C8—C10—C1	120.90 (10)	O1′—C7′—C1′	121.40 (11)
O1—C11—N1	120.03 (10)	N1′—C7′—C1′	116.02 (10)
O1—C11—C8	122.56 (11)		

Refinement was performed on F^2 for all reflections except for five for structure (1) and six for structure (2) with very negative F^2 or flagged for potential systematic errors (*e.g.* extinction).

For both compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: AB1270). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.
Easton, C. J., Gulbis, J. M., Hoskins, B. F., Scharfbillig, I. M. & Tiekink, E. R. T. (1992). *Z. Kristallogr.* **199**, 249–254.
Ponomarev, I. I., Skuratova, N. A., Lindeman, S. V., Sinichkin, M. K., Vinogradova, S. V. & Rusanov, A. L. (1994). *Vysokomol. Soedin. Ser. A*, **36**, 730–738.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

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Biphenyl-2-methanol

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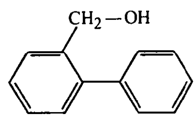
Abstract

Biphenyl-2-methanol, $C_{13}H_{12}O$, crystallizes in the non-centrosymmetric monoclinic space group *Pc* with four molecules in the asymmetric unit. However, the four independent molecules fall into two pairs related by pseudo centres. The dihedral angle between the phenyl rings in each molecule is 53.3 (1), 58.9 (1), 52.6 (1) and 59.6 (1)°; the dihedral angle between the methanol and phenyl group is 45.2 (1), 42.2 (1), 54.9 (1) and 45.4 (1)°, respectively.

Comment

Biphenyl and its derivatives have been studied extensively in the past because of the differences found in the inter-ring torsion angle φ in the solid state (Charbonneau & Delugeard, 1976, 1977; Brock, Blackburn & Haller, 1984; Brock & Haller, 1984*a*,*b*; Samdal, 1985; Brock & Minton, 1989) and in the gas phase (Almenningen & Bastiansen, 1958; Bastiansen & Traetteberg, 1962). These systems have also been investigated for those structures which pose difficulties in refinement because of a low observed-data-to-parameter ratio (a result of the crystals being non-centrosymmetric, having large overall displacement parameters and growing as thin needles or plates) (Brock, Blackburn & Haller, 1984). In a continuation of our on-going research program aimed at investigating the trends in crystallization and crystal growth of some substituted biphenyls from non-aqueous solutions (Rajnikant, Watkin & Tranter, 1995*a*,*b*,*c*), the crystal and molecular structure of the title compound, (I), is presented.



(I)

A general view of the molecule indicating the atom-numbering scheme is shown in Fig. 1 and the packing of the molecules viewed along the *a* axis is depicted in Fig. 2. The four independent molecules in the asymmetric unit are essentially identical except for the torsion angles. The average length of the bond C(*n*01)—C(*n*07) [1.454 (6) Å] is slightly less than the standard value for a single bond length between trigonally linked C atoms (1.477 Å; Cruickshank & Sparks, 1960), but is significantly shorter than the value observed

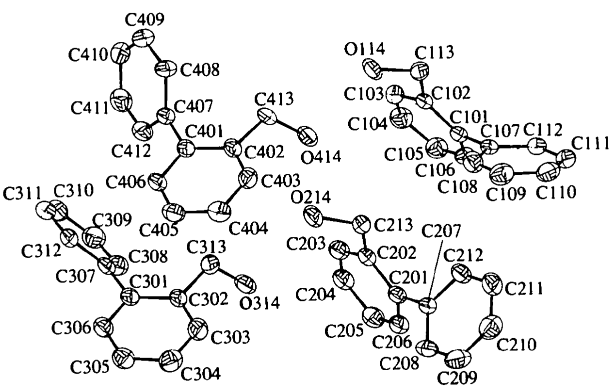


Fig. 1. Minimum overlap view of the four independent molecules in the asymmetric unit of biphenyl-2-methanol. Displacement ellipsoids are drawn at the 50% probability level.

