C16	0.4706 (2)	0.6325 (6)	0.3230 (3)	5.2 (3)
C17	0.4246 (2)	0.7511 (5)	0.2551 (3)	5.4 (5)
C18	0.3678 (2)	0.7284 (5)	0.1452 (3)	4.6 (3)
C19	0.3565 (2)	0.5841 (5)	0.1037 (2)	3.7 (3)
C20	0.4027 (2)	0.4565 (5)	0.1736 (2)	3.6 (3)
C21	0.1607 (2)	0.2598 (5)	0.3420 (3)	4.1 (2)
N22	0.2080 (2)	0.3913 (4)	0.3702 (2)	4.5 (2)
C23	0.1619 (3)	0.5328 (5)	0.3243 (4)	5.7 (5)
C24	0.0981 (3)	0.5191 (5)	0.2050 (4)	6.2 (5)
C25	0.0512 (2)	0.3746 (5)	0.1732 (3)	5.5 (4)
N26	0.0998 (2)	0.2346 (4)	0.2271 (2)	3.9 (2)
C27	0.0502 (2)	0.1036 (5)	0.1991 (3)	4.6 (3)
C28	0.0961 (2)	−0.0417 (5)	0.2602 (3)	4.7 (3)
C29	0.1583 (2)	−0.0050 (5)	0.3780 (3)	5.2 (4)
N30	0.2072 (2)	0.1226 (4)	0.3992 (2)	4.3 (3)
C31	0.2624 (2)	0.0902 (5)	0.4006 (2)	4.0 (2)
C32	0.2943 (2)	−0.0545 (5)	0.4226 (3)	5.4 (3)
C33	0.3512 (2)	−0.0775 (6)	0.4260 (3)	6.5 (5)
C34	0.3754 (2)	0.0373 (6)	0.4058 (3)	6.1 (5)
C35	0.3447 (2)	0.1887 (5)	0.3834 (3)	4.9 (3)
C36	0.3686 (2)	0.3145 (7)	0.3625 (3)	5.9 (4)
C37	0.3395 (2)	0.4580 (6)	0.3444 (3)	6.0 (5)
C38	0.2848 (2)	0.4879 (5)	0.3444 (3)	5.2 (4)
C39	0.2594 (2)	0.3680 (5)	0.3632 (2)	4.1 (2)
C40	0.2881 (2)	0.2165 (5)	0.3814 (2)	4.0 (2)

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

N2—C1	1.450 (5)	N22—C21	1.438 (4)
N2—C3	1.473 (6)	N22—C23	1.453 (5)
N2—C19	1.395 (4)	N22—C39	1.405 (4)
N6—C1	1.471 (4)	N26—C21	1.465 (4)
N6—C5	1.475 (6)	N26—C25	1.471 (4)
N6—C7	1.471 (4)	N26—C27	1.468 (6)
N10—C1	1.434 (4)	N30—C21	1.444 (4)
N10—C9	1.459 (5)	N30—C29	1.474 (4)
N10—C11	1.410 (4)	N30—C31	1.402 (4)
C1—N2—C3	109.9 (2)	C21—N22—C23	110.0 (3)
C1—N2—C19	116.9 (2)	C21—N22—C39	115.6 (3)
C3—N2—C19	119.0 (3)	C23—N22—C39	118.1 (3)
C1—N6—C5	110.4 (2)	C21—N26—C25	109.7 (3)
C1—N6—C7	110.1 (2)	C21—N26—C27	110.5 (2)
C5—N6—C7	107.9 (2)	C25—N26—C27	108.3 (2)
C1—N10—C9	110.3 (2)	C21—N30—C29	109.7 (2)
C1—N10—C11	115.9 (3)	C21—N30—C31	116.3 (3)
C9—N10—C11	117.9 (3)	C29—N30—C31	119.0 (3)

The structure was determined by direct methods using *MULTAN*80 (Main *et al.*, 1980) and contains two independent molecules. H atoms were found by difference Fourier then recalculated in steric positions. The H atoms were placed in calculated positions (C—H = 0.98 Å) at the beginning of each least-squares cycle, with a B value of 4.8 Å^2, and not refined. Dispersion corrections were taken from Cromer (1974).

