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3,6-DIMETHOXY-9H- DIBENZO[a,c]CARBAZOLE

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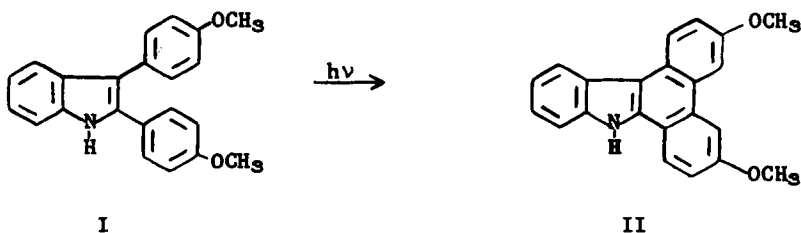
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3,6-DIMETHOXY-9H-DIBENZO[a,c]CARBAZOLE

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In view of the current interest in photodehydrocyclizations^{1,2,3,4,5,6} we would like to report on the easy photochemical conversion of 2,3-bis-(p-methoxyphenyl)indole (I) to the dibenzocarbazole derivative II. Compound I is accessible in one step either by the Fischer-indole synthesis from phenylhydrazine and desoxyanisoin⁷ or by condensation of aniline and anisoin⁷ and, thus, the new polycyclic dibenzocarbazole II becomes accessible in two easy steps.

Experimental

3,6-Dimethoxy-9H-dibenzo[a,c]carbazole A solution of 2,3-bis(p-methoxyphenyl)indole, I (1.4 g; 4.26 mmoles) and 0.65 g of iodine in 170 ml of benzene was placed in a quartz vessel equipped with a Corex filter and irradiated for 24 hr using a 100w 50L Hanovia lamp. The suspension was diluted with benzene and the resulting brown solution was washed with 5% sodium bicarbonate solution (4 x 25 ml), 5% sodium thiosulfate solution and saturated salt solution. The resulting pale yellow solution was dried over magnesium sulfate and evaporated. The oily solid (1.6 g) was suspended in

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10 ml of methylene chloride and the solid was filtered; 0.153 g, mp 203-204° (solid A). The filtrate was chromatographed on 60 g of Florisil collecting 25 ml fractions and using methylene chloride as eluent. Fractions 1-3 gave no material. Fraction 4 (0.46 g) consisted of pure I. Fraction 5 (0.335 g) was a mixture of I and II. Fractions 6, 7 and 8 were crystallized separately from methylene chloride and gave a solid melting at 203-204°. This solid was combined with solid A, dissolved in 10 ml of methylene chloride and 5 ml of methanol, and the solution was concentrated till crystallization began; 0.31 g of II (22% yield), mp 204-205°, unchanged on recrystallization. U.V.: λ_{\max} . 208 (33,550); sh 234 (27,100); λ_{\max} . 268 (62,350); 307 (24,150); sh 324 (18,050); λ_{\max} . 376 (4,100); 396 (4,600). I.R.: NH: 3420; C=C: 1625, 1615, 1585, 1570, 1535, 1510; C-O/C-N: 1285, 1255, 1230, 1190, 1175, 1040; aromatic: 855, 800, 730. Nmr (d_6 DMSO, TMS, 60 mc): two OCH₃ singlets at 242.5 and 245 cps (area 6); aromatic H multiplet at 438-530 cps (area 10); NH singlet at 733 cps (area 1).

Anal. Calcd. for C₂₂H₁₇NO₂: C, 80.71; H, 5.23; N, 4.28. Found: C, 80.41; H, 4.92; N, 4.45.

References

1. F. R. Stermitz in "Organic Photochemistry," Vol. 1, p. 247, O. L. Chapman, Dekker, New York, 1967.
2. H. Güsten and L. Klasinc, *Tetrahedron*, 24, 5499 (1968).
3. H. Jungmann, H. Güsten, and D. Schulte-Frohlinde, *Ber.*, 101, 2699 (1968).
4. W. H. Laarhoven, T. J. H. M. Cuppen and R. J. F. Nivard, *Rec. Trav. Chim.*, 87, 687 (1968).
5. E. Winterfeldt and H. J. Altmann, *Angew. Chem. Intern. Ed. Engl.*, 7, 466 (1968).
6. F. B. Mallory and C. S. Wood, *Org. Syn.*, 45, 91 (1965).
7. J. Szmuszkovicz, E. M. Glenn, R. V. Heinzelman, J. B. Hester, Jr., and G. A. Youngdale, *J. Med. Chem.*, 9, 527 (1966).

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