Nuclear Magnetic Resonance and Stereochemistry of 1,2,3,5,6,7-Hexamethoxy-9,10-diethyl-9,10-dihydroanthracene, a Dimer of 3,4,5-Trimethoxypropenylbenzene

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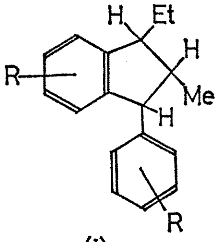
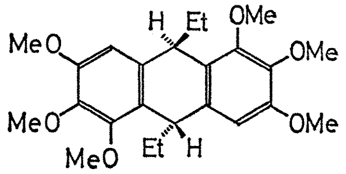
Summary Acid-catalysed dimerisation of 3,4,5-trimethoxypropenylbenzene yields 1,2,3,5,6,7-hexamethoxy-9,10-diethyl-9,10-dihydroanthracene to which the 9,10-*trans*-stereochemistry is allocated from the high-field methyl triplet signal in the n.m.r. spectrum.

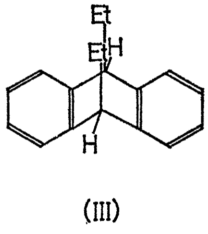
THE formation of tricyclic dimers of propenylbenzenes with acidic reagents is well documented.[1] Although 9,10-dihydroanthracene structures were originally suggested[2] for these dimers, the phenylindane structure (I) was subsequently established for the major products of dimerisation.[1] However Pailer[3] reported the formation of 9,10-diethyl-9,10-dihydro-2,3,6,7-tetramethoxyanthracene from the dimerisation of isoeugenol methyl ether in 0·07% yield. We now report a further example of the acid-catalysed dimerisation of a propenylbenzene to a 9,10-diethyl-9,10-dihydroanthracene and the determination of its stereochemistry.



(I)



(II)



(III)

Di-isoelemicin, obtained[4] in 4% yield by heating isoelemicin (3,4,5-trimethoxypropenylbenzene) in methanolic hydrogen chloride, was kindly provided by Dr. J. F. W. McOmie. The structure (II) follows from high resolution mass spectrometry (M^+ 416·2916, $C_{24}H_{32}O_6$ requires 416·2918), from the u.v. absorption [λ_{max} (hexane) 261 nm. (ϵ 810), $\lambda_{infl.}$ 255 and 271 nm. (ϵ 725 and 642)], and from the n.m.r. spectrum. The latter, determined for a $CDCl_3$ solution at 100 MHz., was distinct from those[1] of the phenylindane dimers (I) and uniquely defined the structure (II). The methyl groups were equivalent, their resonance occurring at τ 9·81 (t, J 7·25 Hz.) As expected from their prochiral environment the methylene protons occurred as two coincident AB-multiplets with J_{AB} 13·5 Hz., centred at τ 8·12 and shown by spin-decoupling to be coupled both to the methyl protons (J 7·25 Hz.) and to a 2H triplet (τ 5·72, J 3·7 Hz.). The latter signal was assigned to the equivalent 9-H and 10-H. The two aromatic protons were equivalent, occurring at τ 3·45 (s, 2H). Of the six methoxy-groups, four coincided at τ 6·16 and two at τ 6·10.

By analogy with the *cis*- and *trans*-isomers of 9,10-diethyl-9,10-dihydro-9,10-dihydroxyanthracene, described in the preceding Communication, it is suggested that di-isoelemicin is the *trans*-isomer (II) with the central ring in a planar conformation. This conclusion agrees with the assignment[5] of the *cis*-stereochemistry and conformation (III) to the major product from the reaction of 9,10-disodioanthracene with ethyl iodide, made on the basis of the splitting pattern of the aryl protons.

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[1] See J. MacMillan, I. L. Martin, and D. J. Morris, *Tetrahedron*, 1969, **25**, 905, for leading references.
[2] G. M. Robinson, *J. Chem. Soc.*, 1915, 269; R. D. Haworth and C. R. Mavin, *ibid.*, 1931, 1363.
[3] M. Pailer, *Monatsh.*, 1947, **77**, 45.
[4] J. A. Godsell, M.Sc. Thesis, University of Bristol, 1954.
[5] D. A. Redford, Ph.D. Thesis, University of Saskatchewan, 1967.