

The integrated intensities were measured using a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) peak-profile analysis procedure. Corrections were applied for Lorentz and polarization effects but not for absorption. Extinction was considered according to Zachariasen (1963) [$g = 4.06(1) \times 10^{-8}$].

The structure was determined by direct methods using *SHELXS86* and refined by anisotropic full-matrix least squares on *F* using *SHELX76*. The H atoms were localized from a final difference Fourier synthesis and refined isotropically.

The atom-atom non-bonded potential-energy calculations were carried out using *ROTENER* (Nardelli, 1988) and the atomic charges calculated by the iterative partial equalization of orbital electronegativity method of Gasteiger & Marsili (1980) using *ATOMCHAR* (Nardelli, 1991).

Throughout this paper, the averaged values are means weighted according to the reciprocals of the variances and the corresponding e.s.d.'s are the largest values of the 'external' and 'internal' standard deviations (Topping, 1960).

The calculations were performed using the ENCORE-GOULD-POWERNODE 6040 computer at the 'Centro di Studio per la Strutturistica Diffraattometrica del CNR (Parma)'.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, together with the results of the Cambridge Structural Database search and difference van der Waals energy profiles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55178 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1005]

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Structure and Conformation of *cis,anti,cis*-4b,4c,8b,9,10,10a-Hexahydro-4b-hydroxybenzo[3,4]cyclobuta[1,2-*a*]biphenylene-4-carbaldehyde

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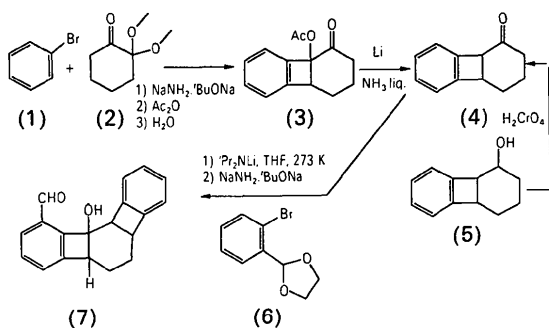
Abstract

The title compound was prepared as part of a study of polyphenylene derivatives obtained by arynic condensation in the presence of complex bases. X-ray analysis shows that the two benzocyclobutene moieties have an *anti* arrangement. The hydroxyl group and the H atoms are *cis* at each junction and *trans* for one junction with respect to the other. The two pseudosymmetrical parts of the core of the molecule show the same geometry in spite of having different substitution. Previously observed deformations for the benzene moiety fused with the cyclobutene ring and lengthening of the cyclobutane Csp^3-Csp^3 bond are confirmed. The orientations of the formyl and hydroxyl groups are discussed.

Comment

Polyphenylene derivatives are very interesting molecules from both the theoretical point of view and with regard to their potential use as starting materials for further syntheses or in devising new electronic materials (Dierck & Vollhardt, 1986). We have previously shown that arynic condensation of appropriate ketone enolates leads easily to benzocyclobutene derivatives which could themselves be used as starting materials for the synthesis of triphenylene derivatives (Zouaoui, Mouaddib, Jamart-Grégoire, Ianelli, Nardelli & Caubère, 1991). Moreover, we also showed that some di- or tetrahydrobiphenylenes perturb DNA replication (Zouaoui, Mouaddib, Jamart-Grégoire, Caubère, Mougine, Caubère & Branlant, 1989). This previously unknown property has opened up a range of new applications.

In order to introduce functional groups on one of the aromatic rings, we performed the condensations shown below. One of the interesting aspects of the product obtained (7) is its aldehydic function which may be easily transformed into a number of other functional groups.



Compound (3) was obtained in the manner previously described by Carré, Grégoire & Caubère (1984). Its reduction by lithium in liquid NH_3 , followed if necessary by oxidation of (5), easily led to (4). Arynic condensation of the enolate (4) with (6) in the presence of a complex base (Caubère, 1991) led to (7), whose stereochemistry was not easy to establish by usual physical methods. The structure, as described in this paper, was established by X-ray diffraction.

