

CRYSTAL STRUCTURE OF 1,2-CYCLO- PENTENOPHENANTHRENE

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ABSTRACT. The crystal structure of 1,2-cyclo-pentenophenanthrene has been determined by the Fourier-synthesis method. A qualitative suggestion about the structure was made by Iball (1935). Following are the crystal data: Molecular formula $C_{17}H_{14}$; Monoclinic system: $a = 18.38 \text{ \AA}$; $b = 5.83 \text{ \AA}$, $c = 23.61 \text{ \AA}$; $\beta = 114^\circ-18'$; space-group $B2_1/c$ (Real space-group $P2_1/c-C^2_{2h}$); density = 1.23 gm/c.c. at 20°C ; No. of molecules per unit cell ('b' face-centred cell) = 8.0.

Zero-layer and equi-inclination Weissenberg photographs were taken in a Unicam Weissenberg goniometer with copper radiation and the intensities estimated microphotometrically for stronger spots and visually for the weaker ones. The substance was kindly supplied by Dr. Devdas Mukherjee of Scottish Gas Board from Dr. London of the Biochemistry Laboratory, Glasgow University.

In finding out the structure by trial, help was taken of the suggestion made by Iball (*loc. cit.*). After a fairly satisfactory trial structure was arrived at, two projections were made about the a and b axes respectively. Because of the large values of a and c axial lengths, electron densities were calculated at intervals of 3° along them, using 3° strips of Lipson and Beevers. Values were calculated at 6° interval along b axis. Of the 17 carbon atoms, 13 were fairly well resolved in the b axis projection (figure 1). Only a very small number of atoms were resolved in the a axis projection, although the 'y' co-ordinates could be fixed to a fair degree of accuracy from the same. The considerations of the hydrogen atoms have been omitted for the present. Taking the new co-ordinates obtained from these projections, the values of structure factors were calculated when the reliability index R had the values 0.28 for both the ($o\ k\ l$) and ($h\ o\ l$) zones, where

$$R = \frac{\sum |F_{obs} - F_{calc}|}{\sum |F_{obs}|}$$

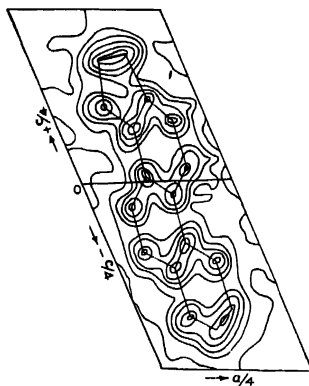


Fig. 1.

This high value of R is due most probably to the presence of a comparatively large number of very weak and absent reflections in these two zones. The coordinates of the carbon atoms of one asymmetric unit (one molecule) are given in Table I. The bond lengths and bond angles are all shown in figure 2. If χ_1, ψ_1, ω_1 be the angles made by the long axis of the molecule formed by joining atoms C_8 and C_{17} with the a, b and c axes respectively and χ_2, ψ_2 and ω_2 be the corresponding angles made by the short axis represented by the straight line joining C_{10} and C_{12} , then

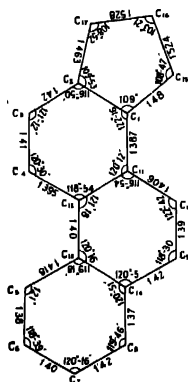


Fig. 2.

$$\begin{array}{ll} \chi_1 = 109^\circ & \chi_2 = 121^\circ - 12' \\ \psi_1 = 90^\circ & \psi_2 = 32^\circ - 22' \\ \omega_1 = 5^\circ - 18' & \omega_2 = 98^\circ - 23' \end{array}$$

The minimum distance of approach between two molecules comes out to be 3.98 Å.

TABLE I
Coordinates of the atoms of one molecule

Atom	$\frac{x}{a}$	$\frac{y}{b}$	$\frac{z}{c}$
C ₁	.1204	.0616	.0656
C ₂	.1629	-.1173	.1060
C ₃	.1948	-.2010	.0804
C ₄	.1827	-.2858	.0174
C ₅	.1656	-.2730	-.1063
C ₆	.1565	-.2701	-.1675
C ₇	.1130	-.0900	-.2060
C ₈	.0797	.0851	-.1824
C ₉	.0539	.2510	-.0991
C ₁₀	.0638	.2410	-.0375
C ₁₁	.1077	.0668	.0035
C ₁₂	.1403	-.1068	-.0212
C ₁₃	.1303	-.1016	-.0832
C ₁₄	.0895	.0773	-.1216
C ₁₅	.0884	.2177	.0993
C ₁₆	.1150	.1320	.1657
C ₁₇	.1596	-.0896	.1664

That the structure is substantially correct is confirmed from the following considerations; (i) The value of $\omega_1 = 5^\circ - 18'$ agrees very well with that of $\omega_1 =$ about 6° as predicted by Bernal (1935) from optical measurements, (ii) The 'b' axis projection of the molecule except the five-membered ring agrees very well with the corresponding electron density projection of phenanthrene crystal as obtained by Basak (1950). The structure obtained agrees with the suggestions put forward by Iball (loc.cit). The bond lengths, bond angles and intermolecular

distances all lie within the limits of values generally obtained in other organic compounds of similar chemical formulae. The structure is now being refined with the help of latest techniques and when completed will give informations about the finer details.

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