

C1	0.57869 (12)	1.0458 (2)	-0.6416 (2)	0.0369 (4)
C2	0.59545 (12)	0.9228 (2)	-0.5713 (2)	0.0392 (4)
C3	0.65336 (12)	0.9165 (2)	-0.4232 (2)	0.0365 (4)
C4	0.69457 (11)	1.0330 (2)	-0.3472 (2)	0.0304 (3)
C5	0.67570 (12)	1.1566 (2)	-0.4220 (2)	0.0377 (4)
C6	0.61792 (13)	1.1633 (2)	-0.5688 (2)	0.0406 (4)
C7	0.75666 (11)	1.0249 (2)	-0.1909 (2)	0.0291 (3)
C8	0.76964 (12)	0.9013 (2)	-0.0948 (2)	0.0324 (4)
C9	0.83692 (11)	0.95473 (15)	0.0535 (2)	0.0283 (3)
C10	0.93677 (11)	0.8840 (2)	0.1088 (2)	0.0318 (4)
C11	0.92848 (11)	0.7468 (2)	0.1598 (2)	0.0334 (4)
C12	0.85166 (14)	1.0292 (2)	0.3512 (2)	0.0473 (5)

Table 2. Selected geometric parameters (\AA , $^\circ$)

CH—C1	1.743 (2)	N2—C11	1.135 (2)
S1—C12	1.794 (2)	C7—C8	1.495 (2)
S1—C9	1.810 (2)	C8—C9	1.527 (2)
O1—N1	1.414 (2)	C9—C10	1.527 (2)
O1—C9	1.448 (2)	C10—C11	1.454 (2)
N1—C7	1.274 (2)		
C12—S1—C9	102.83 (9)	O1—C9—C8	105.04 (12)
N1—O1—C9	109.66 (11)	C8—C9—C10	114.88 (14)
C7—N1—O1	109.54 (13)	C10—C9—S1	113.86 (11)
N1—C7—C4	120.82 (14)	C11—C10—C9	112.54 (13)
N1—C7—C8	114.23 (14)	N2—C11—C10	179.7 (2)
C7—C8—C9	101.21 (13)		
O1—N1—C7—C8	0.2 (2)	C3—C4—C7—C8	-9.0 (2)
C3—C4—C7—N1	169.96 (15)	N1—C7—C8—C9	3.3 (2)
C5—C4—C7—N1	-10.1 (2)	C7—C8—C9—O1	-5.21 (15)

The temperature of the crystal was controlled using an Oxford Cryosystem Cryostream Cooler (Cosier & Glazer, 1986). H atoms were added in calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms, while H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached.

Data collection: Siemens P3R3 system. Cell refinement: Siemens P3R3 system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Redetermination of Octahydrochrysene

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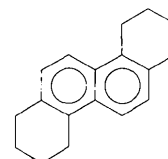
(Received 18 October 1995; accepted 13 November 1995)

Abstract

The central rings of 1,2,3,4,7,8,9,10-octahydrochrysene, $C_{18}H_{20}$, are essentially planar, with the r.m.s. deviation of the atoms defining the plane from the best-fit plane being 0.013 (2) \AA . The outer rings are found to be substantially non-planar, contrary to the conclusion of an earlier study based on photographic data [Ferrier & Iball (1958). *Acta Cryst.* **11**, 325–329]. The C—C single-bond distances in the outer rings are quite uniform, with the range of observed values varying by only 0.017 (6) \AA . There are no notably close intermolecular approaches.

Comment

Octahydrochrysene, (I), is of interest as a potential host crystalline material for magnetic or optical studies of substituted guest molecules. A previous structural analysis by Ferrier & Iball (1958) based on two-dimensional Fourier methods and with fixed H-atom geometry resulted in *R* values of 0.15 and 0.16 for the



(I)

principal axial zones. Considering that the crystals in that study were described as 'very imperfect', further study appeared warranted.

General broad agreement is found between the present results (Fig. 1) and those of Ferrier & Iball. In particular, the molecule is again found at a center of symmetry in space group $P2_1/c$. As expected, however, the present results supersede the previous results quantitatively.

