organic compounds

(1Z,2E)-2-(Hydroxyimino)-N-p-tolyl-acetamide oxime

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In the molecule of the title compound, $C_9H_{11}N_3O_2$, the glyoxime group has an E configuration. In the crystal structure, the molecules are linked into a ribbon-like structure along the c axis by intermolecular O—H⋯N and O—H⋯O hydrogen bonds.

Related literature

For related literature, see: Büyükgüngör *et al.* (2003); Bertolasi *et al.* (1982); Chertanova *et al.* (1994); Etter *et al.* (1990); Hökelek, Batı *et al.* (2001); Hökelek, Zülfikaroğlu *et al.* (2001); Karle *et al.* (1996); Marsman *et al.* (1999); Özcan & Mirzaoğlu (1988).
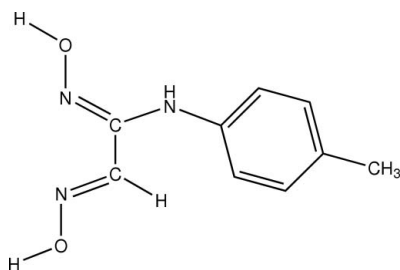
Experimental

Crystal data

$C_9H_{11}N_3O_2$
M_r = 193.21
Monoclinic, Cc
a = 9.0031 (12) Å
b = 19.352 (3) Å
c = 6.9742 (9) Å
β = 126.596 (9)°

V = 975.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.69 × 0.36 × 0.14 mm

Data collection

STOE IPDS II diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.947, T_{max} = 0.987

9760 measured reflections
1072 independent reflections
1024 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.082
S = 1.12
1072 reflections
136 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1A⋯N3[i]	0.90 (3)	1.92 (3)	2.792 (2)	162 (3)
O1—H1A⋯N2[i]	0.90 (3)	2.43 (3)	2.965 (2)	119 (2)
O2—H2A⋯O1[ii]	0.90 (4)	1.88 (4)	2.786 (2)	177 (3)

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2513).

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supplementary materials

(1*Z*,2*E*)-2-(Hydroxyimino)-*N*-*p*-tolylacetamide oxime

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Comment

Intermolecular hydrogen bonding combines moderate strength and directionally (Karle *et al.*, 1996) in linking molecules to form supramolecular structures; this has received considerable attention with respect to directional non-covalent intermolecular interactions (Etter *et al.*, 1990). The oxime (–C=N—OH) moiety is potentially ambidentate, with possibilities of coordination through the N and/or O atoms. It is a functional group that has not been extensively explored in crystal engineering. In the solid state, oximes are usually associated *via* O—H···N hydrogen bonds of length 2.8 Å. Oxime groups posses stronger hydrogen-bonding capabilities than alcohols, phenols and carboxylic acids (Marsman *et al.*, 1999). The hydrogen-bond systems in the crystals of oximes have been analysed and a correlation between a pattern of hydrogen bonding and N—O bond lengths has been suggested (Bertolasi *et al.*, 1982). The configurational and/or conformational isomers of glyoxime derivatives (dioximes) have also been analysed (Chertanova *et al.*, 1994).

The crystal structures of oxime and dioxime derivatives, *viz*. 2,3-dimethylquinoxaline-dimethylglyoxime (1/1) (Hökelek, Batı *et al.*, 2001), 1-(2,6-dimethylphenlamino)propane-1,2-dione dioxime (Hökelek, Zülfikaroğlu *et al.*, 2001) and *N*-hydroxy-2-oxo-2,*N*\'-diphenylacetamidine (Büyükgüngör *et al.*, 2003) have been reported. The structure determination of the title compound (Fig.1) was carried out in order to investigate the strength of the hydrogen bonding capability of the oxime group.

