C15	−0.0668 (2)	0.2126 (2)	0.7474 (1)	3.88 (4)
C16	0.0191 (3)	0.5595 (2)	0.4521 (1)	5.0 (1)
C17	0.0614 (3)	0.5868 (2)	0.5050 (1)	5.3 (1)
C18	0.0430 (3)	0.5289 (2)	0.5519 (1)	5.5 (1)
C19	0.0424 (3)	0.6218 (3)	0.4007 (1)	7.6 (1)

Table 2. *Bond lengths* (Å) *and angles* (°)

P1—N1	1.574 (2)	P1—O2	1.601 (2)
P1—O1	1.601 (2)	P2—O3	1.604 (2)
P2—N1	1.567 (2)	O1—C2	1.402 (3)
N2—P1	1.571 (2)	O3—C12	1.395 (3)
O2—C3	1.401 (3)	C1—C9	1.420 (4)
C1—C2	1.349 (3)	C3—C4	1.343 (3)
C2—C3	1.393 (3)	C5—C6	1.356 (4)
C4—C10	1.418 (3)	C6—C7	1.391 (5)
C5—C10	1.413 (4)	C8—C9	1.416 (4)
C7—C8	1.357 (4)	C11—C12	1.344 (4)
C9—C10	1.418 (5)	C12—C12^{ii}	1.392 (3)
C11—C15	1.421 (4)	C13—C13^{ii}	1.392 (5)
C13—C14	1.357 (4)	C15—C15^{ii}	1.423 (3)
C14—C15	1.405 (4)	C16—C19	1.501 (4)
C16—C18^{i}	1.382 (4)	C17—C18	1.373 (4)
N1—P1—N2	116.8 (1)	N1—P1—O1	110.1 (1)
N1—P1—O2	110.2 (1)	N2—P1—O1	110.52 (9)
N2—P1—O2	110.17 (9)	O1—P1—O2	97.2 (1)
N1—P2—O3	110.2 (1)	N1—P2—N1^{ii}	116.8 (1)
N1—P2—O3^{ii}	110.3 (1)	O3—P2—N1	110.4 (1)
O3—P2—O3^{ii}	97.0 (1)	P1—N1—P2	123.3 (1)
P1—N2—P1^{ii}	123.1 (2)	P1—O1—C2	109.5 (2)
P1—O2—C3	110.1 (2)	P2—O3—C12	110.2 (2)
C2—C1—C9	117.6 (2)	O1—C2—C1	126.4 (2)
O1—C2—C3	111.5 (2)	C1—C2—C3	122.1 (2)
O2—C3—C2	110.7 (2)	O2—C3—C4	126.7 (2)
C2—C3—C4	122.6 (2)	C3—C4—C10	118.0 (2)
C6—C5—C10	120.6 (3)	C5—C6—C7	120.9 (3)
C6—C7—C8	120.2 (2)	C7—C8—C9	121.2 (3)
C1—C9—C8	121.7 (3)	C1—C9—C10	120.2 (2)
C8—C9—C10	118.2 (2)	C4—C10—C5	121.5 (3)
C4—C10—C9	119.6 (2)	C5—C10—C9	118.9 (2)
C12—C11—C15	117.6 (2)	O3—C12—C11	126.2 (2)
O3—C12—C12^{ii}	111.2 (2)	C11—C12—C12^{ii}	122.5 (2)
C14—C13—C13^{ii}	120.0 (3)	C13—C14—C15	121.8 (3)
C11—C15—C14	121.9 (2)	C11—C15—C15^{ii}	119.9 (2)
C14—C15—C15^{ii}	118.2 (2)	C17—C16—C18^{i}	117.1 (2)
C17—C16—C19	121.4 (3)	C16—C17—C18	121.6 (3)
C19—C16—C18^{i}	121.5 (2)	C17—C18—C16^{i}	121.3 (2)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $-x, y, \frac{3}{2}-z$.