An ORTEP drawing of the molecule (Fig. 1) shows that it consists of two benzocyclobutene systems joined together, directly and through a dimethylene bridge, with an *anti* configuration. The H atoms and the hydroxyl group at the junctions are *cis* at each junction and those of one junction are *trans* with respect to those of the other. The torsion angles (Table 2) indicate that the bonds at C8 and C13 are synperiplanar (eclipsed) with respect to those at C9 and C12 respectively. The configurations at these chiral atoms are *R* (C8), *R* (C9), *S* (C12), *R* (C13). The enantiomeric molecule which has the opposite configurations at these atoms is also present in the crystal, the space group being centrosymmetric.

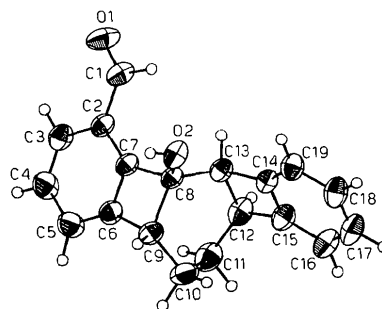


Fig. 1. ORTEP drawing of the molecule with ellipsoids at 50% probability.

From the values of bond distances and angles (Table 2), it appears that the two benzocyclobutene moieties are not significantly different and that deformations caused by the fusion of the benzene and cyclobutene rings are in agreement with those previously observed (Allen, 1981; Ianelli, Nardelli, Belletti, Geoffroy, Carré, Mouaddib & Caubère, 1990; Benassi, Ianelli, Nardelli & Taddei, 1991). In particular, the averaged data given in Table 3 should be considered. These data indicate that the most important distortions are the narrowing of the benzo β angle and the lengthening of the $\text{Csp}^3\text{—Csp}^3$ bond in the cyclobutene ring. This lengthening is responsible for the asymmetry of the cyclobutene endocyclic angles ϵ and ζ as previously observed by Ianelli, Nardelli, Belletti, Jamart-Grégoire, Mouaddib & Caubère (1992a,b).

The two rings of the benzocyclobutene system are nearly coplanar in both moieties, the dihedral angles being $\angle(\text{C2}, \dots \text{C7})(\text{C6}, \dots \text{C9}) = 3.7(1)^\circ$, $\angle(\text{C14}, \dots \text{C19})(\text{C12}, \dots \text{C15}) = 1.5(1)^\circ$. These two moieties, joined together by the C8—C13 bond and the C10—C11 dimethylene bridge, form a central puckered cyclohexane ring with a total puckering amplitude (Cremer & Pople, 1975) of $Q_T = 0.516(3) \text{ \AA}$ and a half-chair conformation with a pseudo twofold axis running along the midpoints of the C8—C13 and C10—C11 bonds [minimum asymmetry displacement parameter $\text{ADP} = D_2(\text{C8—C13}) = 0.026(1)$; Nardelli, 1983b].

The low value of the C3—C2—C1—O1 torsion angle [$-5.8(4)^\circ$] indicates that the orientation of the formyl group is mainly determined by its tendency to exhibit some π conjugation with the benzene ring. This is confirmed by the potential-energy profiles, calculated for the rotation of the group about the C1—C2 bond, which indicate that electronic effects must be present. The importance of these effects can be understood by considering the fact that, in its vapour phase, benzaldehyde has a planar ground state and a twofold barrier to internal rotation between 19 and 23 kJ mol^{-1} (Penner, George & Block, 1987).

Difference potential-energy profiles were calculated in the same way for the hydroxyl group by rotating it about the C8—O2 bond. These indicate that the orientation of the hydroxyl group in the crystal is mainly determined

by the intermolecular hydrogen-bonding interaction. The relevant geometrical parameters of this bond, which is the main influence on molecular packing in the crystal, are O2—H2O 0.78(3), O2...O1ⁱ 2.827(3), H2O...O1ⁱ 2.08(3) Å and O2—H2O...O1ⁱ 161(3)° where $i = 1-x, \frac{1}{2}+y, \frac{1}{2}-z$.

