Stereochemistry of 4,5:10,11 bis-(tetramethylene)-4,10-cyclotridecadiene-2,6,8,12-tetrayn-1-one

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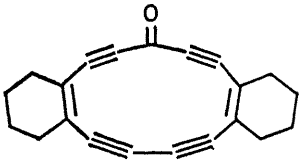
Summary Bond lengths in the title compound have been determined by an X-ray analysis; there is no significant cyclic delocalisation in the molecule.

A NUMBER of macrocyclic conjugated ketones, the annulenones, have been described by Sondheimer *et al.*[1,2,3] The [13]annulenone derivative (**1**) is non-aromatic as evidenced by its n m.r. spectrum[1,3] and we now report bond-length data which confirm this interpretation.



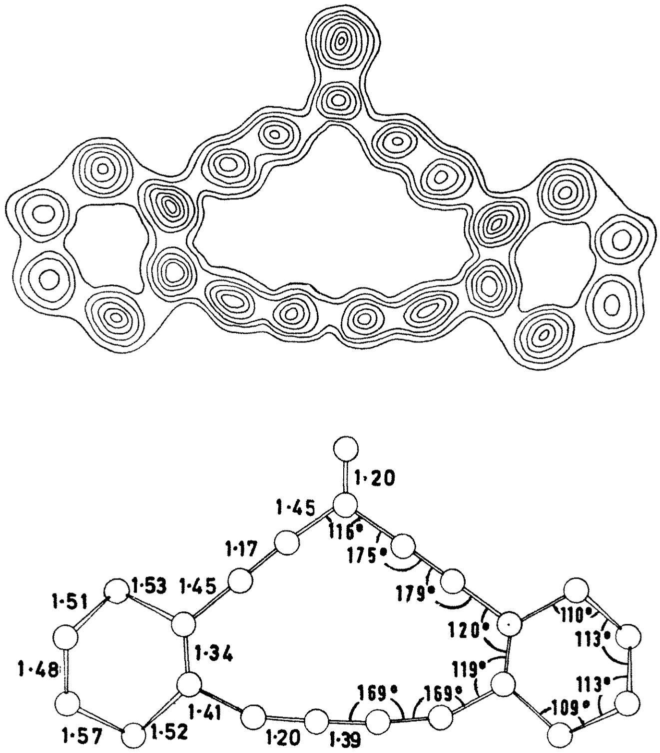
(1)

X-Ray diffraction data were obtained with considerable difficulty from ill formed crystals. The average lifetime of a crystal in an X-ray beam is only two days and reflexion intensity data were based therefore almost entirely on visual estimations of a series of Weissenberg photographs (Cu-K_α); interlayer scaling was achieved *via* the observation of a number of integrated intensities on a Pailred diffractometer (Mo-K_α).

The crystals are monoclinic with a = 12·96, b = 13·36, c = 8·98 Å, β = 93·9°, spacegroup $P2_1/c$; Z = 4. Initial attempts at solution of the structure by direct methods were unsuccessful, owing probably to both the poor quality of the data and the pseudo-special positioning of the molecule with respect to symmetry elements. The orientation of the molecule was determined by an automatic scan of the three-dimensional Patterson series, using a rotational minimum function based on the intramolecular vectors of the probable annulenone skeleton, and its absolute position was determined by means of a translation function[4] coupled with minimum residual calculations. Refinement of atomic positions and vibrational parametres by least-squares methods has converged to a present unweighted discrepancy index of 0·16 Further refinement is in progress.

Within experimental error the annulenone skeleton is planar with C_{2v} symmetry and the conformations of the two cyclohexene rings are such as to give approximate C_2 symmetry to the molecule as a whole. The molecular electron density (contours every eÅ^{-3}, the first contour being 1eÅ^{-3}), bond lengths (Å) and bond angles are shown in the Figure (average e.s.d. of the C–C and C–O bonds is 0·015 Å)

The mean bond length for the formal triple bonds is 1·19 Å, for the double bonds it is 1·34 Å, and for the single bonds between trigonally and digonally hybridised atoms and between two digonal–hybrid carbon atoms the bond lengths are 1·43 and 1·39 Å respectively. None of these values is significantly different from the appropriate 'accepted' values of 1·20, 1·34, 1·45, and 1·42 Å and the carbonyl bond length is also identical, within experimental error, to that for a carbon–oxygen bond of order two. The results confirm that there is no significant cyclic delocalisation in the molecule.



FIGURE

We thank Dr. Pilling and Professor Sondheimer for a sample of the compound.

(*Received, May* 18*th,* 1971; *Com.* 792.)

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[4] A. A. Crowther and D. M. Blow, *Acta Cryst.*, 1967, **23**, 544.