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4-Benzylbiphenyl

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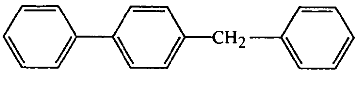
Abstract

In the title compound, $C_{19}H_{16}$, the average C—C bond length in all three phenyl rings is 1.380 (2) Å. Unlike in biphenyl itself, the magnitude of the torsion angle between the phenyl rings, C(2)—C(1)—C(7)—C(8), is −41.6 (4)°. The benzyl group is inclined towards the biphenyl moiety at a dihedral angle of 106.4 (4)°.

Comment

There is substantial literature on the studies of biphenyl and its derivatives. The work of Brock and co-workers (Brock & Haller, 1984; Brock & Morelan, 1986) on 4-hydroxybiphenyl shows that not only are some of these materials difficult to crystallize, but also that a given material may exist in more than one crystalline form. Their compound, like biphenyl itself, has the two phenyl rings coplanar in the solid state (Trotter, 1961; Hargreaves & Rizvi, 1962; Charbonneau & Delugeard, 1976; 1977), unlike in the gas phase (Almenningen & Bastiansen, 1958; Bastiansen & Traetteberg, 1962) where it displays a dihedral twist of 42°.



(I)

The range of crystallization conditions reported so far indicates to us that biphenyl systems would be good subjects for a systematic analysis of growth conditions and morphology modifiers for the preparation of organic crystals from organic solvents. The first stage in this work concerns the crystallization of 4-substituted biphenyls from non-aqueous solutions. The present work reports the crystal and molecular structure of the title compound, (I). A perspective view of the molecule with atomic labelling is given in Fig. 1 and the unit-cell packing viewed down the *b* axis is presented in Fig. 2.

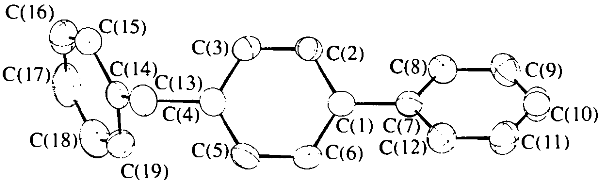


Fig. 1. A general view of 4-benzylbiphenyl. Displacement ellipsoids are plotted at the 50% probability level.

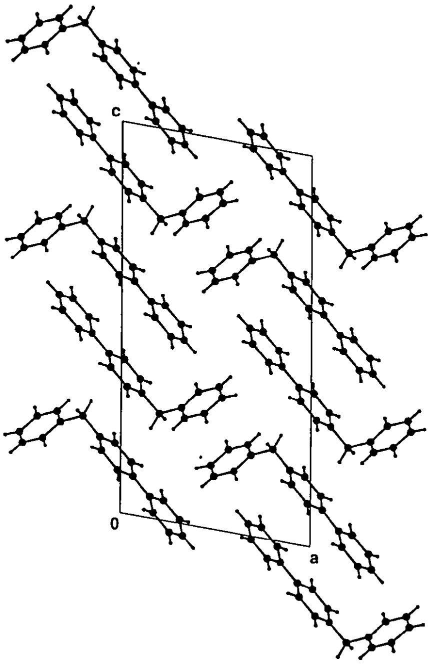


Fig. 2. Molecular packing of 4-benzylbiphenyl as seen down the *b* axis.

The average bond distances for the C(1)–C(6) and C(7)–C(12) phenyl rings are in good agreement with those of other 4-substituted biphenyls. The three internal ring bond angles, at C(1) [117.3 (2)°], C(4) [117.4 (2)°] and C(7) [117.5 (3)°], are significantly smaller than the ideal angle of 120°, but similar to those found in related compounds (Brock & Haller, 1984; Brock & Morelan, 1986). The length of the C(1)—C(7) bond [1.485 (4) Å] is quite close to the standard value for a single-bond length between trigonally linked C atoms

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(Cruickshank & Sparks, 1960). The magnitude of the torsion angle between the phenyl rings of the biphenyl moiety along C(1)—C(7) is −41.6 (4)°. The biphenyl fragment is linear, with C(13) deviating 0.029 Å from the main molecular axis. The molecules adopt a herringbone configuration in the unit cell when viewed down the *b* axis. The benzyl group is inclined at a dihedral angle of 106.35° to the adjacent phenyl group.

Experimental

The material was supplied by Aldrich and crystallization from various solvents was carried out in order to produce good quality crystals. Crystals of 4-benzylbiphenyl were grown from toluene, ethanol, methanol, chloroform and dimethylformamide at room temperature by the slow-evaporation technique. Plate-type crystals were obtained from all of these solvents except dimethylformamide, which yielded a few needle blocks and some hexagonal-shaped crystals. The preliminary examination of all the crystals obtained from different solvents revealed that unlike some of the other 4-substituted biphenyls reported in the literature (Brock & Haller, 1984; Brock & Morelan, 1986), all the methods of crystallization of this material yielded crystals with the same unit cell. Therefore, a good quality colourless rectangular-shaped plate (grown from ethanol) was chosen for data collection.

