C1	0.2729 (3)	0.4366 (6)	0.5650 (2)	0.0506 (8)
C2	0.3708 (3)	0.6132 (7)	0.5642 (2)	0.0577 (9)
C3	0.4664 (3)	0.6578 (7)	0.6318 (2)	0.0574 (9)
C4	0.4651 (3)	0.5107 (7)	0.7043 (2)	0.0574 (9)
C5	0.3671 (4)	0.3358 (7)	0.7083 (2)	0.0644 (10)
C6	0.2726 (4)	0.2998 (7)	0.6398 (2)	0.0611 (10)
C7	0.1738 (3)	0.3958 (7)	0.4898 (2)	0.0539 (9)
C8	0.1638 (4)	0.5693 (8)	0.4155 (2)	0.0709 (12)
C9	0.5682 (4)	0.8561 (9)	0.6283 (3)	0.0799 (13)

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

O1—N1	1.431 (3)	C2—C3	1.382 (5)
N1—C7	1.279 (5)	C3—C4	1.397 (5)
O2—C4	1.362 (4)	C3—C9	1.501 (5)
C1—C2	1.386 (5)	C4—C5	1.386 (5)
C1—C6	1.396 (5)	C5—C6	1.379 (5)
C1—C7	1.486 (4)	C7—C8	1.497 (5)
C7—N1—O1	113.8 (3)	O2—C4—C5	122.5 (3)
C2—C1—C6	116.6 (3)	O2—C4—C3	117.3 (3)
C2—C1—C7	121.4 (3)	C5—C4—C3	120.2 (3)
C6—C1—C7	122.0 (3)	C6—C5—C4	120.4 (3)
C3—C2—C1	124.0 (3)	C5—C6—C1	121.3 (3)
C2—C3—C4	117.5 (3)	N1—C7—C1	116.1 (3)
C2—C3—C9	121.8 (3)	N1—C7—C8	123.0 (3)
C4—C3—C9	120.7 (3)	C1—C7—C8	120.8 (3)

Table 5. *Comparison of hydrogen-bonding geometries* (Å, °) *of the two title structures*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
Compound (I)			
O1—HO1···O2^{i}	1.82 (3)	2.734 (2)	174 (2)
O2—HO2···N1^{ii}	1.74 (3)	2.742 (2)	176 (2)
Compound (II)			
O1—HO1···N1^{iii}	1.75 (4)	2.774 (4)	165 (3)
O2—HO2···O1^{iv}	1.99 (5)	2.825 (4)	165 (4)

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

All the H atoms in structure (I), including those of the disordered methyl group, were located from the $\Delta\rho$ map. In structure (II), oxime and hydroxyl H atoms were found in a $\Delta\rho$ map, while all other H atoms were included in their calculated positions. In the final cycle of refinement of (I), refinement was carried out on the positions of all atoms, with anisotropic displacement parameters for non-H atoms and isotropic displacement coefficients for ordered H atoms. For the disordered H atoms the relative site occupancy [0.60 (2) for atoms *a*, *b* and *c* attached to C8] and a common isotropic displacement parameter were refined. In structure (II), the non-H atoms were refined with anisotropic displacement parameters. Oxime and hydroxyl H-atom positions, the individual isotropic displacement parameters for the benzene-type H atoms, common isotropic displacement parameters and relative site occupancies for disordered methyl H atoms [0.54 (4) for atoms *a*, *b* and *c* attached to C8 and C9] were refined.

There was some trouble with the data collection for (II), caused by the quality of crystals obtained. This, in turn, resulted in a paucity of data and the high values of $R_{\rm int}$ and R but there were no problems in solving and refining the structure.

*SHELXS*86 (Sheldrick, 1990) was used for structure solution; refinement was performed using *SHELXL* (Sheldrick, 1994). Molecular graphics were prepared using *ORTEP* (Johnson, 1976). The material for publication was produced using *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1131). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2′-Hydroxypropiophenone Oxime

JAN K. MAURIN

Institute of Atomic Energy, 05-400 Otwock-Świerk, Poland

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Abstract

The title structure, $C_9H_{11}NO_2$, consists of antiparallel polar chains of molecules, bonded together by a series of intra- and intermolecular hydrogen bonds between oxime and hydroxyl groups. None of the usual oxime–oxime hydrogen bonds has been observed.

