angle between them, 94°, is in the reported range of other $Ph_2P-R-PPh_2$ ligands [73·5–107·6° for $R = -(CH_2)_2-$] though wider than that reported for $[Rh_2\{Ph_2P(CH_2)_4PPh_2\}(\eta\text{-}C_5H_5)_2(CO)_2]$ (70·2°). The (Ph)C—C(Ph) distances are in the range 1·351 (8)–1·399 (8) Å with mean value of 1·380 Å. (Ph)C—C(Ph)—C(Ph) angles are in the range 117·7 (5)–121·6 (5)° with the angle centered on C atoms bonded to P [117·7 (5)°] presenting its usual characteristic of being less than 120° (Rivera, Rodulfo de Gil & Fontal, 1985).

The (Bu)C—P distance [1·838 (5) Å] can be considered identical to the two (Ph)C—P bonds [1·829 (5) and 1·839 (5) Å].

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References

Faraone, F., Bruno, G., Tresoldi, G., Faraone, G. & Bombieri, G. (1981). *J. Chem. Soc. Dalton Trans.* pp. 1651–1656.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Rivera, A. V., Rodulfo de Gil, E. & Fontal, B. (1985). *Inorg. Chim. Acta*, **98**, 153–160.

Sheldrick, G. M. (1977). *SHELX77.* Program for crystal structure determination. Univ. of Cambridge, England.

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Structure of 3,3-Diphenyltetrahydrofuran

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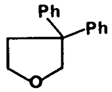
Abstract. $C_{16}H_{16}O$, $M_r = 224{\cdot}3$, monoclinic, $P2_1$, $a = 9{\cdot}252\,(3)$, $b = 7{\cdot}060\,(5)$, $c = 10{\cdot}246\,(3)$ Å, $\beta = 112{\cdot}65\,(3)°$, $V = 617{\cdot}6\,(5)$ Å^3, $Z = 2$, $D_x = 1{\cdot}206$ Mg m^{-3}, $\lambda(\mathrm{Mo}\,K\alpha_1) = 0{\cdot}70926$ Å, $\mu = 0{\cdot}39$ mm^{-1}, $F(000) = 240$, $T = 293$ K, $R = 0{\cdot}049$ for 1257 observed reflections. The phenyl-substituted carbon in the furan ring is above the plane formed by the other four members of the ring. The planes of the two phenyl rings meet at an angle of 77° and make angles of 153 and 82° with the best plane of the furan ring. The bis-benzylic C—C bond forms an angle of 62° with the plane of one of the phenyl rings.

Introduction. The interest in the structure of 3,3-diphenyltetrahydrofuran (I) arises from its photosensitized (electron-transfer) reaction (Arnold, Fahie, Lamont, Wierzchowski & Young, 1987) using 1,4-dicyanobenzene as an electron-accepting photosensitizer and acetonitrile–methanol as solvent. The radical cation of (I) should be able to attain a conformation which would allow sufficient overlap of the SOMO, associated primarily with the diphenylmethyl moiety, with the σ-orbital of the benzylic C—C bond so that cleavage will occur. The objective of the present X-ray structure analysis is to determine the preferred conformation of (I) in the solid state in order to determine the potential for orbital overlap.



(1)

Experimental. Crystals provided by D. R. Arnold and L. J. Lamont, Department of Chemistry, Dalhousie University, grown from 95% ethanol. CAD-4 diffractometer, crystal dimensions 0·2 × 0·3 × 0·3 mm, graphite-monochromatized radiation, cell parameters refined from θ values of 25 reflections ($10 \leq \theta \leq 14°$), ω–2θ scans, three standard reflections (401, $\bar{2}32$, 104), < 2% intensity variation. Intensities reduced to a standard scale using routine procedures (Cameron & Cordes, 1979). Corrections for Lorentz and polarization factors applied, but no corrections made for absorption or extinction. 1558 reflections, $2\theta < 50°$ [h −11 to 11, k −1 to 8, l 0 to 12, 1257 (81%) with $I \geq 3\sigma(I)$, $R_{\text{int}} = 0{\cdot}016$].