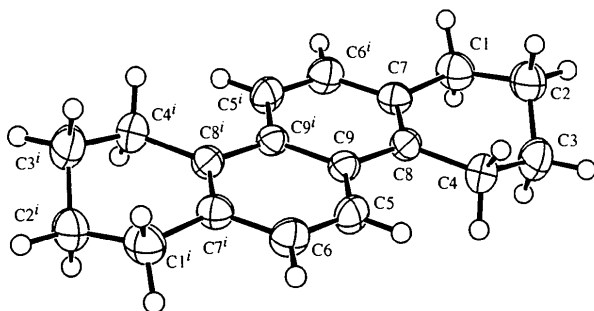


Fig. 1. An ORTEP (Johnson, 1976) drawing of octahydrochrysene showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for all atoms except H atoms, for which they have been set artificially small.

A qualitative point of difference concerns the planarity of the molecule. Ferrier & Iball stated the molecule, excluding the C2 and C3 atoms (present numbering), to be planar, with an r.m.s. deviation of atoms from the mean plane of 0.022 Å. With the present data, again excluding the C2 and C3 atoms (which are again the most deviant), the r.m.s. deviation of the remaining atoms from the least-squares best-fit plane through them is 0.036 (2) Å. However, if atoms C1 and C4 are also excluded, the r.m.s. deviation of the remaining five atoms from their best-fit plane is only 0.013 (2) Å. Furthermore, atom C4, the least deviant of the excluded atoms, deviates by nearly three times as much as the most deviant of the five plane-defining atoms. In summary, the present data show that only the central rings of octahydrochrysene can reasonably be described as planar and that the outer rings are substantially non-planar, as expected (Table 2).

The r.m.s. difference within the 11 pairs of corresponding C—C bond distances for the two studies is 0.03 Å, consistent with the lower precision of the earlier results as acknowledged by Ferrier & Iball. In that study, the values of the five outer C—C bond distances varied by 0.07 Å, while for the present data the variation is only 0.017 (6) Å. These latter results considerably clarify the geometry of the outer rings.

The C—H bond distances determined in this study are in the range 0.95 (2)–1.05 (2) Å, with a mean value of 1.01 (3) Å; the value assumed by Ferrier & Iball was 1.00 Å, which is in good agreement with this experimental value. Molecules are separated by normal van der Waals distances (Bondi, 1964).

Experimental

Octahydrochrysene was synthesized as described by Rahman & Khan (1962) and provided by Dr Rahman. The sample was dissolved in a 2:1 mixture of high-boiling petroleum ether and benzene as described by Rahman & Khan and evaporation of the solvent resulted in faintly amber prisms.

Crystal data

C₁₈H₂₀
 $M_r = 236.36$
 Monoclinic
 $P2_1/c$
 $a = 9.1293 (7) \text{ \AA}$
 $b = 7.2499 (6) \text{ \AA}$
 $c = 10.0753 (8) \text{ \AA}$
 $\beta = 100.793 (6)^\circ$
 $V = 655.05 (9) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.198 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 14.3\text{--}17.5^\circ$
 $\mu = 0.062 \text{ mm}^{-1}$
 $T = 296 \text{ K}$
 Prism
 $0.39 \times 0.35 \times 0.23 \text{ mm}$
 Faintly amber

Data collection

Rigaku AFC-5S diffractometer
 ω scans
 Absorption correction: none
 1729 measured reflections
 1634 independent reflections
 947 observed reflections
 $[I > 3\sigma(I)]$

$R_{\text{int}} = 0.038$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = 0 \rightarrow 11$
 $k = 0 \rightarrow 9$
 $l = -13 \rightarrow 12$
 6 standard reflections monitored every 150 reflections
 intensity variation: $\pm 2.6\%$

Refinement

Refinement on F
 $R = 0.045$
 $wR = 0.053$
 $S = 1.76$
 947 reflections
 123 parameters
 All H atoms refined isotropically
 $w = 1/\sigma^2(F)$
 $(\Delta/\sigma)_{\text{max}} < 0.01$

$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
 Extinction correction: Zachariasen (1963, 1968)
 Extinction coefficient: $8(1) \times 10^{-6}$
 Atomic scattering factors from Stewart, Davidson & Simpson (1965) for H atoms and from Cromer & Waber (1974) for C atoms