Comment

As a continuation of studies of oxime molecules with a second functional group, possessing donor–acceptor capabilities (Maurin, Paul & Curtin, 1992, 1994; Maurin, Winnicka-Maurin & Leś, 1994), several hydroxy–oxime structures have been determined (Maurin, 1994; Maurin, Wieteska & Winnicka-Maurin, 1994). 2′-Hydroxypropiophenone oxime, (I), is the first hydroxy–oxime structure in which intra- and intermolecular hydrogen bonds are combined in a chain formation. The conformation of the molecules and the numbering of the atoms are given in Fig. 1. The hydrogen-

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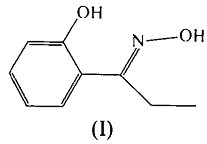
bonding scheme between two independent molecules is also shown.



(I)

The crystal structure is made up of antiparallel molecular chains. The chains are held together by a series of intra- and intermolecular hydrogen bonds, each chain having alternate *A* and *B* molecules. The hydrogen-bonding pattern in each chain is $OH_{hydroxyl}\cdots N_{oxime}$ (intramolecular), $OH_{oxime}\cdots O_{hydroxyl}$ (intermolecular), $OH_{hydroxyl}\cdots N_{oxime}$ (intramolecular), *etc.* The dimensions of the hydrogen-bond geometries are given in Table 3. Both oxime–hydroxyl hydrogen bonds are slightly longer than those observed in 4′-hydroxyacetophenone oxime [2.734 (2) Å; Maurin, 1994]. The intramolecular hydroxyl–oxime hydrogen bonds, however, are considerably shorter than the corresponding intermolecular hydrogen bonds [2.741 (2) Å]. The structure of 2′-hydroxypropiophenone oxime has more in common with oxime–carboxyl structures (Maurin, Paul & Curtin, 1992, 1994), where separated antiparallel polar chains have been observed, than with the previously reported oxime–hydroxyl structures (Maurin, Wieteska & Winnicka-Maurin, 1994; Maurin, 1994). There is a series of intermolecular oxime–hydroxyl hydrogen bonds constructing the infinite polar chains of molecules. The same role in oxime–carboxyl structures is played by oxime–carboxyl and carboxyl–oxime pairs. On the other hand, there is a lack of those hydroxyl–oxime intermolecular hydrogen bonds (they are intramolecular in the present structure) that cause cross-chain interactions in some oxime–hydroxyl structures (Maurin, Winnicka-Maurin & Leś, 1994; Maurin, 1994).

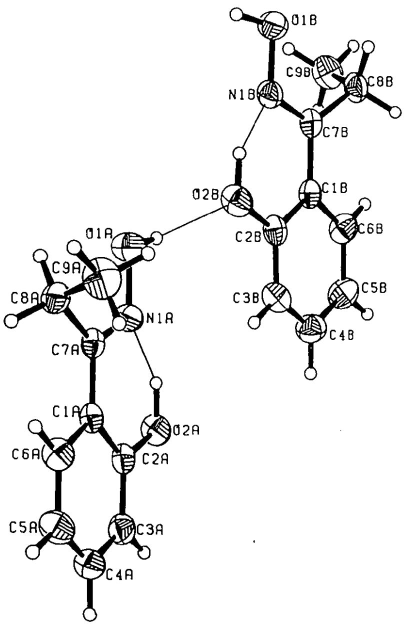


Fig. 1. Conformation of 2′-hydroxypropiophenone oxime molecules and the atomic numbering scheme. Hydrogen bonds are marked as thin lines.

In both molecules of the title compound, the C7 atom lies almost in the plane of the bonded phenyl ring, but the oxime group is twisted slightly around the C1—C7 bond. The dihedral angles between the best plane of the benzene ring and the plane of the oxime group (C1—C7—N1—O1) are 4.69 (18) and 11.55 (20)° for molecules *A* and *B*, respectively. The hydroxyl group of molecule *A* is almost coplanar with the phenyl ring, while that in molecule *B* is slightly out of the plane. In both molecules, the C1—C2 distance is longer than the other phenyl C—C distances, suggesting some repulsion of the *ortho*-substituents. However, the shortening of the C2—C3, C3—C4, C4—C5 and C5—C6 bonds is probably caused by thermal motion. No close intermolecular contacts other than the hydrogen bonds were observed.