Crystal data

$C_{19}H_{16}$
$M_r = 244.3$
Monoclinic
$P2_1/c$
$a = 10.711\ (4)$ Å
$b = 6.115\ (3)$ Å
$c = 21.514\ (4)$ Å
$\beta = 99.919\ (20)^\circ$
$V = 1388.1\ (9)$ Å^3
$Z = 4$
$D_x = 1.17$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 250 reflections
$\theta = 3$–24°
$\mu = 0.613$ mm^{-1}
$T = 120$ K
Rectangular plate
0.70 × 0.30 × 0.20 mm
White

Data collection

Enraf–Nonius FAST diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5539 measured reflections
2055 independent reflections
1102 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.123$

$\theta_{\rm max} = 25^\circ$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 6$
$l = 0 \rightarrow 23$
No standards measured; absence of decay was confirmed by comparing analogous reflections from beginning and end of data collection (7 h in total)

Refinement

Refinement on F
$R = 0.0429$
$wR = 0.0510$
$S = 0.994$
1102 reflections
232 parameters

$(\Delta/\sigma)_{\rm max} = 0.162$
$\Delta\rho_{\rm max} = 1.573$ e Å^{-3}
$\Delta\rho_{\rm min} = -1.162$ e Å^{-3}
Extinction correction: secondary, Larson (1970)
Extinction coefficient: 38.6

All H-atom parameters refined
$w = [weight][1 - (\Delta F/\sigma + \sigma F)^2]^2$
weighting coefficients: 11.5, −0.68, 8.27

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	$U_{\rm eq}$
C(1)	0.0635 (2)	−0.2596 (4)	−0.9014 (1)	0.046 (3)
C(2)	0.0545 (2)	−0.0707 (5)	−0.8665 (1)	0.053 (3)
C(3)	−0.0296 (2)	−0.0566 (5)	−0.8246 (1)	0.055 (3)
C(4)	−0.1072 (2)	−0.2298 (4)	−0.8154 (1)	0.050 (3)
C(5)	−0.0981 (3)	−0.4180 (5)	−0.8499 (1)	0.057 (3)
C(6)	−0.0151 (2)	−0.4331 (5)	−0.8924 (1)	0.054 (3)
C(7)	0.1541 (2)	−0.2745 (4)	−0.9463 (1)	0.050 (3)
C(8)	0.1718 (2)	−0.0999 (5)	−0.9856 (1)	0.058 (3)
C(9)	0.2561 (3)	−0.1143 (7)	−1.0272 (1)	0.070 (3)
C(10)	0.3259 (3)	−0.3026 (7)	−1.0300 (2)	0.079 (3)
C(11)	0.3101 (3)	−0.4761 (7)	−0.9915 (2)	0.078 (3)
C(12)	0.2253 (3)	−0.4630 (5)	−0.9505 (2)	0.066 (3)
C(13)	−0.1999 (3)	−0.2096 (6)	−0.7699 (1)	0.061 (3)
C(14)	−0.3150 (2)	−0.0723 (4)	−0.7952 (1)	0.049 (3)
C(15)	−0.3322 (3)	0.1303 (5)	−0.7694 (1)	0.062 (3)
C(16)	−0.4369 (4)	0.2532 (6)	−0.7912 (2)	0.076 (3)
C(17)	−0.5273 (4)	0.1766 (8)	−0.8388 (2)	0.079 (3)
C(18)	−0.5118 (3)	−0.0232 (8)	−0.8654 (2)	0.078 (3)
C(19)	−0.4059 (3)	−0.1486 (6)	−0.8435 (1)	0.063 (3)