Data collection: CAD-4 diffractometer software. Cell refinement: CAD-4 diffractometer software. Data reduction: local program. Structure solution: *MULTAN*80. Structure refinement: local program. Molecular graphics: *ORTEP*II (Johnson, 1976). Preparation of material for publication: local program.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1136). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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6-(3,5-Di-*O*-acetyl-β-D-2-deoxyribofuranosyl)-3,4-dihydro-8*H*-pyrimido[4,5-*c*][1,2]-oxazin-7(6*H*)-one

Luc Van Meervelt

Laboratorium voor Macromoleculaire Structuurchemie, KU Leuven, Celestijnenlaan 200F, B-3001 Leuven, Belgium

Paul Kong Thoo Lin

School of Applied Sciences, The Robert Gordon University, St Andrew Street, Aberdeen AB1 1HG, Scotland

Daniel M. Brown

Laboratory of Molecular Biology, Medical Research Council, Hills Road, Cambridge CB2 2QH, England

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Abstract

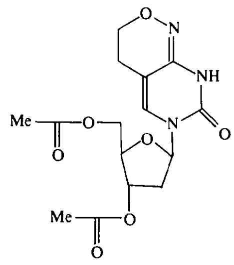
The title compound, $C_{15}H_{19}N_3O_7$ (P), crystallizes with two molecules in the asymmetric unit which differ in sugar and oxazino ring puckering; both occur as the imino tautomer and form P:P wobble base pairs.

Comment

The preferred *syn* conformation of the methoxyl group, with respect to N3, in the base analogue N^4-methoxycytosine M (Van Meervelt, 1991) destabilizes Watson–Crick M:A and M:G base pairs in oligonucleotide duplexes. In the left-handed double helical fragment d(CGCGMG)$_2$, wobble base pairing is found for the M:G base pairs with M in the imino form (Van

Meervelt, Moore, Kong Thoo Lin, Brown & Kennard, 1990).

The related title compound, P, in which the N—O bond is *anti*, is also ambivalent in its hydrogen-bonding potential. In an NMR study the G:P pair showed slow chemical exchange between Watson–Crick (P in amino form) and wobble (P in imino form) conformations (Nedderman, Stone, Williams, Kong Thoo Lin & Brown, 1993). This crystal structure analysis of the 3′,5′-diacetyl derivative of the P deoxynucleoside was undertaken to determine in which tautomer (imino–amino) the nucleoside analogue occurs in the solid state.



P

The compound crystallizes from ethanol in the monoclinic space group $P2_1$ and the asymmetric unit contains two independent molecules (*A* and *B*). The observed disorder in the oxazino moiety of molecule *B* was refined by assuming two alternative positions for atoms O4 and C7 (O4*B*1 and O4*B*2, and C7*B*1 and C7*B*2, in Table 1). The molecular structure of molecule *B* with labelling is depicted in Fig. 1.

The modified base points away from the sugar (*anti* orientation) as reflected by the glycosidic torsion angle χ [−113.3 (3) for *A*, −120.9 (4)° for *B*] which is in the usual range for pyrimidine nucleosides (Saenger, 1984). The pseudorotation phase angle of residue *A* [165 (1)°] is typical of a C2′-*endo* conformation; for residue *B* a symmetrical C2′-*endo*–C1′-*exo* conformation [143 (1)°] is found.

Within the two independent molecules, the C═N distances of 1.279 (5) and 1.288 (5) Å are close to the mean C═N bond length of 1.281 (13) Å in $(C,H)_2$–C═N–OH oximes (Allen *et al.*, 1987) and the C═N distance of 1.315 (8) Å in M (Van Meervelt, 1991). As a consequence the P base analogue is in the imino tautomeric form.

According to the Cremer & Pople (1975) treatment, the oxazino rings can be described by a puckering amplitude Q, a phase angle φ and a polar position θ. For residue *A* these values [0.455 (4) Å, 179.2 (1)°, 50.2 (1)°] are close to those of a half-boat conformation. For residue *B* a twist-boat conformation [0.165 (6) Å, 287.2 (1)°, 119.4 (1)° for conformation (1)] and half-boat conformation [0.424 (6) Å, 25.2 (1)°, 120.1 (1)° for conformation (2)] are found. For both half-boat conformations, C7 is at the top of the envelope.

The orientation about the C4′—C5′ bond, described by γ, is +synclinal (Klyne & Prelog, 1960) [γ = 58.8 (4) for *A* and 50.1 (5)° for *B*].