The dihedral angles between the glyoxime planes A (O2/N3/C8), B (O1/N2/C9) and benzene ring C (C1—C6) are A/B= 33.0 (2)°, A/C=42.1 (2)° and B/C=45.1 (2)°. In the glyoxime moiety, the O2—N3 [1.414 (2) Å] bond length is longer than O1—N2 [1.392 (2) Å], while the O1—N2—C9 [111.5 (2)°] bond angle is larger than O2—N3—C8 [110.5 (2)°], reflecting the types and electron-withdrawing or -donating properties of the substituents bonded to C atoms of the glyoxime moiety.

The glyoxime moiety has an E configuration [C9—C8—N3—O2 = −180.0 (2)° and C8—C9—N2—O1 = 178.0 (2)°] (Chertanova *et al.*, 1994). In this configuration, both oxime groups are involved as donors in intermolecular hydrogen bonds (Table 2).

In the crystal structure, the molecules are linked into a ribbon-like structure (Fig. 2) along the *c* axis by intermolecular O—H···N and O—H···O hydrogen bonds (Fig. 2).

Experimental

The title compound was prepared as described by Özcan and Mirzaoğlu (1988), using *p*-toluidine and anti-chloroglyoxime as starting materials in absolute ethanol at 263 K (white precipitate; yield 45%; m.p. 445 K). Colourless crystals of the title compound suitable for X-ray crystallographic analysis were obtained by slow evaporation of a DMF solution at room temperature.

supplementary materials

Refinement

Atoms H1A and H22 were located in a difference map and refined isotropically. The remaining H atoms were positioned geometrically [N—H = 0.86 Å and C—H = 0.93 Å (aromatic) or 0.96 Å (methyl)] and constrained to ride on their parent atom, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl H, and $x = 1.2$ for other H atoms. 942 Friedel pairs were averaged before the final refinement as the absolute structure could not be determined unambiguously.
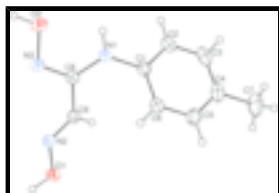
Figures

Fig. 1. The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
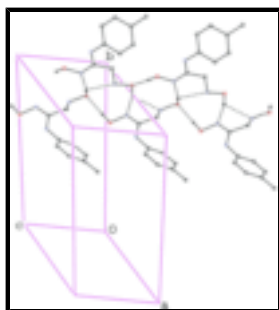

Fig. 2. Part of the crystal packing of the title compound, showing a hydrogen-bonded (dashed lines) ribbon. H atoms not involved in hydrogen bonding have been omitted for clarity.

(1*Z*,2*E*)-2-(Hydroxyimino)-*N*-*p*-tolylacetamide oxime

Crystal data

$C_9H_{11}N_3O_2$	$F_{000} = 408$
$M_r = 193.21$	$D_x = 1.315$ Mg m^{-3}
Monoclinic, Cc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: C -2yc	Cell parameters from 24450 reflections
$a = 9.0031$ (12) Å	$\theta = 3.0$–27.3º
$b = 19.352$ (3) Å	$\mu = 0.10$ mm^{-1}
$c = 6.9742$ (9) Å	$T = 296$ K
$\beta = 126.596$ (9)º	Prism, pale yellow
$V = 975.6$ (3) Å^3	$0.69 \times 0.36 \times 0.14$ mm
$Z = 4$	

Data collection

STOE IPDS II diffractometer	1072 independent reflections
Radiation source: fine-focus sealed tube	1024 reflections with $I > 2\sigma(I)$

Monochromator: graphite	$R_{int} = 0.060$
Detector resolution: 6.67 pixels mm^{-1}	$\theta_{max} = 27.0°$
$T = 296$ K	$\theta_{min} = 3.0°$
ω scan	$h = -11 \rightarrow 11$
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	$k = -24 \rightarrow 24$
$T_{min} = 0.947$, $T_{max} = 0.987$	$l = -8 \rightarrow 8$
9760 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.1463P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.082$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.12$	$\Delta\rho_{max} = 0.13$ e Å^{-3}
1072 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
136 parameters	Extinction correction: none
3 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Experimental. 484 frames, detector distance = 70 mm