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

C(1)—C(2)	1.390 (3)	C(9)—C(10)	1.380 (5)
C(1)—C(6)	1.389 (4)	C(10)—C(11)	1.373 (5)
C(1)—C(7)	1.485 (4)	C(11)—C(12)	1.374 (4)
C(2)—C(3)	1.381 (4)	C(13)—C(14)	1.513 (4)
C(3)—C(4)	1.382 (4)	C(14)—C(15)	1.382 (4)
C(4)—C(5)	1.383 (4)	C(14)—C(19)	1.378 (4)
C(4)—C(13)	1.514 (4)	C(15)—C(16)	1.364 (5)
C(5)—C(6)	1.383 (4)	C(16)—C(17)	1.366 (6)
C(7)—C(8)	1.395 (4)	C(17)—C(18)	1.371 (5)
C(7)—C(12)	1.393 (4)	C(18)—C(19)	1.383 (5)
C(8)—C(9)	1.379 (4)		
C(2)—C(1)—C(6)	117.3 (2)	C(8)—C(9)—C(10)	120.1 (3)
C(2)—C(1)—C(7)	121.1 (2)	C(9)—C(10)—C(11)	119.6 (3)
C(6)—C(1)—C(7)	121.6 (2)	C(10)—C(11)—C(12)	120.4 (3)
C(1)—C(2)—C(3)	121.2 (3)	C(7)—C(12)—C(11)	121.3 (3)
C(2)—C(3)—C(4)	121.5 (3)	C(4)—C(13)—C(14)	113.4 (2)
C(3)—C(4)—C(5)	117.4 (2)	C(13)—C(14)—C(15)	120.7 (3)
C(3)—C(4)—C(13)	120.5 (3)	C(13)—C(14)—C(19)	120.6 (3)
C(5)—C(4)—C(13)	122.0 (3)	C(15)—C(14)—C(19)	118.7 (3)
C(4)—C(5)—C(6)	121.6 (3)	C(14)—C(15)—C(16)	120.9 (3)
C(1)—C(6)—C(5)	121.0 (3)	C(15)—C(16)—C(17)	120.3 (4)
C(1)—C(7)—C(8)	121.3 (2)	C(16)—C(17)—C(18)	119.8 (3)
C(1)—C(7)—C(12)	121.3 (2)	C(17)—C(18)—C(19)	120.2 (4)
C(8)—C(7)—C(12)	117.5 (3)	C(14)—C(19)—C(18)	120.0 (3)
C(7)—C(8)—C(9)	121.1 (3)		

Isotropic refinement of the structure by least-squares methods was followed by anisotropic refinement of all the non-H atoms. All H atoms were located from a difference Fourier map.

Data collection: Enraf–Nonius FAST system. Data reduction: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Structure solution: *SIR*92 (Altomare, Burla, Cascarano, Giacovazzo & Guagliardi, 1993). Structure refinement: *CRYSTALS*. Molecular graphics: *CAMERON* (Pearce, Watkin & Prout, 1992). Preparation of material for publication: *CRYSTALS*.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HU1135). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1:1 Molecular Complex of Theophylline and *p*-Nitroaniline

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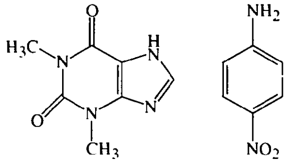
Abstract

Theophylline (3,7-dihydro-1,3-dimethyl-1*H*-purine-2,6-dione) and *p*-nitroaniline molecules in the title complex, $C_7H_8N_4O_2.C_6H_6N_2O_2$, are packed in layers. These layers are held together to form a sheet by a system of intermolecular hydrogen bonding which includes C—H···O hydrogen bonds.

Comment

The structure determination of the title crystal was undertaken during the course of a study of a number of complexes involving methylxanthine. The structure of the 2:1 complex of theophylline and 5-fluorouracil has been reported (Zaitu, Miwa & Taga, 1995), and the structures of 1:1 theophylline–*p*-nitrophenol (Aoki, Ichikawa, Koinuma & Iitaka, 1987) and 1:1 theophylline–urea (Wiedenfeld & Knoch, 1986) have also been determined. Fig. 1 shows the molecular structure of the title complex, the 1:1 molecular complex of theophylline and *p*-nitroaniline, (I).



(I)

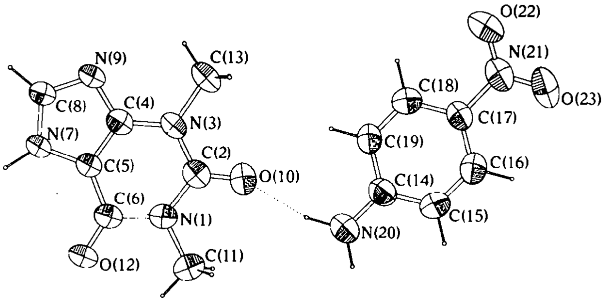


Fig. 1. Perspective view of the title complex with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The fused theophylline rings are essentially planar and the bond lengths and angles within the molecule are in fairly good agreement with those observed in theophylline monohydrate (Sutor, 1958) and other theophylline complexes, *e.g.* 1:1 theophylline–sulfathiazole (Shefter & Sackman, 1971). The *p*-nitroaniline molecule has practically a twofold axis collinear with the C—N bonds and, as has been observed in *p*-nitroaniline itself (Colapietro, Domenicano, Marciante & Portalone, 1982), the C—C bonds of the six-membered ring along the long molecular axis are shorter than the other C—C bonds. The planar molecules are packed by stacking forces and hydrogen bonds (Table 3) to form theophylline–*p*-nitroaniline layers (Fig. 2). C(8)—H···O(12) interactions are considered to be C—H···O hydrogen bonds; similar C(8)—H hydrogen bonds to