The anisotropic atomic displacement coefficients have been analysed, using *THMV* (Trueblood, 1984), in terms of the LST rigid-body model (Schomaker & Trueblood, 1968), taking into account the internal motions in the one-parameter model approximation (Dunitz & White, 1973; Trueblood, 1978). The results (Table 4) show that internal motions have some relevance; the atoms of the rings librate about directions perpendicular to the mean planes through the rings. The largest anisotropic thermal motion (or statistical disorder) is observed in the aldehydic O atom for which the ratio between the principal axes of the displacement ellipsoid r_{\max}/r_{\min} is 3.12; for the other atoms, this ratio is in the range 1.44–2.73. Bond-distance corrections do not exceed 2σ and are therefore not included in Table 2.

Experimental

Crystal data

C₁₉H₁₆O₂
 $M_r = 276.33$
 Monoclinic
 $P2_1/c$
 $a = 13.737$ (2) Å
 $b = 6.9879$ (5) Å
 $c = 15.124$ (5) Å
 $\beta = 94.37$ (1)°
 $V = 1447.6$ (5) Å³
 $Z = 4$
 $D_x = 1.2679$ Mg m⁻³

Cu $K\alpha_1$ radiation
 $\lambda = 1.540562$ Å
 Cell parameters from 30 reflections
 $\theta = 21\text{--}40^\circ$
 $\mu = 0.6066$ mm⁻¹
 $T = 293$ (2) K
 Thick prisms
 $0.51 \times 0.42 \times 0.36$ mm
 Colourless

Data collection

Siemens-AED diffractometer
 Peak profile: θ - 2θ scans
 Absorption correction: not applied
 2268 measured reflections
 2183 observed reflections [$I > 2\sigma(I)$]
 $R_{\text{int}} = 0.0268$

$\theta_{\max} = 70^\circ$
 $h = -16 \rightarrow 16$
 $k = 0 \rightarrow 8$
 $l = 0 \rightarrow 18$
 1 standard reflection monitored every 50 reflections
 intensity variation: none

Refinement

Refinement on F
 Final $R = 0.0453$
 $wR = 0.0468$
 $S = 0.7321$
 2183 reflections
 254 parameters
 All H-atom parameters refined
 Unit weights applied

$(\Delta/\sigma)_{\max} = 0.03$
 $\Delta\rho_{\max} = 0.08$ e Å⁻³
 $\Delta\rho_{\min} = -0.12$ e Å⁻³
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2A, 2.2C and 2.3.1)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)

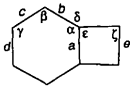
$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O1	0.4950 (1)	-0.1726 (3)	0.1067 (1)	0.0672 (8)
O2	0.3900 (1)	-0.0078 (3)	0.3960 (1)	0.0487 (6)
C1	0.4539 (2)	-0.1166 (4)	0.1698 (2)	0.0499 (9)
C2	0.3992 (2)	0.0623 (4)	0.1714 (1)	0.0411 (8)
C3	0.3964 (2)	0.1952 (4)	0.1016 (2)	0.0515 (9)
C4	0.3416 (2)	0.3616 (5)	0.1038 (2)	0.0600 (11)
C5	0.2878 (2)	0.4071 (4)	0.1760 (2)	0.0590 (10)
C6	0.2936 (2)	0.2790 (4)	0.2455 (2)	0.0458 (8)
C7	0.3448 (2)	0.1103 (4)	0.2422 (1)	0.0391 (8)
C8	0.3140 (2)	0.0366 (4)	0.3310 (1)	0.0383 (8)
C9	0.2611 (2)	0.2402 (4)	0.3388 (2)	0.0479 (8)
C10	0.1516 (2)	0.2407 (5)	0.3496 (2)	0.0633 (12)
C11	0.0987 (2)	0.1012 (5)	0.2857 (2)	0.0652 (12)
C12	0.1334 (2)	-0.1035 (5)	0.3005 (2)	0.0509 (9)
C13	0.2476 (2)	-0.1375 (4)	0.3248 (2)	0.0409 (8)
C14	0.2179 (2)	-0.2190 (4)	0.4121 (2)	0.0412 (8)
C15	0.1195 (2)	-0.1884 (4)	0.3917 (2)	0.0478 (9)
C16	0.0502 (2)	-0.2357 (5)	0.4496 (2)	0.0608 (11)
C17	0.0863 (2)	-0.3198 (5)	0.5292 (2)	0.0634 (11)
C18	0.1850 (2)	-0.3548 (5)	0.5483 (2)	0.0592 (11)
C19	0.2542 (2)	-0.3047 (4)	0.4898 (2)	0.0504 (8)