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.5005 (3)	0.26817 (10)	0.3450 (4)	0.0396 (4)
C2	0.4256 (3)	0.33277 (12)	0.3234 (4)	0.0471 (5)
H2	0.3711	0.3418	0.3997	0.057*
C3	0.4312 (4)	0.38366 (12)	0.1898 (5)	0.0569 (6)
H3	0.3815	0.4269	0.1790	0.068*
C4	0.5096 (4)	0.37194 (13)	0.0704 (4)	0.0539 (6)

C5	0.5878 (3)	0.30800 (13)	0.0987 (4)	0.0513 (5)
H5	0.6433	0.2991	0.0237	0.062*
C6	0.5859 (3)	0.25656 (11)	0.2356 (4)	0.0439 (5)
H6	0.6420	0.2142	0.2541	0.053*
C7	0.5105 (5)	0.42754 (17)	−0.0808 (6)	0.0820 (10)
H7A	0.4318	0.4649	−0.1019	0.123*
H7B	0.4658	0.4087	−0.2339	0.123*
H7C	0.6343	0.4443	−0.0028	0.123*
C8	0.4705 (3)	0.14747 (10)	0.4385 (3)	0.0373 (4)
C9	0.4235 (3)	0.11959 (10)	0.2112 (3)	0.0375 (4)
H9	0.3545	0.1452	0.0708	0.045*
N1	0.4869 (3)	0.21670 (9)	0.4769 (3)	0.0441 (4)
H1	0.4893	0.2308	0.5957	0.053*
N2	0.4806 (3)	0.05983 (9)	0.2152 (3)	0.0422 (4)
N3	0.4826 (3)	0.10245 (9)	0.5830 (3)	0.0443 (4)
O1	0.4217 (3)	0.03900 (8)	−0.0112 (3)	0.0513 (4)
O2	0.5278 (3)	0.13481 (9)	0.7934 (3)	0.0566 (5)
H1A	0.456 (4)	−0.0057 (18)	0.009 (5)	0.060 (9)*
H2A	0.497 (5)	0.1034 (19)	0.860 (6)	0.073 (10)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0436 (11)	0.0315 (10)	0.0392 (10)	−0.0042 (8)	0.0223 (9)	−0.0034 (8)
C2	0.0519 (12)	0.0341 (10)	0.0535 (12)	−0.0017 (8)	0.0304 (10)	−0.0061 (8)
C3	0.0571 (14)	0.0312 (9)	0.0616 (14)	0.0013 (9)	0.0242 (11)	0.0012 (10)
C4	0.0532 (13)	0.0446 (12)	0.0486 (12)	−0.0101 (10)	0.0221 (11)	0.0064 (10)
C5	0.0555 (13)	0.0494 (13)	0.0501 (12)	−0.0124 (10)	0.0321 (10)	−0.0033 (10)
C6	0.0503 (12)	0.0334 (10)	0.0508 (11)	−0.0016 (9)	0.0317 (10)	−0.0016 (8)
C7	0.092 (2)	0.0601 (17)	0.073 (2)	−0.0113 (16)	0.0385 (18)	0.0197 (15)
C8	0.0459 (11)	0.0324 (10)	0.0405 (10)	−0.0025 (8)	0.0295 (9)	−0.0027 (8)
C9	0.0498 (11)	0.0299 (9)	0.0367 (9)	−0.0009 (8)	0.0279 (8)	0.0018 (7)
N1	0.0645 (11)	0.0340 (9)	0.0464 (9)	−0.0022 (8)	0.0398 (9)	−0.0049 (7)
N2	0.0653 (11)	0.0339 (8)	0.0386 (8)	0.0003 (8)	0.0371 (8)	−0.0001 (7)
N3	0.0639 (11)	0.0395 (9)	0.0427 (9)	−0.0055 (8)	0.0390 (8)	−0.0043 (7)
O1	0.0877 (13)	0.0352 (8)	0.0436 (8)	0.0018 (8)	0.0459 (9)	−0.0020 (6)
O2	0.0891 (14)	0.0523 (10)	0.0487 (9)	−0.0105 (9)	0.0521 (10)	−0.0087 (7)