The two molecules in the asymmetric unit form a P:P wobble base pair (Fig. 2) which differs from the standard Watson–Crick base pair by the loss of the twofold symmetry between the glycosyl C1′—N bonds. The corresponding bond lengths and angles are N3*A*—H3*A* 0.90, H3*A*···O2*B* 2.040 (5), N3*A*···O2*B* 2.872 (5) Å, N3*A*—H3*A*···O2*B* 153.1 (1)°; N4*A*···H3*B* 2.042 (5), H3*B*—N3*B* 0.90, N4*A*···N3*B* 2.925 (5) Å, N4*A*···H3*B*—N3*B* 166.7 (1)°. The dihedral angle between the individual pyrimidine base planes (omitting O4, C7 and C8), known as propeller twist, is +15.9 (2)°. The usual propeller twists in A- and B-DNA are negative (see Dickerson *et al.*, 1989, for conventions).

The observed disorder in one of the oxazino rings can be related to the short intermolecular contacts C7*B*1···O4*B*1 [3.09 (1) Å] and C7*B*2···O4*B*2 [2.90 (1) Å] found for molecule *B*.

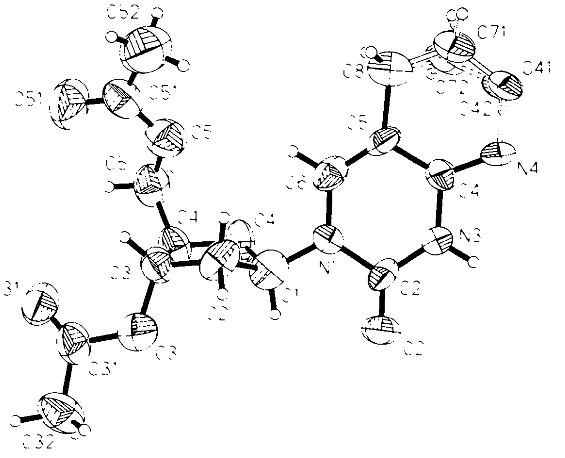


Fig. 1. Structure of molecule *B* with labelling (omitting the '*B*' identifiers of Table 1) showing 50% probability displacement ellipsoids (*SHELXTL-PC*; Sheldrick, 1990).

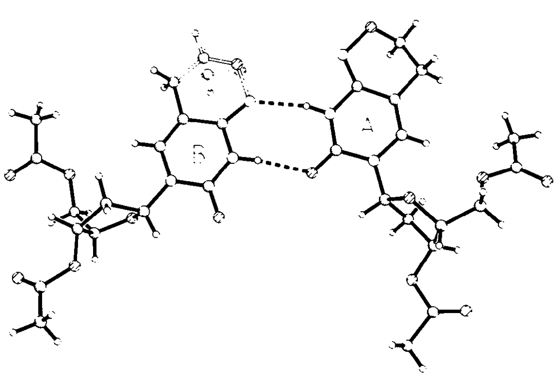


Fig. 2. P:P wobble base-pair formation between two independent molecules in the asymmetric unit (*SHELXTL-PC*; Sheldrick, 1990).

Experimental

Crystal data

$C_{15}H_{19}N_3O_7$
$M_r = 353.33$
Monoclinic
$P2_1$
$a = 14.662\,(3)$ Å
$b = 5.4190\,(10)$ Å
$c = 21.475\,(4)$ Å
$\beta = 101.66\,(3)°$
$V = 1671.1\,(6)$ Å^3
$Z = 4$
$D_x = 1.404$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 20 reflections
$\theta = 11$–$21°$
$\mu = 0.959$ mm^{-1}
$T = 289\,(2)$ K
Needle
$0.3 \times 0.05 \times 0.05$ mm
White

Data collection

Siemens *P*4PC diffractometer
θ–2θ scans
Absorption correction: none
2121 measured reflections
2121 independent reflections
1256 observed reflections $[I > 2\sigma(I)]$

$\theta_{max} = 47.22°$
$h = -13 \rightarrow 13$
$k = -1 \rightarrow 5$
$l = 0 \rightarrow 20$
3 standard reflections monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0692$
$wR(F^2) = 0.1468$
$S = 1.313$
2121 reflections
470 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2 + 5.4135P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.198$
$\Delta\rho_{max} = 0.242$ e Å^{-3}
$\Delta\rho_{min} = -0.271$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.00018 (8)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: assigned to agree with the known chirality at C4′ arising from its synthetic precursor