D_m was determined by flotation in an *n*-heptane/carbon tetrachloride mixture. The space group *Pbcn* was determined from systematic absences (0*kl*, *k* odd; *h*0*l*, *l* odd; *hk*0, *h* + *k* odd; *h*00, *h* odd; 0*k*0, *k* odd; 00*l*, *l* odd). A Lorentz–polarization correction was applied but no absorption or extinction corrections. The structure was solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by a block-diagonal least-squares method. The C, N, O and P atoms were refined with anisotropic temperature factors. H atoms were determined from difference Fourier syntheses and refined with isotropic temperature factors. N2 and P2 were fixed on the twofold axes. All computations were performed on a FACOM M382 computer at the Data Processing Center of Kyoto University using *KPPXRAY* (Taga, Masuda, Higashi & Iizuka, 1991).

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55579 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1012]

References

Allcock, H. R. (1984). *Inclusion Compounds*, Vol. 1, edited by J. L. Atwood, J. E. D. Davies & D. D. MacNicol, pp. 351–374. London: Academic Press.

Allcock, H. R. & Kugel, R. L. (1966). *Inorg. Chem.* **5**, 1016–1020.

Allcock, H. R. & Stein, M. T. (1974). *J. Am. Chem. Soc.* **96**, 49–52.

Allcock, H. R. Stein, M. T. & Bissell, E. C. (1974). *J. Am. Chem. Soc.* **96**, 4795–4800.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN*78. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Taga, T., Masuda, K., Higashi, T. & Iizuka, H. (1991). *KPPXRAY. Kyoto Program Package for X-ray Crystal Structure Analysis.* Kyoto Univ., Japan.

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Structure of 2-Phenylacetophenone

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Abstract

The crystal structure of 2-phenylacetophenone (C_6H_5—CH_2—CO—C_6H_5) (CAS registry number 451-40-1) has been determined from 945 measured X-ray reflections with $I \geq \sigma(I)$. The dihedral angle between the two benzene ring planes is 64.2°.

Comment

This work was undertaken as part of a study of the structure of thermotropic liquid crystals. Typical thermotropic liquid-crystal molecules are composed of a rigid central core with two flexible aliphatic chains attached at either end. We are interested in correlating the packing and conformation of a particular core in its crystalline form (2-phenylacetophenone) with the appearance of liquid-crystal phases in molecules containing that core (Demus, Demus & Zashke, 1974). Fig. 1 shows the molecular structure of 2-phenylacetophenone and the numbering scheme employed. The material was obtained from the Aldrich Chemical Co. and recrystallized from hot methanol.

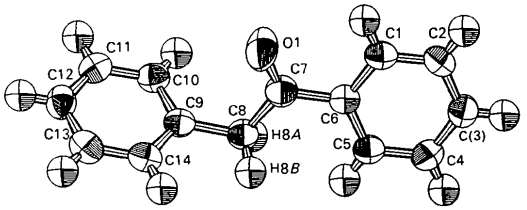

Fig. 1. Molecular structure (*ORTEP*II; Johnson, 1976) of 2-phenylacetophenone and the numbering scheme used. The thermal ellipsoids are plotted with 50% probability.

The molecular dimensions are typical (Table 2) with aromatic C—C bonds ranging from 1.371 to 1.398 Å, non-aromatic C—C bonds ranging from 1.486 to 1.502 Å and a C—O length of 1.220 Å. The ring angles where the bridging group [C(6)—C(9) and O(1)] attaches are smaller than 120° by up to 10σ. All other bond angles are as expected.

This molecule can be divided into three planar sections: the phenyl rings and the bridging group. There is an angle of 64.2° between the two phenyl ring planes. The plane of the bridging group is twisted by $-8.2°$ from the plane of the first phenyl ring [C(1)–C(6)] and 72.3° from the second [C(9)–C(14)]. These values are very similar to those observed in phenyl benzoate (C_6H_5—COO—C_6H_5) and benzoin [C_6H_5—CH(OH)—CO—C_6H_5] [55.7, -9.8 and 65.1° respectively for phenyl benzoate (Adams & Morsi, 1976); 64.5, -12 and 72.2° for benzoin (Haisa, Kashino & Morimoto, 1980)]. It is surprising that the angles for 2-phenylacetophenone and benzoin are so similar as an H atom is replaced by a much larger O atom.

