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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: BK1262). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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sym-Hexahydropyrene

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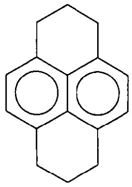
Abstract

In 1,2,3,6,7,8-hexahydropyrene, $C_{16}H_{16}$, the unsaturated rings lie essentially in a plane, the r.m.s. deviation of the atoms defining this plane from the best-fit plane being 0.006 (2) Å. The saturated rings are substantially non-planar, but the character of the non-planarity of the saturated rings is quite different from that observed in octahydrochrysene. The C—C bond distances in the saturated rings are quite uniform, ranging from 1.505 (2) to 1.520 (2) Å. No intermolecular distance is less than the sum of the corresponding van der Waals radii; closest approaches involve both C···H and H···H interactions.

Comment

Hydroaromatic hydrocarbons are of interest as potential host crystal materials for magnetic or optical studies of dilutely substituted guest molecules such as aromatic hydrocarbons. This study of *sym*-hexahydropyrene, (I), follows a study of octahydrochrysene (Burd, Dobson & Gerkin, 1996).



(I)

In this $P2_1/c$ structure, the asymmetric unit is half of the *sym*-hexahydropyrene molecule, the other half being generated by an inversion center. These features duplicate those of octahydrochrysene.

The atoms constituting the naphthalenoid core of this molecule (C4–C8 and their inversion partners) are described by a best-fit plane from which their r.m.s. deviation is 0.006 Å, the e.s.d. of the individual deviations being 0.001 Å. The corresponding value for the naphthalenoid core of octahydrochrysene is 0.013 (2) Å. Atoms C1, C3, C1^{i} and C3^{i} [symmetry code: (i) $1-x$, $1-y$, $1-z$] lie close to this plane (Fig. 1). Indeed, the r.m.s. deviation of all the non-H atoms, except for the 'terminal' C2 and C2^{i} atoms, from the best-fit plane describing them is only 0.018 Å, the e.s.d. of the individual deviations being 0.001 Å. The deviations of atoms C2 and C2^{i} from this plane are of magnitude 0.665 Å. The saturated rings are thus substantially non-planar, as expected, but due to only one of the three outer C atoms; in octahydrochrysene, the non-planarity involves all (four) outer C atoms. In contrast, in pyrene itself, the r.m.s. deviation for the entire set of C atoms from the best-fit plane through them is variously 0.007 Å [as calculated from the neutron diffraction data of Hazell, Larsen & Lehmann (1972)], 0.008 Å [as calculated from the X-ray data of Allmann (1970)] or 0.010 Å [as calculated from the 113 K X-ray data of Kai, Hama, Yasuoka & Kasai (1978)]. The dihedral angle between the sets of noncoplanar molecules in the present structure is 12.72 (4)°. This may be contrasted with the analogous dihedral angle in pyrene itself of 83.7° [as calculated from the data of Hazell, Larsen & Lehmann (1972)].

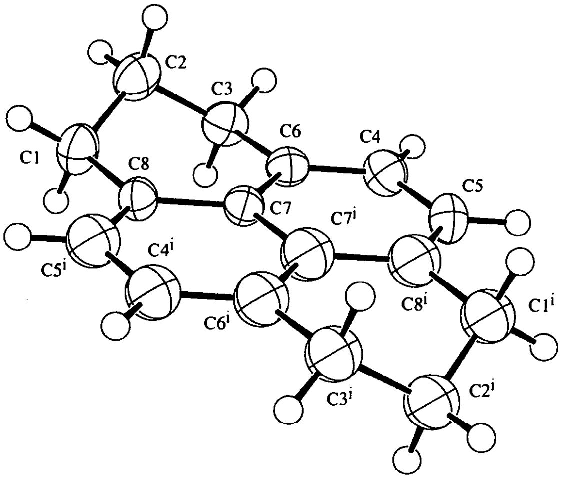


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of *sym*-hexahydropyrene showing the atomic numbering scheme. Displacement ellipsoids are drawn at 50% probability for all atoms except H atoms, for which they have been set artificially small. The midpoint of the C7—$C7^i$ bond lies at a center of symmetry which relates corresponding atoms to their symmetry generated positions [symmetry code: (i) $1-x$, $1-y$, $1-z$].

The intramolecular C—C bond distances (Table 2) display pseudo-mirror symmetry; the r.m.s. deviation within the four pairs of distances which would be identical under mirror symmetry is 0.003 Å and the e.s.d. of each distance is 0.002 Å. The nominally single C—C bond distances in the saturated rings are 1.505 (2) and 1.510 (2) Å for $C_{aromatic}$—$C_{saturated}$, and 1.520 (2) and 1.519 (2) Å for $C_{saturated}$—$C_{saturated}$. Thus, while the range of distances is small, the distances appear to fall into two groups consistent with the symmetry and pseudosymmetry. This range of C—C single-bond distances [0.015 (4) Å] is very similar to that seen in octahydrochrysene [0.017 (6) Å]. The final refined values of C—H bond distances range from 0.96 (2) to 1.03 (2) Å, with a mean value 0.99 (2) Å.

In the structure of (I), no intermolecular closest approach was less than the sum of the corresponding Bondi (1964) radii. Closest approaches involve both C···H and H···H interactions.

Experimental

sym-Hexahydropyrene, obtained from Henley and Co. Inc., was recrystallized twice from ethanol. This process produced numerous usable columnar specimens.

