far, prevented isolation; other systems (n = 2, 4, 5, 6) are predicted to be nonplanar, judging from molecular models.

Further work in progress (X-ray diffraction, C^{13} nmr) should provide more information about the properties and character of this molecule.

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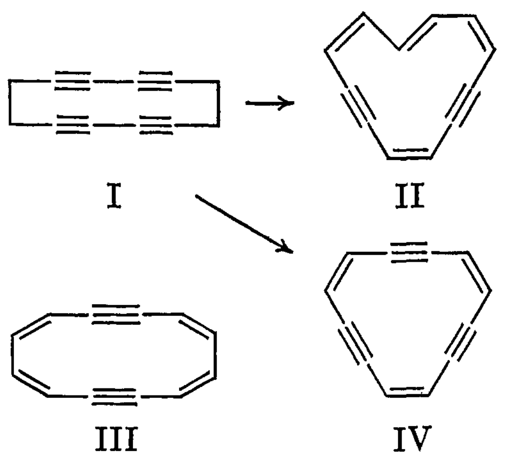
1,5,9-Tridehydro[12]annulene[1]

Sir:

It has been reported by our group[2] that treatment of 1,3,7,9-cyclododecatetrayne (I, $C_{12}H_8$) with potassium *t*-butoxide leads to two different dehydro[12]annulenes. The elemental analysis[2b] of each of these substances indicated the anticipated empirical formula $C_{12}H_8$, and they were therefore both considered to be bisdehydro[12]annulenes. The dehydroannulene obtained in larger amount ("isomer A," complex nmr spectrum) was assigned structure II, while the one obtained in lesser amount ("isomer B," nmr singlet at τ 5.58 in CCl_4)[3] was assigned structure III.



Very recently, Untch and Wysocki[5] have carried out an elegant synthesis of 1,5,9-tridehydro[12]annulene (IV, $C_{12}H_6$)[6] and have observed that this compound possesses properties essentially identical with those reported by us for isomer B. Dr. Untch kindly informed us of his work prior to publication, and we therefore reexamined the structure of isomer B. The mass spectrum was determined (AEI MS9 spectrometer), and the molecular ion (found 150.048) revealed that the empirical formula was in fact $C_{12}H_6$ (calcd 150.047) and not $C_{12}H_8$ (calcd 152.063).[7] Consequently, dehydrogenation must have taken place during the base treatment of I.[8] Isomer B was then shown to be identical with Untch's compound by direct comparison (thin layer chromatography, mass and ultraviolet spectra), and thus is 1,5,9-tridehydro[12]annulene (IV).[10]

The mass spectrum of "isomer A" (molecular ion, found 152.063) clearly showed the $C_{12}H_8$ formula to be correct, and the arguments[2] for assigning structure II to this compound remain unchanged.

Acknowledgment. We wish to thank Dr. K. G. Untch for kindly providing a sample of IV, and to C.S.I.R.O. for an Overseas Postgraduate Studentship (to I.C.C.).

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[1] Part XLI in the series Unsaturated Macrocyclic Compounds. For part XL, see R. Wolovsky and F. Sondheimer, *J. Am. Chem. Soc.*, **88**, 1525 (1966).

[2] (a) F. Sondheimer, *Pure Appl. Chem.*, 7, 363 (1963); (b) R. Wolovsky and F. Sondheimer, *J. Am. Chem. Soc.*, **87**, 5720 (1965).

[3] Whereas the nmr spectrum of isomer A provided confirmation for a $C_{12}H_8$ formulation, such confirmation could not be provided by the spectrum of isomer B. The fact that the latter spectrum showed only a sharp singlet was disturbing (since III possesses two different types of protons), although this type of observation is not without precedent.[4] Separation can occur by change of solvent,[4] but the spectrum of isomer B, determined in different solvents, in all cases exhibited only a singlet ($CDCl_3$, τ 5.52 at 20°, 5.44 at −60°; $(CD_3)_2CO$, 5.34 at 20°, 5.20 at −80°; C_6D_6, 6.00 at 20°).

[4] *Inter alia*, G. Farges and A. S. Dreiding, *Helv. Chim. Acta*, **49**, 552 (1966); F. Sondheimer and P. J. Garratt, unpublished observations.

[5] K. G. Untch and D. C. Wysocki, *J. Am. Chem. Soc.*, **88**, 2608 (1966).

[6] See T. J. Sworski, *J. Chem. Phys.*, **16**, 550 (1948).

[7] The previously reported microanalysis (Found: C, 95.02; H, 5.01.[2b] Calcd for $C_{12}H_8$: C, 94.70; H, 5.30. Calcd for $C_{12}H_6$: C, 95.97; H, 4.03) must have been in error. This points to the inherent danger in assigning an empirical formula merely on the basis of microanalytical data.

[8] Analogous dehydrogenations have already been observed to occur in the C_{14}[2a,9a] and the C_{18} series.[9b]

[9] (a) F. Sondheimer, Y. Gaoni, L. M. Jackman, N. A. Bailey, and R. Mason, *J. Am. Chem. Soc.*, **84**, 4595 (1962); (b) R. Wolovsky, *ibid.*, **87**, 3638 (1965).

[10] It is of interest that the tribenzo analog of IV has been prepared very recently by two groups (H. A. Staab and F. Graf, *Tetrahedron Letters*, 751 (1966); I. D. Campbell, G. Eglinton, W. Henderson, and R. A. Raphael, *Chem. Commun.*, 87 (1966)).

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Electron Spin Resonance Determination of the Heat of Reaction for Triphenylmethyl Radical and Oxygen[1]

Sir:

We wish to report an experimental determination of the heat of reaction for the formation of a peroxy radical from a hydrocarbon radical and oxygen. This value has been obtained for triphenylmethyl radicals trapped in a crystal lattice permeable to oxygen.

Triphenylmethyl radicals were produced in the solid state by radiolysis of triphenylmethyl chloride, triphenylmethyl bromide, or triphenylacetic acid.[2]

$$Ph_3CX \rightsquigarrow PH_3C\cdot \qquad (1)$$

$$X = Cl, Br, COOH$$

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[1] Electron Spin Resonance Studies of Thermal Decomposition Mechanisms. Peroxides. I.

[2] γ-Radiation from cobalt-60 was used. The total dose was 4–10 × 10^{18} ev/g. The dose rate was 5 × 10^{15} ev/g sec^{-1}.