Fig. 2 shows the packing of the non-H atoms in the unit cell. The nearest neighbors are arranged with their long axes roughly parallel and the phenyl rings stacked in herringbone pattern. Finally, we note the prolate spheroid for the O atom (see Figs. 1 and 2) which is elongated approximately normal to the long axis of the molecule.

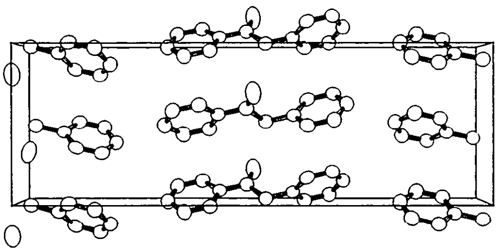

Fig. 2. Packing diagram showing the non-H atoms and the unit cell (viewed down the *b* axis).

Experimental

Crystal data

$C_{14}H_{12}O$
$M_r = 196.25$
Orthorhombic
$Pca2_1$
$a = 8.028$ (4) Å
$b = 5.456$ (3) Å
$c = 24.035$ (9) Å
$V = 1052.8$ (9) Å^3
$Z = 4$
$D_x = 1.238$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 10.14$–13.68°
$\mu = 0.071$ mm^{-1}
$T = 223$ K
Platelet
0.80 × 0.74 × 0.08 mm
Colorless

Data collection

Nicolet *P3F* diffractometer
ω scans
Absorption correction: none
3501 measured reflections
957 independent reflections
945 observed reflections [$I \geq \sigma(I)$]
$R_{\rm int} = 0.0267$

$\theta_{\rm max} = 25.0°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 6$
$l = 0 \rightarrow 28$
3 standard reflections monitored every 97 reflections intensity variation: 3%

Refinement

Refinement on F
Final $R = 0.033$
$wR = 0.037$
$S = 2.79$
945 reflections
137 parameters
H-atom parameters not refined
$w = [2F_o/(\sigma(F_o^2))]^2$
$(\Delta/\sigma)_{\rm max} = 0.003$

$\Delta\rho_{\rm max} = 0.109$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.161$ e Å^{-3}
Extinction correction: type II (Zachariasen, 1967; Larson, 1967)
Extinction coefficient: 8.6×10^{-5} (mm)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}$
O1	0.30725 (31)	0.9691 (5)	0.4999 (4)	10.70
C1	0.38168 (27)	0.9455 (4)	0.6132 (4)	5.31
C2	0.41658 (30)	0.9204 (4)	0.6689 (4)	5.58
C3	0.50863 (29)	0.7236 (4)	0.6878 (4)	5.78
C4	0.56735 (31)	0.5518 (4)	0.6501 (4)	5.89
C5	0.53353 (28)	0.5756 (4)	0.5944 (4)	5.16
C6	0.43983 (26)	0.7725 (4)	0.5749 (4)	4.74

C7	0.40283 (32)	0.8081 (5)	0.5148 (4)	5.62
C8	0.48586 (29)	0.6440 (4)	0.4730 (4)	5.16
C9	0.44540 (25)	0.6933 (4)	0.4134 (4)	4.67
C10	0.50690 (28)	0.8997 (4)	0.3862 (4)	5.25
C11	0.47195 (29)	0.9402 (4)	0.3309 (4)	5.67
C12	0.37303 (30)	0.7784 (5)	0.3015 (4)	5.88
C13	0.30981 (27)	0.5739 (4)	0.3281 (4)	5.78
C14	0.34644 (27)	0.5317 (4)	0.3833 (4)	5.22
H1A*	0.3182	1.0829	0.6000	5.48
H2A*	0.3752	1.0391	0.6950	5.48
H3A*	0.5326	0.7053	0.7267	5.48
H4A*	0.6313	0.4151	0.6632	5.48
H5A*	0.5759	0.4567	0.5686	5.48
H8A*	0.6042	0.6600	0.4778	5.48
H8B*	0.4572	0.4772	0.4815	5.48
H10A*	0.5750	1.0147	0.4061	5.48
H11A*	0.5151	1.0829	0.3126	5.48
H12A*	0.3484	0.8085	0.2630	5.48
H13A*	0.2416	0.4608	0.3077	5.48
H14A*	0.3021	0.3885	0.4012	5.48

*Refined isotropically.