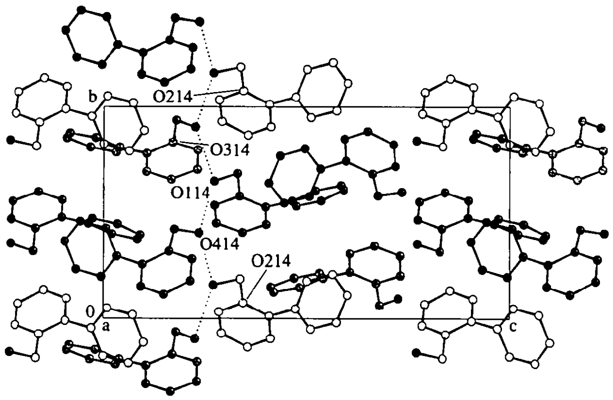


Fig. 2. Packing diagram of biphenyl-2-methanol viewed along the *a* axis. Note that there is a pseudo-screw operator relating molecule (1) to molecule (3), with a translation component of approximately 0.25, and a pseudo-screw operator relating molecule (2) to molecule (4), with a translation component of approximately 0.24. Note also that there is a translation of approximately 0.5 between corresponding atoms in the rings adjacent to O(114) and O(214), and between O(314) and O(414), but that this correspondence does not extend to the remote rings.

in the case of biphenyl (1.50 Å; Hargreaves & Rizvi, 1962; Charbonneau & Delugeard, 1976, 1977). The distribution of bond angles around C(*n*01) is quite similar to some of the reported 2-substituted biphenyls with angle C(*n*02)—C(*n*01)—C(*n*06) considerably less than 120° and C(*n*07)—C(*n*01)—C(*n*02) greater than 120° (Sutherland, Hogg & Williams, 1974; Rajnikant, Watkin & Tranter, 1995*b*).

The torsion angle C(*n*02)—C(*n*01)—C(*n*07)—C(*n*08) between the phenyl rings in each of the four independent molecules is 55.1 (1), −55.7 (1), 53.5 (1) and −61.9 (1)°; the dihedral angle between the methanol and phenyl group is 45.2 (1), 42.2 (1), 54.9 (1) and 45.4 (1)°, respectively. The four independent molecules form a continuous hydrogen-bonded chain [O(114)···O(314)(*x*, *y* + 1, *z*) 2.74 (1), O(114)···O(414) 2.72 (1), O(214)···O(314) 2.78 (1) and O(214)···O(414) 2.77 (1) Å; the H atom was not located].

There is a pseudo twofold screw axis relating molecule (1) to molecule (3) and molecule (2) to molecule (4). However, the translational component of these screws is not a rational fraction of the cell length, so that the true space group can not use this pseudosymmetry. In common with other substituted-biphenyl compounds we have examined, structures with multiple molecules in the asymmetric unit related by pseudo-operators are usually very difficult to crystallize and the crystal quality is always poor, leading to relatively poor *R* factors.

Experimental

The material, obtained from Aldrich, showed resistance to crystallization with most commonly used organic solvents except toluene, from which poor quality thin rectangular white plates were obtained.

Crystal data

$C_{13}H_{12}O$
$M_r = 183.2$
Monoclinic
Pc
$a = 10.712\ (4)$ Å
$b = 9.902\ (1)$ Å
$c = 19.562\ (3)$ Å
$\beta = 101.25\ (2)°$
$V = 2035.1\ (9)$ Å^3
$Z = 8$
$D_x = 1.20$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 20$–$39°$
$\mu = 0.54$ mm^{-1}
$T = 290$ K
Rectangular plate
0.80 × 0.50 × 0.15 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3989 measured reflections
3300 independent reflections
2440 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.046$
$\theta_{max} = 72°$
$h = -12 \rightarrow 11$
$k = -11 \rightarrow 10$
$l = -5 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity decay: 5%

Refinement

Refinement on *F*
$R = 0.0706$
$wR = 0.0764$
$S = 1.18$
2440 reflections
514 parameters
H-atom parameters not refined
Weighting scheme: 3-term Chebychev (12.8, 6.44, 10.7)

$(\Delta/\sigma)_{max} = 0.0795$
$\Delta\rho_{max} = 0.441$ e Å^{-3}
$\Delta\rho_{min} = -0.425$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 36.3 (99)
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)

