Short Communications

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Observed and calculated bond lengths in acedianthrone.

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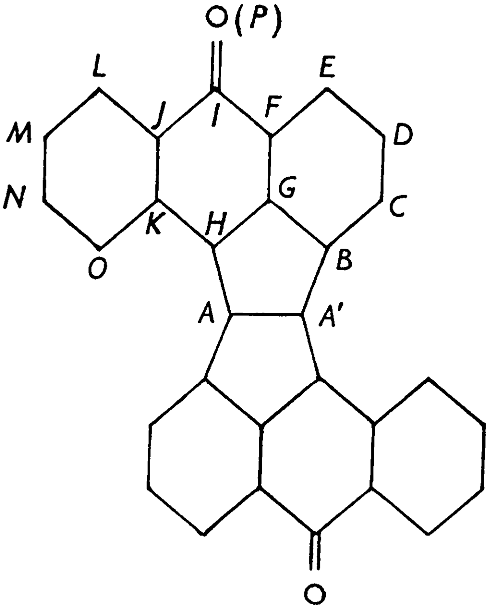
Introduction

Acedianthrone (I), also known as aceanthrone-2′:1′:1:2-aceanthrone,



(I)

was, when we began this investigation, of interest for two special reasons. In the first place it was desirable to confirm or contradict the chemical formula postulated by Clar (1939), and, in the second, the bond lengths and angles in the rather unusual five-membered rings which it contains seemed well worth measuring.

Crystallographic measurements

Acedianthrone, $C_{30}O_2H_{14}$; mol.wt. 406·11; m.p. > 360° C.; $d_{\text{calc.}} = 1{\cdot}490$, $d_{\text{obs.}} = 1{\cdot}498$ g.cm.$^{-3}$. Monoclinic prismatic: $a = 5{\cdot}64 \pm 0{\cdot}01$, $b = 10{\cdot}96 \pm 0{\cdot}02$, $c = 15{\cdot}49 \pm 0{\cdot}03$ Å, $\beta = 108{\cdot}2 \pm 0{\cdot}5°$. Absent spectra: $\{h0l\}$ absent when l odd, $\{0k0\}$ absent when k odd. Space group $P2_1/c$. Two centrosymmetrical molecules per unit cell; electrons per unit cell = $F(000) = 420$. Absorption coefficient for Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54$ Å) = 8·7 cm.$^{-1}$. The crystals are fine well formed needles elongated parallel to the a axis.

Structure analysis

Attempts to elucidate the structure by the usual methods were unsuccessful. Eventually the method of the Fourier transform was applied, using the technique adopted by Goodwin & Vand (private communication) for benzanthrone. By this means a generally satisfactory correlation of observed and calculated geometrical structure factors was secured with discrepancies $R = 100||F_o|-|F_c||\div|F_o|$

Table 1. *Atomic co-ordinates*

	X	x	X'	Y	y	Z	z	Z'
A	0·06	0·011	0·21	0·62	0·057	−0·47	−0·030	−0·45
B	1·27	0·225	0·75	−0·68	−0·062	1·66	0·107	1·58
C	1·85	0·328	0·94	−1·44	−0·131	2·90	0·187	2·76
D	3·14	0·557	1·93	−1·14	−0·104	3·87	−0·250	3·68
E	3·96	0·702	2·82	−0·14	−0·013	3·68	0·238	3·50
F	3·36	0·596	2·57	0·70	0·064	2·52	0·163	2·39
G	2·10	0·372	1·63	0·38	0·035	1·51	0·097	1·44
H	1·46	0·259	1·42	1·06	0·097	0·14	0·009	0·13
I	4·20	0·745	3·54	1·76	0·161	2·12	0·137	2·01
J	3·57	0·633	3·30	2·46	0·225	0·86	0·055	0·82
K	2·21	0·392	2·24	2·28	0·208	−0·10	−0·006	−0·10
L	4·34	0·770	4·17	3·52	0·322	0·54	+0·035	0·51
M	3·86	0·684	4·04	4·30	0·392	−0·58	−0·037	−0·55
N	2·52	0·446	3·03	4·10	0·374	−1·63	−0·105	−1·55
O	1·84	0·326	2·24	3·12	0·285	−1·29	−0·083	−1·23
P	5·42	0·960	4·56	1·98	0·181	2·74	0·177	2·60

Here (X, Y, Z) and (X', Y, Z') are atomic co-ordinates (in Å) with respect to monoclinic (a, b, c) and pseudo orthorhombic (a, b, c^*) axes; (x, y, z) are fractional co-ordinates with respect to the monoclinic axes.