Experimental

The title compound was synthesized by the reaction of 2′-hydroxypropiophenone and hydroxylamine hydrochloride in alkaline solution. The recrystallization of the solid product from ethanol–water solution gave colourless prismatic crystals. A fragment cut from a larger crystal was used for data collection.

Crystal data

$C_9H_{11}NO_2$
$M_r = 165.19$
Monoclinic
$P2_1/n$
$a = 9.274\ (2)$ Å
$b = 14.753\ (2)$ Å
$c = 13.290\ (2)$ Å
$\beta = 108.40\ (2)°$
$V = 1725.4\ (5)$ Å^3
$Z = 8$
$D_x = 1.272$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.5–10.9°$
$\mu = 0.090$ mm^{-1}
$T = 253\ (2)$ K
Prismatic, transparent
0.60 × 0.45 × 0.40 mm
Colourless

Data collection

CAD-4 κ-axis four-circle diffractometer
Profile data from ω/θ scans
Absorption correction: none
4398 measured reflections
4152 independent reflections
2222 observed reflections $[I > 2\sigma(I)]$

$R_{int} = 0.013$
$\theta_{max} = 27.97°$
$h = -12 \rightarrow 11$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 17$
3 standard reflections monitored every 200 reflections intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0441$
$wR(F^2) = 0.1095$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.130$ e Å^{-3}
$\Delta\rho_{min} = -0.188$ e Å^{-3}

$S = 0.910$
4152 reflections
305 parameters
All H-atom parameters refined
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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	$U_{\rm eq}$
C1*A*	−0.0716 (2)	0.55378 (9)	0.74258 (10)	0.0395 (3)
C2*A*	−0.1922 (2)	0.51035 (9)	0.66521 (11)	0.0424 (4)
C3*A*	−0.3378 (2)	0.51229 (11)	0.67073 (14)	0.0524 (4)
C4*A*	−0.3687 (2)	0.55541 (12)	0.75322 (15)	0.0579 (5)
C5*A*	−0.2542 (2)	0.59798 (13)	0.83043 (15)	0.0605 (5)
C6*A*	−0.1087 (2)	0.59697 (11)	0.82466 (13)	0.0522 (4)
C7*A*	0.0854 (2)	0.55290 (9)	0.73940 (10)	0.0407 (4)
C8*A*	0.2110 (2)	0.60172 (12)	0.82110 (13)	0.0507 (4)
C9*A*	0.2879 (3)	0.5422 (2)	0.9151 (2)	0.0776 (7)
N1*A*	0.10925 (15)	0.50514 (9)	0.66502 (9)	0.0465 (3)
O1*A*	0.26130 (13)	0.50491 (9)	0.66789 (10)	0.0610 (3)
O2*A*	−0.16895 (14)	0.46549 (8)	0.58157 (8)	0.0542 (3)
C1*B*	0.2082 (2)	0.74457 (9)	1.07936 (11)	0.0423 (4)
C2*B*	0.2675 (2)	0.79876 (10)	1.01492 (11)	0.0443 (4)
C3*B*	0.4197 (2)	0.79548 (12)	1.02358 (14)	0.0547 (4)
C4*B*	0.5176 (2)	0.74053 (13)	1.09804 (15)	0.0606 (5)
C5*B*	0.4639 (2)	0.68842 (13)	1.16442 (15)	0.0629 (5)
C6*B*	0.3118 (2)	0.69006 (12)	1.15399 (13)	0.0551 (4)
C7*B*	0.0454 (2)	0.74464 (9)	1.06989 (11)	0.0431 (4)
C8*B*	−0.0204 (2)	0.67514 (11)	1.12598 (14)	0.0519 (4)
C9*B*	−0.0252 (3)	0.7087 (2)	1.2325 (2)	0.0677 (6)
N1*B*	−0.03563 (15)	0.80753 (8)	1.01210 (10)	0.0473 (3)
O1*B*	−0.18876 (14)	0.80397 (8)	1.00698 (10)	0.0622 (4)
O2*B*	0.1770 (2)	0.85614 (8)	0.94125 (9)	0.0578 (3)

