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AN X-RAY DETERMINATION OF THE CONFORMATION OF DIBENZO[b,f]TROPONE

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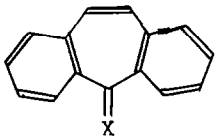
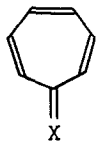
It is well known that the properties of dibenzoheptafulvene derivatives (I) are considerably different from those of the corresponding non-condensed ones (II) (1, 2).



(I)



(II)

a: X = CH_2

b: X = O

c: X = $C(CN)_2$

d: X = $CHCH=\overset{+}{N}(CH_3)_2$ ClO_4^-

The discrepancies were supposed to arise from the non-planarity of the seven-membered ring of the formers (3). The present investigation has been undertaken to find the definite molecular geometry of (Ib), as a part of serial structural studies of aromatic seven-membered ring compounds.

Dibenzo[b,f]tropone, $C_{15}H_{10}O$, (Ib) crystallizes in a monoclinic space group Ic, with four molecules in a unit cell of dimensions: a=18.53Å, b=3.95Å, c=14.50Å, and β=98.3°.

Multiple-film equi-inclination Weissenberg photographs were taken for the layer lines from 0 to 2 about the b axis and from 0 to 10 about the c, using CuKα radiation, and the integrated intensities were estimated visually against

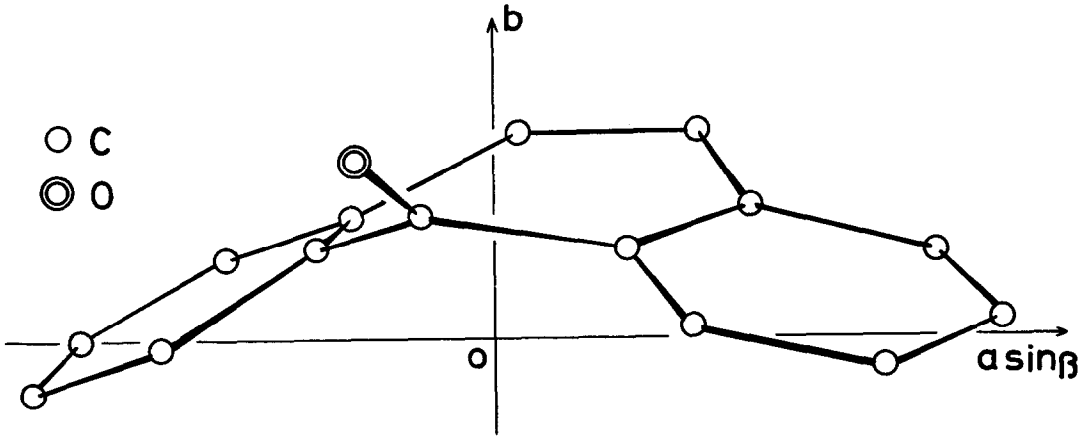
a standard scale. The independent reflections from 698 planes were observed.

The structure was solved by interpreting Patterson function. The positional and thermal parameters of the non-hydrogen atoms so obtained were refined by the least-squares method. Anisotropic temperature factors were applied. After five cycles of refinement the discrepancy factor, $R=\sum||F_o|-|F_c||/\sum|F_o|$, was reduced to 0.11.

The molecule is not planar as shown in Fig.; the seven-membered ring is in the boat conformation similar to cycloheptatriene (4, 5). The two benzene ring planes are inclined to each other at the angle of about 39°. The bond lengths and angles indicate that there is no contribution from the dipolar resonance form often assumed in (II).

Further refinements are in progress. A detailed account of this work will be presented in the near future.

Fig.



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