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Table 2. *Bond lengths*

Values in Ångström units

Bond	Length obs.	Length calc.	Δ	Bond	Length obs.	Length calc.	Δ
AA'	1·59	1·42	+0·17	*GH*	1·49	1·43	+0·06
AB'	1·48	1·44	+0·04	*HK*	1·49	1·43	+0·06
AH	1·41	1·40	+0·01	*IJ*	1·40	1·45	−0·05
BC	1·42	1·40	+0·02	*IP*	1·20	1·22	−0·02
BG	1·39	1·41	−0·02	*JK*	1·42	1·41	+0·01
CD	1·38	1·39	−0·01	*JL*	1·41	1·39	+0·02
DE	1·35	1·39	−0·04	*KO*	1·41	1·40	+0·01
EF	1·41	1·40	+0·01	*LM*	1·32	1·39	−0·07
FG	1·37	1·41	−0·04	*MN*	1·44	1·39	+0·05
FI	1·49	1·45	+0·04	*NO*	1·30	1·40	−0·10

of 20·0, 27·8 and 18·8 in the $\{hk0\}$, $\{h0l\}$ and $\{0kl\}$ zones respectively. The structure which gave these results had the atomic co-ordinates listed in Table 1, leading to the interatomic distances quoted in Table 2 and the bond angles in the pentalene ring set out in Table 3. One of the later projections along *a* is shown in Fig. 1.

Table 3. *Angles in pentalene ring*

Values in degrees, all ±5°

A'AH	104
AA'B	107
A'BG	108
BGH	108
GHA	110

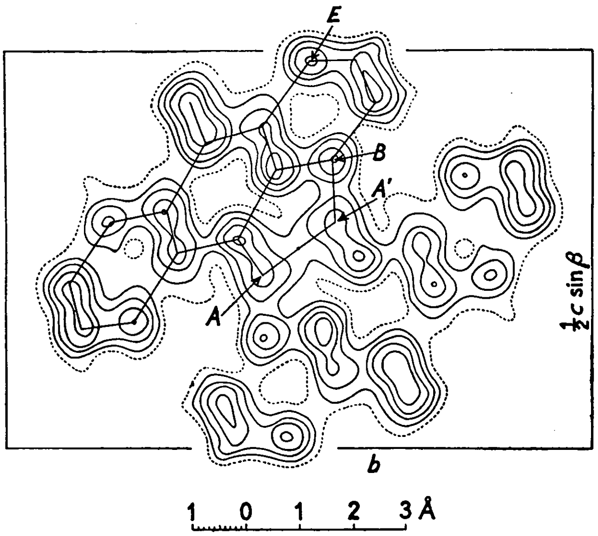

Fig. 1.

Although there is comparatively little information concerning bond lengths in compounds like acedianthrone we felt rather surprised about the lengths indicated for bonds *AA'*, *AB'*, *IJ*, *LM*, *NO*, *HK*, even allowing for the fact that the first two form part of the curious five-membered ring. One of us (T. H. G.) has therefore calculated the bond lengths in acedianthrone by the method of molecular orbitals with the results shown in column 3 of Table 2. The fourth column of this table gives the difference between 'observed' and 'calculated' bond lengths. The 'observed' bond lengths are considered to be subject to an error of ±0·06 Å; this is due to the overlap of molecules in the Fourier projection along *b* and to non-resolution of certain atoms in other projections. Within these limits, therefore, there is general agreement between the X-ray results and the wave-mechanical calculations, although there is clearly room for improvement in the case of the bonds *AA'*, *NO* and *LM* and probably also *GH* and *HK*; indeed bond *AA'* must be wrong and we hope to return to the examination of this compound and to resolve these discrepancies. Nevertheless the results show that the crystal structure must be substantially as given and, in particular, confirm the formula (I) for acedianthrone.

Molecular arrangement

The equation of the molecular plane, calculated from the co-ordinates (Table 1) of atoms *E* and *L* and the fact that it passes through the origin, is

$$-0{\cdot}593X'+0{\cdot}629Y+0{\cdot}503Z' = 0\,.$$

All the atoms lie within 0·1 Å of this plane except *F* and *P* (the quinonoid oxygen) which are 0·12 and 0·15 Å away, deviations which we feel are scarcely significant.

When viewed parallel to the line $0{\cdot}593X' = 0{\cdot}503Z'$, $Y = 0$ the molecule at the origin and that derived from it by the operation of the screw axis are seen edge-on and their planes are nearly perpendicular. The intermolecular distance perpendicular to the molecular planes is 3·4 Å. These last observations are both in accord with the arrangement in many crystals containing disc-like molecules.

Experimental

The sample of acedianthrone, given to us by Messrs Imperial Chemical Industries Limited, deposited dark green opalescent crystals from nitrobenzene solution. These were examined by rotation, oscillation and Weissenberg photographs about the principal axes, intensity measurements being made by the multiple-film technique.

We are grateful to Messrs Imperial Chemical Industries Limited for the supply of acedianthrone and the loan of apparatus, to Dr V. Vand for helpful discussions, to Dr E. Clar for his interest, and to the Department of Scientific and Industrial Research for a maintenance grant to one of us (P. H. F.).

Reference

Clar, E. (1939). *Ber. dtsch. chem. Ges.* **72**, 2134.