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}
N1A	0.3420 (2)	0.0501	0.20934 (12)	0.0478 (12)
C2A	0.3935 (3)	0.2388 (8)	0.24522 (15)	0.055 (2)
N3A	0.3764 (2)	0.2614 (7)	0.30448 (11)	0.0476 (12)
C4A	0.3124 (3)	0.1260 (8)	0.33145 (14)	0.050 (2)
C5A	0.2667 (2)	−0.0732 (8)	0.29375 (14)	0.0428 (15)
C6A	0.2814 (2)	−0.0977 (9)	0.2350 (2)	0.047 (2)
O2A	0.4477 (2)	0.3609 (6)	0.22321 (10)	0.0533 (11)
N4A	0.3090 (2)	0.2008 (8)	0.38743 (14)	0.078 (2)
O4A	0.2416 (2)	0.0558 (7)	0.41490 (12)	0.0845 (13)
C7A	0.2337 (3)	−0.1846 (9)	0.3941 (2)	0.071 (2)
C8A	0.1998 (3)	−0.2149 (10)	0.32499 (15)	0.062 (2)
C1′A	0.3481 (2)	0.0204 (8)	0.1421 (2)	0.045 (2)
C2′A	0.3683 (3)	−0.2368 (8)	0.12240 (14)	0.050 (2)
C3′A	0.3179 (2)	−0.2555 (8)	0.05396 (13)	0.046 (2)
C4′A	0.2360 (2)	−0.0870 (9)	0.0509 (2)	0.054 (2)
O4′A	0.2572 (2)	0.0808 (6)	0.10421 (11)	0.0517 (10)
O3′A	0.3808 (2)	−0.1542 (7)	0.01520 (10)	0.0600 (11)
C31A	0.3604 (3)	−0.2132 (11)	−0.0474 (2)	0.073 (2)
O31A	0.2902 (2)	−0.3066 (8)	−0.07063 (14)	0.118 (2)
C32A	0.4304 (3)	−0.1031 (11)	−0.0809 (2)	0.096 (2)
C5′A	0.1448 (2)	−0.2115 (9)	0.0507 (2)	0.054 (2)
O5′A	0.1526 (2)	−0.3579 (6)	0.10736 (11)	0.0570 (11)
C51A	0.0985 (3)	−0.5528 (8)	0.1089 (2)	0.075 (2)
O51A	0.0441 (2)	−0.6203 (8)	0.06143 (15)	0.115 (2)
C52A	0.1073 (3)	−0.6646 (10)	0.1706 (2)	0.075 (2)
N1B	0.3325 (2)	0.5674 (8)	0.71771 (13)	0.0561 (13)
C2B	0.4031 (3)	0.4251 (10)	0.7541 (2)	0.062 (2)
N3B	0.4544 (2)	0.2902 (8)	0.72021 (12)	0.0590 (14)
C4B	0.4461 (2)	0.3013 (10)	0.6540 (2)	0.058 (2)
C5B	0.3779 (3)	0.4689 (9)	0.6203 (2)	0.060 (2)
C6B	0.3247 (3)	0.5899 (9)	0.6519 (2)	0.057 (2)
O2B	0.4096 (2)	0.4041 (7)	0.81080 (11)	0.0613 (12)
N4B	0.5045 (2)	0.1606 (7)	0.63359 (12)	0.0582 (14)
O4B1†	0.5112 (3)	0.2192 (13)	0.5681 (2)	0.063 (2)
C7B1†	0.4432 (4)	0.3381 (18)	0.5282 (3)	0.080 (3)
O4B2†	0.4870 (4)	0.1351 (14)	0.5644 (2)	0.077 (2)
C7B2†	0.4052 (5)	0.2348 (14)	0.5360 (3)	0.064 (3)
C8B	0.3749 (3)	0.4842 (10)	0.5498 (2)	0.084 (2)
C1′B	0.2676 (3)	0.6941 (8)	0.7482 (2)	0.067 (2)
C2′B	0.2578 (2)	0.9667 (8)	0.7381 (2)	0.061 (2)
C3′B	0.1582 (2)	1.0235 (8)	0.7424 (2)	0.062 (2)
C4′B	0.1056 (2)	0.7907 (8)	0.7220 (2)	0.062 (2)
O4′B	0.1751 (2)	0.5997 (6)	0.71856 (14)	0.0660 (12)
O3′B	0.1588 (2)	1.0863 (7)	0.80889 (12)	0.0795 (13)
C31B	0.0895 (3)	1.2380 (10)	0.8201 (2)	0.071 (2)
O31B	0.0256 (2)	1.2851 (7)	0.78081 (13)	0.0791 (13)
C32B	0.1104 (3)	1.3075 (11)	0.8878 (2)	0.089 (2)
C5′B	0.0434 (3)	0.8047 (10)	0.6568 (2)	0.077 (2)
O5′B	0.0937 (2)	0.8976 (7)	0.61145 (12)	0.0856 (14)
C51B	0.0639 (3)	1.0868 (10)	0.5740 (2)	0.077 (2)
O51B	−0.0101 (2)	1.1834 (8)	0.5770 (2)	0.115 (2)
C52B	0.1239 (3)	1.1564 (11)	0.5315 (2)	0.116 (3)