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

C1*A*—C6*A*	1.397 (2)	C1*B*—C6*B*	1.397 (2)
C1*A*—C2*A*	1.412 (2)	C1*B*—C2*B*	1.404 (2)
C1*A*—C7*A*	1.470 (2)	C1*B*—C7*B*	1.475 (2)
C2*A*—O2*A*	1.368 (2)	C2*B*—O2*B*	1.364 (2)
C2*A*—C3*A*	1.376 (2)	C2*B*—C3*B*	1.380 (2)
C3*A*—C4*A*	1.374 (2)	C3*B*—C4*B*	1.376 (3)
C4*A*—C5*A*	1.374 (3)	C4*B*—C5*B*	1.376 (3)
C5*A*—C6*A*	1.376 (3)	C5*B*—C6*B*	1.373 (3)
C7*A*—N1*A*	1.289 (2)	C7*B*—N1*B*	1.285 (2)
C7*A*—C8*A*	1.502 (2)	C7*B*—C8*B*	1.506 (2)
C8*A*—C9*A*	1.509 (3)	C8*B*—C9*B*	1.513 (3)
N1*A*—O1*A*	1.399 (2)	N1*B*—O1*B*	1.401 (2)
O1*A*—H1*A*O	0.86 (2)	O1*B*—H1*B*O	0.91 (2)
O2*A*—H2*A*O	0.96 (2)	O2*B*—H2*B*O	0.98 (2)
C6*A*—C1*A*—C2*A*	116.21 (15)	C6*B*—C1*B*—C2*B*	116.6 (2)
C6*A*—C1*A*—C7*A*	121.14 (14)	C6*B*—C1*B*—C7*B*	121.12 (14)
C2*A*—C1*A*—C7*A*	122.64 (13)	C2*B*—C1*B*—C7*B*	122.32 (13)
O2*A*—C2*A*—C3*A*	117.54 (14)	O2*B*—C2*B*—C3*B*	117.56 (15)
O2*A*—C2*A*—C1*A*	121.32 (14)	O2*B*—C2*B*—C1*B*	121.38 (15)
C3*A*—C2*A*—C1*A*	121.15 (15)	C3*B*—C2*B*—C1*B*	121.06 (15)
C4*A*—C3*A*—C2*A*	120.5 (2)	C4*B*—C3*B*—C2*B*	120.4 (2)
C5*A*—C4*A*—C3*A*	120.1 (2)	C3*B*—C4*B*—C5*B*	120.0 (2)
C4*A*—C5*A*—C6*A*	119.6 (2)	C6*B*—C5*B*—C4*B*	119.6 (2)
C5*A*—C6*A*—C1*A*	122.4 (2)	C5*B*—C6*B*—C1*B*	122.4 (2)
N1*A*—C7*A*—C1*A*	115.98 (13)	N1*B*—C7*B*—C1*B*	116.20 (13)
N1*A*—C7*A*—C8*A*	122.37 (15)	N1*B*—C7*B*—C8*B*	122.4 (2)
C1*A*—C7*A*—C8*A*	121.60 (14)	C1*B*—C7*B*—C8*B*	121.42 (13)
C7*A*—C8*A*—C9*A*	112.0 (2)	C7*B*—C8*B*—C9*B*	112.13 (15)
C7*A*—N1*A*—O1*A*	113.32 (12)	C7*B*—N1*B*—O1*B*	113.25 (12)

Table 3. *Selected intra- and intermolecular hydrogen-bond distances* (Å) *and angles* (°)

D	H	*A*	*D*···*A*	*D*—H···*A*
O2*A*	H2*A*O	N1*A*	2.534 (2)	146 (2)
O2*B*	H2*B*O	N1*B*	2.542 (2)	151 (2)
O1*B*	H1*B*O	O2*A*$^{\rm i}$	2.797 (2)	166 (2)
O1*A*	H1*A*O	O2*B*$^{\rm ii}$	2.788 (2)	154 (2)

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

Data collection, cell refinement and data reduction: Enraf–Nonius software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and a stereoview of the crystal packing have been deposited with the IUCr (Reference: AB1142). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(1*S*,2*S*,5*R*,6*S*)-(+)-6-Carbanilino-1,5-dimethyltricyclo[3.2.0.0^{2,6}]heptane

TING-TING JONG, MEEI-YUEH JEAN AND TA-JUNG LU*

Department of Chemistry, National Chung-Hsing University, Taichung, Taiwan 40227

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Abstract

The asymmetric unit of the title compound, *N*-phenyl-6-(1,5-dimethyltricyclo[3.2.0.0^{2,6}]heptane)carboxamide, $C_{16}H_{19}NO$, contains three independent molecules. There are intermolecular hydrogen bonds of the N—H···O type.

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