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

	x	*y*	*z*	U_{eq}
C(101)	0.6265 (5)	0.5255 (6)	0.7971 (3)	0.0416
C(102)	0.5654 (5)	0.5775 (6)	0.7326 (3)	0.0405
C(103)	0.6043 (6)	0.5343 (7)	0.6728 (3)	0.0527
C(104)	0.7080 (7)	0.4484 (8)	0.6768 (3)	0.0630
C(105)	0.7732 (6)	0.4036 (7)	0.7398 (3)	0.0635
C(106)	0.7320 (5)	0.4420 (6)	0.7986 (3)	0.0505
C(107)	0.5863 (5)	0.5543 (6)	0.8626 (3)	0.0434
C(108)	0.4630 (5)	0.5305 (6)	0.8727 (3)	0.0612
C(109)	0.4253 (6)	0.5563 (7)	0.9345 (3)	0.0696
C(110)	0.5116 (6)	0.6078 (8)	0.9896 (4)	0.0656
C(111)	0.6359 (6)	0.6289 (6)	0.9830 (3)	0.0634
C(112)	0.6717 (6)	0.6033 (6)	0.9205 (3)	0.0539
C(113)	0.4587 (7)	0.6861 (7)	0.7275 (4)	0.0482
O(114)	0.3566 (5)	0.6496 (5)	0.6717 (4)	0.0642
C(201)	0.6119 (6)	0.0113 (6)	0.8065 (3)	0.0454
C(202)	0.5714 (6)	0.0749 (6)	0.7416 (3)	0.0442
C(203)	0.6166 (7)	0.0325 (7)	0.6834 (3)	0.0561
C(204)	0.7092 (6)	−0.0683 (7)	0.6898 (4)	0.0586
C(205)	0.7512 (6)	−0.1334 (7)	0.7531 (3)	0.0595
C(206)	0.7014 (6)	−0.0934 (6)	0.8097 (3)	0.0543
C(207)	0.5712 (6)	0.0457 (6)	0.8707 (3)	0.0474
C(208)	0.5170 (6)	−0.0494 (6)	0.9092 (3)	0.0610
C(209)	0.4860 (7)	−0.0174 (7)	0.9722 (3)	0.0734
C(210)	0.5066 (8)	0.1117 (7)	0.9993 (4)	0.0751
C(211)	0.5596 (6)	0.2087 (6)	0.9629 (3)	0.0673

C(212)	0.5901 (6)	0.1764 (5)	0.8998 (3)	0.0556
C(213)	0.4662 (7)	0.1864 (7)	0.7343 (4)	0.0501
O(214)	0.3793 (6)	0.1592 (5)	0.6697 (4)	0.0722
C(301)	0.0188 (5)	0.2197 (6)	0.5053 (3)	0.0471
C(302)	0.0822 (7)	−0.1677 (7)	0.5699 (3)	0.0490
C(303)	0.0397 (7)	−0.2026 (7)	0.6304 (4)	0.0637
C(304)	−0.0600 (7)	−0.2947 (8)	0.6294 (3)	0.0677
C(305)	−0.1258 (6)	−0.3416 (7)	0.5660 (3)	0.0614
C(306)	−0.0872 (5)	−0.3050 (6)	0.5054 (3)	0.0548
C(307)	0.0586 (5)	−0.1911 (6)	0.4395 (3)	0.0464
C(308)	0.1827 (5)	−0.2139 (6)	0.4308 (3)	0.0599
C(309)	0.2184 (6)	−0.1910 (7)	0.3673 (3)	0.0712
C(310)	0.1299 (6)	−0.1485 (8)	0.3100 (4)	0.0689
C(311)	0.0062 (6)	−0.1251 (6)	0.3173 (3)	0.0643
C(312)	−0.0308 (5)	−0.1469 (5)	0.3805 (3)	0.0518
C(313)	0.1807 (8)	−0.0631 (8)	0.5784 (5)	0.0593
O(314)	0.2884 (5)	−0.0951 (5)	0.6243 (3)	0.0648
C(401)	0.0259 (6)	0.2543 (6)	0.4955 (3)	0.0445
C(402)	0.0684 (6)	0.3153 (7)	0.5610 (3)	0.0469
C(403)	0.0122 (6)	0.2794 (7)	0.6171 (3)	0.0583
C(404)	−0.0788 (7)	0.1774 (7)	0.6127 (4)	0.0667
C(405)	−0.1172 (6)	0.1149 (7)	0.5487 (3)	0.0606
C(406)	−0.0663 (6)	0.1517 (5)	0.4920 (3)	0.0517
C(407)	0.0716 (6)	0.2922 (6)	0.4331 (3)	0.0469
C(408)	0.0527 (6)	0.4236 (6)	0.4059 (3)	0.0556
C(409)	0.0866 (6)	0.4551 (6)	0.3428 (3)	0.0658
C(410)	0.1414 (7)	0.3604 (7)	0.3048 (4)	0.0674
C(411)	0.1621 (7)	0.2310 (6)	0.3327 (3)	0.0710
C(412)	0.1262 (6)	0.1967 (6)	0.3945 (3)	0.0593
C(413)	0.1674 (7)	0.4196 (7)	0.5739 (5)	0.0549
O(414)	0.2562 (5)	0.4022 (6)	0.6335 (3)	0.0664