† Occupancy = 0.5.

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

N1A—C6A	1.391 (5)	N1B—C1′B	1.433 (5)
N1A—C2A	1.405 (4)	C2B—O2B	1.208 (4)
N1A—C1′A	1.474 (4)	C2B—N3B	1.360 (5)
C2A—O2A	1.201 (5)	N3B—C4B	1.404 (4)
C2A—N3A	1.351 (4)	C4B—N4B	1.288 (5)
N3A—C4A	1.405 (5)	C4B—C5B	1.434 (6)
C4A—N4A	1.279 (5)	C5B—C6B	1.308 (6)
C4A—C5A	1.432 (5)	C5B—C8B	1.508 (5)
C5A—C6A	1.329 (5)	N4B—O4B1	1.465 (5)
C5A—C8A	1.505 (6)	N4B—O4B2	1.462 (5)
N4A—O4A	1.475 (5)	O4B1—C7B1	1.341 (8)
O4A—C7A	1.375 (6)	C7B1—C8B	1.425 (9)
C7A—C8A	1.476 (4)	O4B2—C7B2	1.344 (8)
C1′A—O4′A	1.452 (4)	C7B2—C8B	1.471 (9)
C1′A—C2′A	1.503 (6)	C1′B—O4′B	1.469 (4)
C2′A—C3′A	1.509 (4)	C1′B—C2′B	1.496 (6)
C3′A—O3′A	1.467 (4)	C2′B—C3′B	1.513 (5)
C3′A—C4′A	1.500 (5)	C3′B—O3′B	1.467 (4)
C4′A—O4′A	1.447 (5)	C3′B—C4′B	1.498 (6)
C4′A—C5′A	1.496 (5)	C4′B—O4′B	1.465 (5)
O3′A—C31A	1.356 (4)	C4′B—C5′B	1.510 (5)
C31A—O31A	1.165 (5)	O3′B—C31B	1.366 (5)
C31A—C32A	1.491 (6)	C31B—O31B	1.155 (4)
C5′A—O5′A	1.438 (4)	C31B—C32B	1.474 (5)
O5′A—C51A	1.325 (5)	C5′B—O5′B	1.427 (5)
C51A—O51A	1.217 (5)	O5′B—C51B	1.322 (6)
C51A—C52A	1.439 (6)	C51B—O51B	1.219 (6)
N1B—C6B	1.400 (4)	C51B—C52B	1.438 (6)
N1B—C2B	1.396 (5)		
C6A—N1A—C2A	121.2 (3)	C2B—N1B—C1′B	119.6 (3)
C6A—N1A—C1′A	119.7 (2)	O2B—C2B—N3B	123.3 (4)
C2A—N1A—C1′A	119.0 (3)	O2B—C2B—N1B	121.0 (4)
O2A—C2A—N3A	125.7 (4)	N3B—C2B—N1B	115.1 (3)
O2A—C2A—N1A	120.6 (3)	C2B—N3B—C4B	125.1 (3)
N3A—C2A—N1A	113.7 (3)	N4B—C4B—N3B	113.1 (3)
C2A—N3A—C4A	127.4 (3)	N4B—C4B—C5B	130.4 (3)
N4A—C4A—N3A	112.3 (4)	N3B—C4B—C5B	116.5 (4)
N4A—C4A—C5A	131.8 (4)	C6B—C5B—C4B	119.0 (3)
N3A—C4A—C5A	115.9 (3)	C6B—C5B—C8B	126.4 (4)