Structure solved by a multisolution application of the tangent formula (Karle & Hauptman, 1958) using

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Table 1. *Positional* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2)

U_{eq} is defined as $(U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}
C(1)	7332 (3)	−963 (6)	−2421 (3)	0·0423
C(2)	8118 (4)	−2804 (6)	−1664 (3)	0·0539
C(3)	7865 (4)	−4129 (6)	−2873 (4)	0·0677
O(4)	6431 (3)	−3576 (5)	−3979 (3)	0·0764
C(5)	5873 (4)	−1927 (7)	−3534 (4)	0·0569
C(11)	6862 (4)	389	−1539 (4)	0·0475
C(12)	5667 (4)	1694 (7)	−2174 (4)	0·0652
C(13)	5237 (6)	2994 (9)	−1395 (7)	0·0898
C(14)	6010 (6)	3043 (9)	50 (6)	0·1047
C(15)	7185 (5)	1818 (9)	698 (5)	0·0892
C(16)	7630 (4)	462 (7)	−77 (4)	0·0635
C(21)	8334 (4)	74 (5)	−3064 (3)	0·0448
C(22)	7741 (4)	824 (7)	−4394 (4)	0·0662
C(23)	8671 (7)	1860 (8)	−4900 (5)	0·0926
C(24)	10211 (8)	2133 (8)	−4133 (7)	0·1039
C(25)	10829 (5)	1415 (8)	−2782 (6)	0·0892
C(26)	9908 (4)	408 (7)	−2259 (4)	0·0658

Table 2. *Interatomic distances* (Å) *and angles* (°)

C(1)–C(2)	1·545 (6)	C(13)–C(14)	1·375 (9)
C(1)–C(5)	1·549 (5)	C(14)–C(15)	1·347 (8)
C(1)–C(11)	1·490 (5)	C(15)–C(16)	1·402 (8)
C(1)–C(21)	1·517 (6)	C(21)–C(22)	1·365 (5)
C(2)–C(3)	1·497 (6)	C(21)–C(26)	1·390 (5)
C(3)–O(4)	1·428 (5)	C(22)–C(23)	1·374 (9)
O(4)–C(5)	1·418 (6)	C(23)–C(24)	1·350 (9)
C(11)–C(12)	1·393 (5)	C(24)–C(25)	1·374 (9)
C(11)–C(16)	1·390 (5)	C(25)–C(26)	1·368 (8)
C(12)–C(13)	1·372 (9)		

C(2)–C(1)–C(5)	96·2 (4)	C(11)–C(12)–C(13)	121·8 (5)
C(2)–C(1)–C(11)	115·2 (3)	C(12)–C(13)–C(14)	119·7 (7)
C(2)–C(1)–C(21)	112·1 (4)	C(13)–C(14)–C(15)	120·0 (7)
C(5)–C(1)–C(11)	110·7 (3)	C(14)–C(15)–C(16)	121·1 (6)
C(5)–C(1)–C(21)	113·4 (4)	C(11)–C(16)–C(15)	119·7 (5)
C(11)–C(1)–C(21)	108·9 (3)	C(1)–C(21)–C(22)	122·9 (4)
C(1)–C(2)–C(3)	102·5 (4)	C(1)–C(21)–C(26)	120·0 (4)
C(2)–C(3)–O(4)	106·7 (4)	C(22)–C(21)–C(26)	116·9 (4)
C(3)–O(4)–C(5)	108·1 (4)	C(21)–C(22)–C(23)	121·0 (5)
C(1)–C(5)–O(4)	106·0 (4)	C(22)–C(23)–C(24)	122·0 (7)
C(1)–C(11)–C(12)	120·3 (3)	C(23)–C(24)–C(25)	118·1 (7)
C(1)–C(11)–C(16)	122·0 (3)	C(24)–C(25)–C(26)	120·4 (6)
C(12)–C(11)–C(16)	117·6 (3)	C(21)–C(26)–C(25)	121·6 (5)