Geometric parameters (Å, °)

C1—C2	1.386 (3)	C7—H7B	0.96
C1—C6	1.387 (3)	C7—H7C	0.96
C1—N1	1.410 (3)	C8—N3	1.287 (3)
C2—C3	1.377 (4)	C8—N1	1.357 (3)
C2—H2	0.93	C8—C9	1.478 (3)
C3—C4	1.394 (4)	C9—N2	1.259 (3)
C3—H3	0.93	C9—H9	0.93
C4—C5	1.378 (4)	N1—H1	0.86
C4—C7	1.510 (4)	N2—O1	1.392 (2)

C5—C6	1.387 (3)	N3—O2	1.414 (2)
C5—H5	0.93	O1—H1A	0.90 (3)
C6—H6	0.93	O2—H2A	0.90 (4)
C7—H7A	0.96		
C2—C1—C6	118.7 (2)	C4—C7—H7B	109.5
C2—C1—N1	118.84 (19)	H7A—C7—H7B	109.5
C6—C1—N1	122.47 (18)	C4—C7—H7C	109.5
C3—C2—C1	120.4 (2)	H7A—C7—H7C	109.5
C3—C2—H2	119.8	H7B—C7—H7C	109.5
C1—C2—H2	119.8	N3—C8—N1	124.44 (19)
C2—C3—C4	121.6 (2)	N3—C8—C9	115.44 (18)
C2—C3—H3	119.2	N1—C8—C9	120.00 (18)
C4—C3—H3	119.2	N2—C9—C8	117.38 (17)
C5—C4—C3	117.2 (2)	N2—C9—H9	121.3
C5—C4—C7	121.7 (3)	C8—C9—H9	121.3
C3—C4—C7	121.1 (3)	C8—N1—C1	127.63 (18)
C4—C5—C6	121.9 (2)	C8—N1—H1	116.2
C4—C5—H5	119.1	C1—N1—H1	116.2
C6—C5—H5	119.1	C9—N2—O1	111.50 (16)
C5—C6—C1	120.1 (2)	C8—N3—O2	110.52 (17)
C5—C6—H6	120.0	N2—O1—H1A	104 (2)
C1—C6—H6	120.0	N3—O2—H2A	104 (2)
C4—C7—H7A	109.5		
C6—C1—C2—C3	1.9 (3)	N3—C8—C9—N2	−32.1 (3)
N1—C1—C2—C3	−178.0 (2)	N1—C8—C9—N2	151.7 (2)
C1—C2—C3—C4	0.8 (4)	N3—C8—N1—C1	170.6 (2)
C2—C3—C4—C5	−2.4 (4)	C9—C8—N1—C1	−13.6 (4)
C2—C3—C4—C7	178.4 (2)	C2—C1—N1—C8	147.7 (2)
C3—C4—C5—C6	1.3 (4)	C6—C1—N1—C8	−32.2 (3)
C7—C4—C5—C6	−179.4 (2)	C8—C9—N2—O1	177.96 (19)
C4—C5—C6—C1	1.3 (3)	N1—C8—N3—O2	−4.0 (3)
C2—C1—C6—C5	−2.9 (3)	C9—C8—N3—O2	−179.96 (19)
N1—C1—C6—C5	177.0 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···N3[i]	0.90 (3)	1.92 (3)	2.792 (2)	162 (3)
O1—H1A···N2[i]	0.90 (3)	2.43 (3)	2.965 (2)	119 (2)
O2—H2A···O1[ii]	0.90 (4)	1.88 (4)	2.786 (2)	177 (3)

Symmetry codes: (i) *x*, −*y*, *z*−1/2; (ii) *x*, *y*, *z*+1.

Fig. 1

Fig. 2