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *CRYSTALS*. Molecular graphics: *CAMERON* (Pearce, Watkin & Prout, 1992). Software used to prepare material for publication: *CRYSTALS*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1180). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Almenningen, A. & Bastiansen, O. (1958). *K. Nor. Vidensk. Selsk. Skr.* No. 4.
Bastiansen, O. & Traetteberg, M. (1962). *Tetrahedron*, **17**, 147–154.
Brock, C. P., Blackburn, J. R. & Haller, K. L. (1984). *Acta Cryst.* B**40**, 493–498.
Brock, C. P. & Haller, K. L. (1984*a*). *Acta Cryst.* C**40**, 1387–1390.
Brock, C. P. & Haller, K. L. (1984*b*). *J. Phys. Chem.* **88**, 3570–3574.
Brock, C. P. & Minton, P. (1989). *J. Am. Chem. Soc.* **111**, 4586–4593.
Charbonneau, G. P. & Delugeard, Y. (1976). *Acta Cryst.* B**32**, 1420–1423.
Charbonneau, G. P. & Delugeard, Y. (1977). *Acta Cryst.* B**33**, 1586–1588.
Cruickshank, D. W. J. & Sparks, R. A. (1960). *Proc. R. Soc. London Ser. A*, **258**, 270–285.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5. Enraf–Nonius, Delft, The Netherlands.
Hargreaves, A. & Rizvi, S. H. (1962). *Acta Cryst.* **15**, 365–373.
Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
Pearce, L., Watkin, D. J. & Prout, C. K. (1992). *CAMERON. Program for Plotting Molecular Structures*. Chemical Crystallography Laboratory, Univ. of Oxford, England.
Rajnikant, Watkin, D. J. & Tranter, G. (1995*a*). *Acta Cryst.* C**51**, 1452–1454.
Rajnikant, Watkin, D. J. & Tranter, G. (1995*b*). *Acta Cryst.* C**51**. In the press.
Rajnikant, Watkin, D. J. & Tranter, G. (1995*c*). *Acta Cryst.* C**51**. In the press.
Samdal, S. (1985). *J. Mol. Struct.* **128**, 115–125.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Sutherland, H. H., Hogg, J. H. C. & Williams, D. J. (1974). *Acta Cryst.* B**30**, 1562–1565.
Watkin, D. J., Carruthers, J. R. & Betteridge, P. W. (1985). *CRYSTALS User's Guide*. Chemical Crystallography Laboratory, Univ. of Oxford, England.

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Studies on Avarol Derivatives. 2′,5′-Diacetylavarol from *Dysidea Avara*

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Abstract

The X-ray analysis of the natural product 2′,5′-diacetylavarol [De Giulio, De Rosa, Di Vincenzo & Strazzullo (1990). *Tetrahedron*, **46**, 7971–7976], 2-[(1,2,3,4,4a,7,8,8a-octahydro-1,2,4a,5-tetramethyl-1-naphthyl)methyl]-1,4-benzenediyl diacetate, $C_{25}H_{34}O_4$, is reported. Short intramolecular contacts between the bulky substituents of the bicyclic system cause significant distortions of the molecular geometry. The $\Delta^{3,4}$ cyclohexene ring adopts a conformation intermediate between half chair and half boat, and the cyclohexane ring is in a nearly ideal chair conformation. The hydroquinone system is almost perpendicular to the *trans*-fused sesquiterpene residue. The acetyl groups are