*SHELX*76 (Sheldrick, 1976). An *E* map calculated from the set of phases with the highest figure of merit revealed a distorted phenyl ring. Fourier synthesis revealed in stages first the furan ring and finally the second phenyl ring. Origin in polar space group fixed by holding y value of C(11) constant. Anisotropic temperature factors for C and O atoms, isotropic for H. H positions from difference Fourier map calculated at $R = 0{\cdot}105$. Full-matrix least squares, refined on F. C—H geometry fixed during refinement to ensure convergence (C—H bond lengths held to 1·08 Å). $w = [\sigma^2(F_o) + 0{\cdot}0003F_o^2]^{-1}$, final $R = 0{\cdot}049$, $wR = 0{\cdot}049$ for 1257 observed reflections and 218 least-squares parameters, $R = 0{\cdot}072$ for all 1558 reflections; $(\Delta/\sigma)_{\max} = 0{\cdot}44$, final difference Fourier map featureless, with maximum and minimum peaks of 0·22 and −0·15 e Å^{-3}. 020 and 120 reflections suppressed owing to suspected secondary-extinction effects. Scattering factors as in *SHELX*76. Calculations performed on a Perkin–Elmer 3230 computer.

Discussion. Final atomic parameters are in Table 1.* Bond lengths and angles are given in Table 2. A stereoview of the molecule is shown in Fig. 1 with the atom labelling indicated. The packing diagram, projected down the unique b axis, is shown in Fig. 2.

The furan ring is non-planar, with the disubstituted C(1) lying 0·7 (1) Å above the plane defined by C(2)—C(3)—O(4)—C(5). The two phenyl rings are planar, with average deviations from planarity of 0·01 (2) Å. C(1) lies in the planes of both phenyl rings, which meet at an angle of 77 (1)°. The plane of phenyl ring 1 [C(11) to C(16)] meets the best plane through the furan ring at an angle of 153 (1)° while phenyl ring 2 meets the furan plane at an angle of 82 (1)°. All C—C bonds in the phenyl rings are between 1·347 (8) and 1·402 (8) Å and internal angles lie between 116·9 and 122·9°.

The two C—O bonds in the furan ring are the same [C(3)–O(4): 1·428 (5); O(4)–C(5): 1·418 (6) Å] as

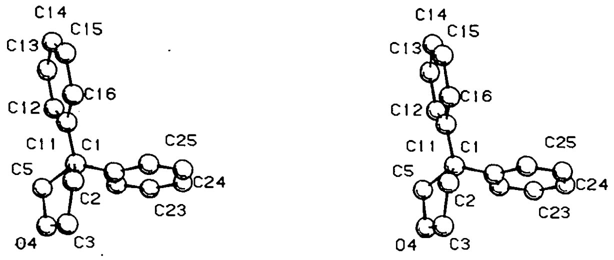

Fig. 1. Stereoview of the title molecule.

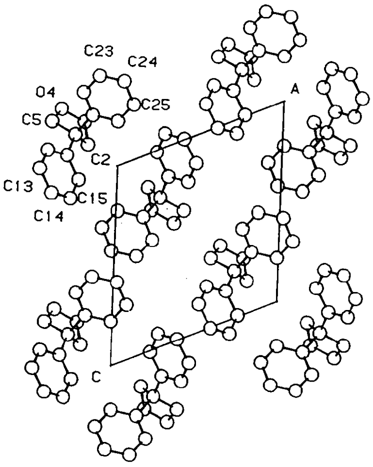

Fig. 2. Packing diagram viewed down b axis.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44387 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

are the two C—C bonds involving the phenyl-substituted C(1) [C(1)—C(2): 1·545 (6); C(1)—C(5): 1·549 (5) Å]. However, the third C—C bond in the furan ring, C(2)—C(3), is considerably shorter than this at 1·497 (6) Å. The angles within the ring range from a low of 96·2 (4)° at C(1) to a high of 108·1 (4)° at the oxygen.