Table 2. Comparison of bond distances (Å), bond angles (°) and selected torsion angles (°) with *e.s.d.*'s in parentheses

			Av.
O1—C1	1.209 (3)	—	—
O2—C8	1.413 (3)	—	—
C1—C2	1.459 (4)	—	—
C8—C13	1.519 (4)	C10—C11	1.519 (5)
C9—C10	1.526 (4)	C11—C12	1.520 (5)
C2—C3	1.404 (4)	C18—C19	1.391 (4)
C4—C5	1.400 (4)	C16—C17	1.396 (4)
C3—C4	1.387 (4)	C17—C18	1.386 (4)
C2—C7	1.393 (3)	C14—C19	1.378 (4)
C5—C6	1.378 (4)	C15—C16	1.381 (4)
C6—C7	1.377 (4)	C14—C15	1.380 (3)
C6—C9	1.537 (4)	C12—C15	1.527 (4)
C7—C8	1.527 (3)	C13—C14	1.522 (3)
C8—C9	1.607 (4)	C12—C13	1.601 (3)
O1—C1—C2	124.2 (2)	—	—
O2—C8—C7	116.5 (2)	—	—
O2—C8—C9	117.2 (2)	—	—
O2—C8—C13	105.9 (2)	—	—
C1—C2—C3	122.6 (2)	C1—C2—C7	121.8 (2)
C3—C2—C7	115.7 (2)	C14—C19—C18	115.5 (3)
C4—C5—C6	116.4 (3)	C15—C16—C17	115.5 (3)
C2—C3—C4	121.4 (2)	C17—C18—C19	121.8 (3)
C3—C4—C5	121.9 (3)	C16—C17—C18	122.2 (3)
C5—C6—C7	121.9 (2)	C14—C15—C16	122.1 (3)
C2—C7—C6	122.7 (2)	C15—C14—C19	122.8 (2)
C2—C7—C8	142.7 (2)	C13—C14—C19	143.1 (2)
C5—C6—C9	144.3 (3)	C12—C15—C16	143.6 (2)
C7—C6—C9	93.8 (2)	C12—C15—C14	94.2 (2)
C6—C7—C8	94.6 (2)	C13—C14—C15	94.1 (2)
C7—C8—C9	85.6 (2)	C12—C13—C14	86.1 (2)
C6—C9—C8	85.6 (2)	C13—C12—C15	85.6 (2)
C7—C8—C13	114.9 (2)	C8—C13—C14	116.5 (2)
C9—C8—C13	116.1 (2)	C8—C13—C12	117.9 (2)
C8—C9—C10	117.7 (2)	C11—C12—C13	117.7 (2)
C9—C10—C11	111.0 (3)	C10—C11—C12	112.4 (3)
C6—C9—C10	117.1 (2)	C11—C12—C15	116.1 (3)
C7—C8—C9—C6	178.1 (2)	C9—C10—C11—C12	60.6 (3)
C6—C9—C10—C11	55.0 (4)	C10—C11—C12—C15	60.7 (3)
C7—C8—C9—C6	3.8 (2)	C15—C12—C13—C14	0.4 (2)
C7—C8—C9—H9	-110 (2)	H12—C12—C13—C14	-113 (2)
C13—C8—C9—C10	6.5 (3)	C11—C12—C13—C8	-0.4 (4)
O2—C8—C9—H9	7 (2)	H12—C12—C13—H13	0.3 (22)