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	x	y	z	U_{eq}
C1	-0.3555 (2)	-0.0178 (3)	0.1858 (2)	0.0509 (6)
C2	-0.3334 (3)	0.1400 (3)	0.2876 (2)	0.0569 (7)
C3	-0.2930 (3)	0.3140 (3)	0.2211 (2)	0.0565 (7)
C4	-0.1441 (2)	0.2902 (3)	0.1755 (2)	0.0463 (6)
C5	0.1025 (2)	0.2212 (3)	0.0336 (2)	0.0428 (5)
C6	0.2156 (2)	0.1958 (3)	-0.0348 (2)	0.0454 (6)
C7	-0.2303 (2)	-0.0312 (3)	0.1063 (2)	0.0392 (5)
C8	-0.1297 (2)	0.1097 (2)	0.1044 (2)	0.0361 (5)
C9	-0.0078 (2)	0.0840 (2)	0.0347 (2)	0.0341 (5)

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Table 2. Selected geometric parameters (Å, °)

C1—C2	1.524 (3)	C5—C9	1.416 (2)
C1—C7	1.517 (3)	C6—C7'	1.414 (2)
C2—C3	1.507 (3)	C7—C8	1.375 (2)
C3—C4	1.524 (3)	C8—C9	1.435 (2)
C4—C8	1.509 (2)	C9—C9'	1.425 (3)
C5—C6	1.357 (3)		
C2—C1—C7	112.7 (2)	C6'—C7—C8	119.5 (2)
C1—C2—C3	110.0 (2)	C4—C8—C7	121.3 (2)
C2—C3—C4	110.2 (2)	C4—C8—C9	119.3 (2)
C3—C4—C8	113.8 (2)	C7—C8—C9	119.5 (2)
C6—C5—C9	121.2 (2)	C5—C9—C8	122.0 (2)
C5—C6—C7'	121.8 (2)	C5—C9—C9'	117.4 (2)
C1—C7—C6'	118.4 (2)	C8—C9—C9'	120.6 (2)
C1—C7—C8	122.1 (2)		
C1—C2—C3—C4	−63.5 (2)	C4—C8—C7—C1	−3.5 (3)
C2—C3—C4—C8	45.3 (2)	C8—C7—C1—C2	−14.6 (3)
C3—C4—C8—C7	−12.1 (3)	C7—C1—C2—C3	47.6 (3)

Symmetry code: (i) $-x, -y, -z$.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including least-squares-planes data and torsion angles, together with a stereoview of the unit-cell contents, have been deposited with the IUCr (Reference: FG1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structural Aspects of the Transposition Reaction of Oxatricyclo[6.5.0.0^{2,7}]tridecane-6,7-diol Monomesylate

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Abstract

The structure and conformation of the molecules (1*RS*, 2*SR*, 6*SR*, 7*SR*, 8*SR*)-6,7-dihydroxy-3-oxatricyclo[6.5.0.0^{2,7}]tridec-1-yl acetate, (2), and (1*SR*, 2*SR*, 6*SR*, 7*RS*)-11-oxo-10-oxatricyclo[5.3.1.0^{2,6}]undec-2-yl acetate, (4), are illustrated and discussed. Crystals of (2) are triclinic, *P* $\bar{1}$, with two molecules in the asymmetric unit which show small but significant differences in conformation. These molecules are joined in ribbons running along a crystallographic direction by a system of bifurcated intra- and intermolecular hydrogen bonds formed by the hydroxy groups. The tricyclic systems in the molecules of both compounds have an *anti* conformation with *cis* substitutions at the ring junctions. In (4) the keto group is transoid to the acyclic atoms at the junction of the two five-membered rings (with respect to the plane through the central ring).

Comment

As part of a program dealing with new synthetic routes to heterocyclic derivatives (Caubère, 1991; Caubère, Caubère, Renard, Bizot-Espiart & Jamart-Grégoire, 1993; Caubère, Caubère, Ianelli, Nardelli & Jamart-Grégoire, 1994; Jamart-Grégoire, Léger & Caubère, 1990; Lalloz & Caubère, 1975), we previously showed that dehydropyranyl cyclobutanols (1) could be conveniently obtained by nucleophilic condensation of ketone enolates with 3,4-dihydrodihydropyran generated from 5-bromo-3,4-dihydropyran. On the other hand, we also previously showed that the carbocyclic analogues of the alcohols (1) could be easily transformed into polycyclic cyclopentane derivatives (Jamart-Grégoire, Brosse, Ianelli, Nardelli & Caubère, 1993). Thus we thought that polycyclic tetrahydrofuran could be obtained in the same way. When the strategy previously