Crystal data

$C_{16}H_{16}$
$M_r = 208.30$
Monoclinic
$P2_1/c$
$a = 4.8366\,(7)$ Å
$b = 12.5390\,(9)$ Å
$c = 9.3207\,(9)$ Å
$\beta = 93.03\,(1)°$
$V = 564.5\,(1)$ Å^3
$Z = 2$
$D_x = 1.225$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.4$–$17.4°$
$\mu = 0.064$ mm^{-1}
$T = 296$ K
Cut column
$0.42 \times 0.38 \times 0.27$ mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
ω–2θ scans
Absorption correction: none
3048 measured reflections
1370 independent reflections
946 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.028$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 6$
$k = -16 \rightarrow 16$
$l = -12 \rightarrow 12$
6 standard reflections monitored every 150 reflections
intensity decay: see below

Refinement

Refinement on F
$R = 0.035$
$wR = 0.047$
$S = 2.04$
946 reflections
107 parameters
All H-atom parameters refined
$w = 1/\sigma_F^2$
$(\Delta/\sigma)_{max} = < 0.01$

$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: Zachariasen (1963, 1968)
Extinction coefficient: $1.0\,(2) \times 10^{-5}$
Atomic scattering factors from Stewart, Davidson & Simpson (1965) for H atoms and Cromer & Waber (1974) for C atoms

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.1328 (3)	0.4354 (1)	0.7601 (2)	0.0460 (4)
C2	0.1655 (3)	0.5462 (1)	0.8248 (2)	0.0484 (4)
C3	0.1234 (3)	0.6296 (1)	0.7075 (1)	0.0414 (4)
C4	0.4135 (3)	0.69699 (10)	0.5110 (2)	0.0402 (4)
C5	0.5903 (3)	0.68110 (10)	0.3981 (2)	0.0409 (4)
C6	0.3201 (3)	0.61337 (9)	0.5895 (1)	0.0337 (3)
C7	0.4093 (2)	0.50800 (9)	0.5571 (1)	0.0311 (3)
C8	0.3212 (3)	0.41842 (10)	0.6379 (1)	0.0346 (3)

Table 2. *Selected geometric parameters (Å, °)*

C1—C2	1.520 (2)	C4—C6	1.369 (2)
C1—C8	1.510 (2)	C5—$C8^i$	1.367 (2)
C2—C3	1.519 (2)	C6—C7	1.427 (2)
C3—C6	1.505 (2)	C7—$C7^i$	1.429 (2)
C4—C5	1.405 (2)	C7—C8	1.430 (2)
C2—C1—C8	111.9 (1)	C4—C6—C7	118.9 (1)
C1—C2—C3	109.7 (1)	C6—C7—$C7^i$	119.5 (1)

C2—C3—C6	111.5 (1)	C6—C7—C8	120.9 (1)
C5—C4—C6	121.5 (1)	C7^{i}—C7—C8	119.6 (1)
C4—C5—C8^{i}	121.6 (1)	C1—C8—C5^{i}	121.5 (1)
C3—C6—C4	121.7 (1)	C1—C8—C7	119.7 (1)
C3—C6—C7	119.3 (1)		

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Each of the two subsets of data [subset 1: indices $h,k,\pm l$; subset 2, collected immediately following collection of subset 1: indices $h,-k,\pm l$] was corrected to account for decay; -1.9% for subset 1 and -2.3% for subset 2. Following this, the subsets were merged, with the inclusion of a second (refined) scale factor, to form the final data file. The greatest positive residual electron density occurred $\sim$0.7 Å from the C7 and C8 atoms, while the greatest negative density occurred near the centers of the unsaturated rings.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

The authors thank R. H. McElheny for recrystallizing the sample and Dr J. C. Gallucci for help of various sorts. The diffractometer was purchased with funds provided in part by an NIH grant.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1217). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Hydroxybiphenyl-3-carboxylic Acid (3-Phenylsalicylic Acid)

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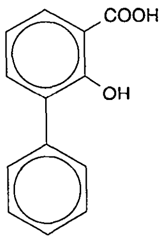
Abstract

In the title compound, $C_{13}H_{10}O_3$, there is a single type of intermolecular hydrogen bond. From this is formed a cyclic dimer about a twofold axis. In addition, there is an intramolecular hydrogen bond which is virtually identical to that observed in salicylic acid.

Comment

This structural study of 2-hydroxybiphenyl-3-carboxylic acid, (I), is one of a continuing series on hydrogen bonding in carboxylic acids. Of additional interest was the potential involvement of the hydroxyl group in the hydrogen-bonding scheme; since in this molecule, the carboxyl and hydroxyl functions are located *ortho* with respect to one another, a likely possibility was that a carboxyl O atom would act as an acceptor of an intramolecular hydrogen bond from the hydroxyl group (see, for example, Blackburn, Dobson & Gerkin, 1996*a*, and references therein).



(I)

In the title acid (Figs. 1 and 2), there is a single type of intermolecular hydrogen bond, which forms cyclic dimers about a twofold symmetry axis. Also, as anticipated, an intramolecular hydrogen bond as described above is present; geometric details are given in Table 3. Distances and angles involving the carboxylic group atoms show that these O atoms are ordered and that the carboxylic H atom is also ordered (Tables 2 and 3).