These values are fairly typical of substituted furans, where disubstitution of any moiety at a carbon center tends to decrease the angle at that carbon. In α-2-ethyl-5-methyl-3,3-diphenyltetrahydrofuran (Singh & Ahmed, 1969), the angle at the phenyl-disubstituted carbon is slightly larger than that reported here, 99·4 (2)°, while the angle at the oxygen is also larger, 110·8 (2)°. However, the two C—O bonds in that structure, while equivalent, are considerably longer than those found in (I), averaging 1·445 (4) Å. The C—C bond not involving the phenyl-substituted carbon in that compound is also significantly shorter than the others in the furan ring.

The small value of the bond angle inside the furan ring at C(1) is consistent with other disubstituted carbon atoms in furan rings, and is not limited to those with bulky substituents. The angles at the substituted carbons in 3,3,4,4-tetrahydrofurantetrol (Mighell & Jacobson, 1964) are 6° smaller than those at the other two carbon centers in the ring, and 10° smaller than the angle at the O atom. These angles, at 100·3 (3)°, are slightly larger than in phenyl-substituted furans.

The angle at the oxygen atom tends to be larger than the four internal carbon angles in similar compounds. In 2,3,3,4,4,5-hexamethoxytetrahydrofuran (Iten, Weber & Eugster, 1976) the C—O—C angle is 112·6°, 7° larger than any of the O—C—C or C—C—C angles. Similarly, in (2*S*,4*R*,5*R*)-2-carboxymethyl-5-carboxy-2,4,5-trimethyl-2,3,4,5-tetrahydrofuran (Kirfel, Will, Wiedenfeld & Roeder, 1980), the C—O—C angle is 5·3° larger than all other internal ring angles.

There are no unusually close non-bonded contacts between molecules of (I).

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References

Arnold, D. R., Fahie, B. J., Lamont, L. J., Wierzchowski, J. & Young, K. M. (1987). In the press.

Cameron, T. S. & Cordes, R. E. (1979). *Acta Cryst.* B**35**, 748–750.

Iten, P. X., Weber, H. P. & Eugster, C. H. (1977). *Helv. Chim. Acta*, **60**, 410–416.

Karle, J. & Hauptman, H. (1958). *Acta Cryst.* **11**, 264–266.

Kirfel, A., Will, G., Wiedenfeld, H. & Roeder, E. (1980). *Cryst. Struct. Commun.* **9**, 363–369.

Mighell, A. D. & Jacobson, R. A. (1964). *Acta Cryst.* **17**, 1554–1560.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Singh, P. & Ahmed, F. R. (1969). *Acta Cryst.* B**25**, 2401–2408.

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Structure of the Dipeptide L-Prolyl-L-lysine Acetate. A New Conformation of the Lysine Side Chain

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Abstract. $C_{11}H_{22}N_3O_3.C_2H_3O_2$, $M_r = 303\cdot36$, monoclinic, $P2_1$, $a = 15\cdot712\ (3)$, $b = 8\cdot927\ (2)$, $c = 5\cdot484\ (2)$ Å, $\beta = 94\cdot31\ (2)°$, $V = 767\ (2)$ Å^3, $Z = 2$, $D_x = 1\cdot31$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0\cdot109$ mm^{-1}, $F(000) = 328\cdot0$, room temperature. The crystal structure was solved by direct methods and refined to an R value of 0·043 using the full-matrix least-squares method for 907 observed reflections. The molecule exists as a zwitterion with the N atom of the lysine side chain protonated. The pyrrolidine ring has an envelope conformation (N1, C*A*1, C*B*1 and C*D*1 atoms in the same plane and C*G*1 in position *exo* to

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