C6*A*—C5*A*—C4*A*	117.7 (4)	C4*B*—C5*B*—C8*B*	114.6 (4)
C6*A*—C5*A*—C8*A*	128.1 (4)	C5*B*—C6*B*—N1*B*	123.0 (4)
C4*A*—C5*A*—C8*A*	113.9 (3)	C4*B*—N4*B*—O4*B*1	112.1 (4)
C5*A*—C6*A*—N1*A*	123.9 (4)	C4*B*—N4*B*—O4*B*2	114.2 (4)
C4*A*—N4*A*—O4*A*	111.3 (3)	C7*B*1—O4*B*1—N4*B*	122.1 (5)
C7*A*—O4*A*—N4*A*	113.2 (3)	O4*B*1—C7*B*1—C8*B*	122.7 (6)
O4*A*—C7*A*—C8*A*	114.9 (4)	C7*B*2—O4*B*2—N4*B*	112.3 (5)
C7*A*—C8*A*—C5*A*	105.9 (3)	O4*B*2—C7*B*2—C8*B*	123.8 (6)
C6*B*—N1*B*—C2*B*	120.8 (3)	C7*B*1—C8*B*—C5*B*	114.6 (4)
C6*B*—N1*B*—C1′*B*	119.5 (3)	C7*B*2—C8*B*—C5*B*	101.7 (5)

The low number of observed reflections, as a result of poor crystal quality, made it necessary to apply similarity restraints for the 1,2 and 1,3 distances in the two molecules, and similar U_{ij} restraints for atoms closer than 1.7 Å. During refinement, the behaviour of the oxazino ring in residue *B* indicated local disorder. Two alternative conformations (with site-occupancy factors 0.5) for atoms O4 and C7 (O4*B*1 and O4*B*2, and C7*B*1 and C7*B*2) were used to describe this disorder.

The H atoms were placed at calculated positions (C—H = 0.98 Å for tertiary C—H, C—H = 0.97 Å for secondary CH_2, C—H = 0.93 Å for C_{sp^2}—H, C—H = 0.96 Å for an idealized methyl group and N—H = 0.90 Å, $U = 1.2U_{eq}$ of the parent atom) and refined in the riding mode or rotating mode (methyl groups).

Absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Table 4.2.4.2).

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: Siemens *REDUCE*. Program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1102). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(±)-*threo*-3-Hydroxy-2,3-diphenylpropanoic Acid Methyl Ester

TSONKO KOLEV, HANS PREUT AND PAUL BLECKMANN

Fachbereich Chemie, Universität Dortmund,
D-44227 Dortmund, Germany

IVAN JUCHNOVSKI

Institute for Organic Chemistry, Bulgarian Academy
of Sciences, Sofia 1113, Bulgaria

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Abstract

The crystal structure of methyl (±)-*threo*-3-hydroxy-2,3-diphenylpropanoate, $C_{16}H_{16}O_3$, has been determined. In the crystal, the phenyl group at the chiral α-C atom and the hydroxy group at the chiral β-C atom are in the energetically preferred *trans* conformation. The torsion angle O—C_β—C_α—C_{phenyl} is 175.8 (2)°. The H atoms at the chiral C atoms are also in an antiperiplanar arrangement. Pairs of molecules are linked *via* a pair of weak intermolecular hydrogen bonds between hydroxyl H and carbonyl O atoms. The IR spectrum verifies the presence of this hydrogen bond. The dihedral angle between the least-squares planes through the phenyl rings is 53.0 (1)°. The distance between the α and β chiral C atoms is 1.530 (3) Å. The bond distances and angles of the molecule are as expected.

Comment

We are interested in the effects which different substituents have on the stereochemistry of substituted diphenylethanes possessing two chiral C atoms. This work is part of a project involving the structural investigation of substituted chiral and diastereoisomeric diphenylethanes. This structural information is significant because the diphenylethane skeleton is present in some natural products and affects fundamental stereochemical reactions (Hayashi, Matsumoto, Kiyoi, Ito, Kohira, Tominaga & Hosomi, 1988; Gong & Streitwieser,