Table 2. *Bond lengths* (Å) *and angles* (°)

O1—C7	1.2204 (30)	C8—C7	1.5017 (32)
C1—C2	1.374 (4)	C8—C9	1.493 (4)
C1—C6	1.3984 (34)	C9—C10	1.3927 (32)
C3—C2	1.3808 (35)	C9—C14	1.3913 (32)
C3—C4	1.386 (4)	C11—C10	1.376 (4)
C5—C4	1.371 (4)	C11—C12	1.382 (4)
C5—C6	1.3929 (32)	C13—C12	1.384 (4)
C6—C7	1.486 (4)	C13—C14	1.377 (4)
C2—C1—C6	120.37 (22)	O1—C7—C8	120.76 (23)
C1—C2—C3	120.51 (23)	C7—C8—C9	115.97 (18)
C2—C3—C4	119.46 (25)	C8—C9—C10	121.30 (20)
C3—C4—C5	120.46 (22)	C8—C9—C14	120.68 (21)
C4—C5—C6	120.57 (22)	C10—C9—C14	118.02 (22)
C1—C6—C5	118.63 (23)	C9—C10—C11	120.78 (22)
C5—C6—C7	122.39 (21)	C10—C11—C12	120.59 (22)
C1—C6—C7	118.97 (21)	C11—C12—C13	119.26 (25)
C6—C7—C8	118.90 (20)	C12—C13—C14	120.15 (22)
O1—C7—C6	120.34 (23)	C9—C14—C13	121.19 (21)

Systematic absences allowed for the space groups $Pca2_1$ and $Pcam$ but $Pcam$ was ruled out as it would require $Z = 8$ (leading to an unrealistic density) or disorder of the molecules. The successful refinement of the structure confirmed our choice.

The lattice parameters and atomic coordinates of the non-H atoms were determined by direct methods and refined using *SHELXTL-Plus* (Sheldrick, 1987). The H-atom positions were calculated using a fixed C—H bond length of 0.96 Å and a fixed isotropic thermal parameter, taking into account the geometry of the site. The data were then re-refined using *GSAS* (Larson & Von Dreele, 1990). The origin along z was fixed at the centre of mass of the molecule and all atom positions refined relative to that origin.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55519 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1010]

References

Adams, J. M. & Morsi, S. E. (1976). *Acta Cryst.* **B32**, 1345–1347.
Demus, D., Demus, H. & Zashke, H. (1974). *Flüssige Kristallen in Tabellen.* Leipzig: VEB Deutscher Verlag für Grundstoffindustrie.
Haisa, M., Kashino, S. & Morimoto, M. (1980). *Acta Cryst.* **B36**, 2832–2834.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Larson, A. C. (1967). *Acta Cryst.* **23**, 664–665.
Larson, A. C. & Von Dreele, R. B. (1990). *GSAS.* Report LA-UR 86-748. Los Alamos National Laboratory, New Mexico, USA.
Sheldrick, G. M. (1987). *SHELXTL-Plus.* Version 2.1. Nicolet Instrument Corporation, Madison, Wisconsin, USA.
Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

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Structure of *Z*-L-(αMe)Phe-(L-Ala)$_2$-OMe Monohydrate

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Abstract

This folded N- and C-protected tripeptide is stabilized by a (peptide) N—H···O=C (urethane) intramolecular hydrogen bond which closes a ten-membered ring. This results in a β-turn structure that can be classified as type I on the basis of the φ, ψ conformational angles for the L-(αMe)Phe^1 and L-Ala^2 residues. The χ^1 side-chain conformational angle for the L-(αMe)Phe residue is *gauche*$^+$.

Comment

As a part of our ongoing conformational analysis of C^α-methylphenylalanine [(αMe)Phe] containing peptides, the X-ray diffraction structural determination of the title compound (I) (N^α-benzyloxycarbonyl-C^α-methyl-L-phenylalanyl-L-alanyl-L-alanine methyl ester) was undertaken to establish the influence of an optically active protein amino acid (L-Ala) on the screw sense of the helical conformation adopted by a