Table 3. Averaged bond lengths (Å) and bond angles (°) for benzocyclobutenes



	Compound (7)	Literature*	Calc.†
a	1.379 (2)	1.383 (3)	1.386
b	1.381 (3)	1.389 (1)	1.371
c	1.398 (3)	1.395 (2)	1.396
d	1.386 (3)	1.391 (3)	1.387
e	1.603 (3)	1.598 (4)	1.599
α	122.6 (3)	122.5 (1)	122.2
β	115.7 (2)	115.3 (1)	116.2
γ	121.8 (2)	122.1 (1)	121.5
δ	143.4 (4)	143.4 (1)	—
ε	94.2 (2)	94.1 (1)	94.0
ζ	85.6 (1)	85.8 (1)	86.0

* From 27 molecules retrieved from the Cambridge Structural Database (January 1992 release; Allen *et al.*, 1991) and including data from Ianelli *et al.* (1992a,b).

† From MO *ab initio* calculations at the 3-21G level (Benassi *et al.*, 1991).

Table 4. Analysis of the anisotropic atomic displacements in terms of LST rigid-body motion and internal motions

C1Z is the centroid of the C2,...C7 ring, P1Z is the normal to the C2,...C7 ring at C1Z, C2Z is the centroid of the C8,...C13 ring, P2Z is the normal to the C8,...C13 ring at C2Z, C3Z is the centroid of the C14,...C19 ring and P3Z is the normal to the C14,...C19 ring at C3Z. Δ is the mean difference of the mean-square vibrational amplitudes along the interatomic directions for all pairs of atoms, $\Delta U = U_{ij}(\text{obs.}) - U_{ij}(\text{calc.})$, $R_{wU} = [\sum(w\Delta U)^2 / \sum(wU_o)^2]^{1/2}$, $\sigma(w\Delta U) = [\sum(w\Delta U)^2 / \sum w^2]^{1/2}$, $\sigma(U_o)$ is the mean e.s.d. of U_o 's.

Treatment	$\Delta \times 10^4$ (Å)	$\sigma(w\Delta U) \times 10^4$	$\sigma(U_o) \times 10^4$	R_{wU}
Rigid-body	30 (41)	33	17	0.102
Internal motions		25		0.078
Group librating	Libration along	Libration amplitude (°)		
C2,...C7	C1Z—P1Z	2.1 (8)		
C8,...C13	C2Z—P2Z	2.9 (5)		
C14,...C19	C3Z—P3Z	3.2 (6)		
O1	C1—C2	4.9 (22)		
O2	C8—C9	3.3 (10)		

The integrated intensities were obtained by a modified version (Belletti, Uguzzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) peak-profile analysis procedure. All reflections were corrected for Lorentz and polarization effects but not for absorption. Correction for extinction was carried out according to Zachariassen (1963) [$g = 5.73(2) \times 10^{-8}$].

The structure was determined by direct methods using SHELXS86 (Sheldrick, 1986) and refined by anisotropic full-matrix least squares on F , using SHELX76 (Sheldrick, 1976). The H atoms were located by difference Fourier synthesis and refined isotropically.

Throughout this paper, the averaged values are means weighted according to the reciprocals of the variances and the corresponding e.s.d.'s are the largest of the 'external' and 'internal' standard deviations (Topping, 1960).

The calculations were carried out on the ENCORE-GOULD-POWERNODE 6040 and ENCORE 91 computers of the 'Centro di Studio per la Strutturistica Diffraattometrica del CNR (Parma)'. In addition to the quoted programs, LQPARM

(Nardelli & Mangia, 1984), PARST (Nardelli, 1983a) and ORTEP (Johnson, 1965) have been used.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, together with principal axes of thermal ellipsoids, statistics for the benzocyclic parameters, difference van der Waals energy profiles and results of the Cambridge Database search, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55191 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1010]

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