr): 3400 (N—H), 1120 cm^{-1} (CH_2—O—CH_2). UV spectrum (ethanol), λ_{max} (log ε): 220 (4.45), 271 (shoulder) (3.80), 298 nm (3.96). PMR spectrum in DMSO-d_6: 10.7 (1H, br.s, N—H), 7.3 (1H, d.d, $J_{1,2}$ = 2.4 Hz, 2-H), 6.3 (1H, m, $J_{2,3}$ = 3.1 Hz), $J_{3,21}$ = 0.6 Hz, 3-H), 7.1 (1H, s, 4-H), 6.9 ppm (1H, d, $J_{3,21}$ = 0.6 Hz, 21-H). The methylene protons of the ether ring fragment are found at 3.57-4.04 ppm in the PMR spectrum.

A mass spectrometric study of IV and VI showed that the molecular ion values corresponded to the calculated values, while the nature of the fragmentation was in accord with the proposed structures.

The elemental analysis data for IV-VI corresponded to the calculated values.

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