anti-1-(Benzylamino)glyoxime

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In the title compound, $C_9H_{11}N_3O_2$, all bond lengths and angles are normal. The conformation of the molecule is determined by intra- and intermolecular hydrogen bonds.

Comment

Coordination compounds containing vic-dioxime ligands have been known and studied since the beginning of this century. Numerous dioximes and their transition metal complexes have been investigated (Chakravorthy, 1974). When the C atom of the C=N group is replaced by two different substituents, such as R or R' (R and R' are an alkyl and an aryl), the oximes form geometric isomers named with *syn*, *amphi* or *anti* prefixes (Eloy & Leavers, 1962). The *anti* form is more stable than the *amphi* form (Piloty & Steinback, 1902; Weyl, 1968). The exceptional stability and unique electronic properties of these complexes can be attributed to their planar structures, stabilized by hydrogen bonding (Schrauzer & Kohnle, 1964). The high stability of the complexes prepared with vic-dioxime ligands has been used extensively for various purposes. For example, some of these complexes may possess the properties of semiconductors (Schrauzer & Windgassen, 1967) or show high conductivity (Kobayashi et al., 1993).

In the title compound, (I) (Fig. 1), the glyoxime moiety is nearly planar, the maximum deviation being 0.02 (13) Å for atom N2. The dihedral angle between the benzene ring and the glyoxime moiety is 73.38 (5)°. The crystal packing and molecular conformation in (I) are stabilized by intra- and intermolecular hydrogen bonds (Table 2). The hydroxy groups of the glyoxime moiety form two intermolecular hydrogen bonds, namely O—H···N and O—H···O (Table 2).

(I)
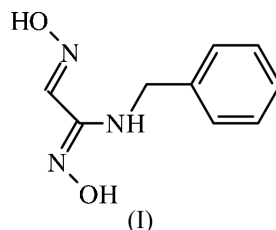
## Experimental

Benzylamine (2.2 ml, 2×10^{-2} mol) was dissolved in ethanol (5 ml). A solution containing *anti*-chloroglyoxime (1.225 g, 1×10^{-2} mol) in ethanol (5 ml) was added slowly at room temperature with constant stirring. The mixture was stirred for 2 h. The pH of the mixture was about 7.0–7.5. Its volume was then doubled by adding distilled water. The resulting white precipitate was filtered off, washed with cold water, dried and crystallized from water–ethanol (1:3) (yield 82.5%,
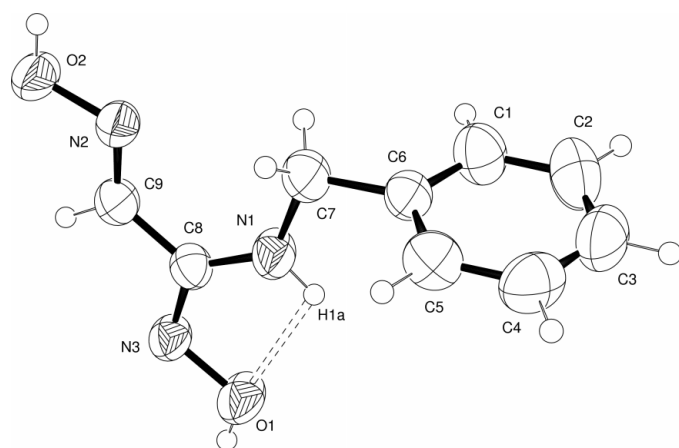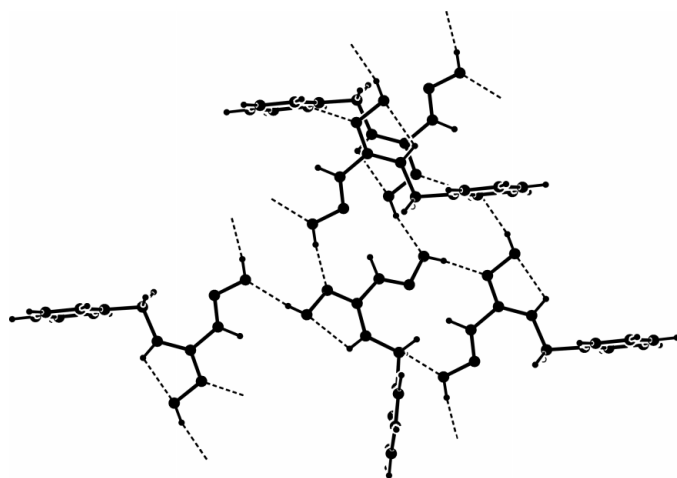
Figure 1
View of (I), with 50% probability displacement ellipsoids and the atom-numbering scheme.

Figure 2
Crystal packing and hydrogen bonding (dashed lines) in (I).

m.p 429 K). The title compound is soluble in ether, DMF, DMSO, dioxane and ethanol, and less soluble in water and chloroform. Analysis calculated for $C_9H_{11}N_3O_2$: C 55.96, H 6.12, N 21.65%; found: C 55.81, H 6.04, N 21.48%. IR data (KBr, cm^{-1}): 3390 (N—H), 3195 (O—H), 1640 (C=N), 960 (N—O), 2830 (C—H, aliphatic), 3035 (C—H, aromatic). ^{1}H NMR (d_6, p.p.m.):11.22 (s, O—H), 10.18 (s, O—H), 5.89 (s, N—H), 7.30–7.17 (m, aromatic H), 4.62 (s, C—H), 8.16 (s, H—C=N–).

Crystal data

$C_9H_{11}N_3O_2$	Mo $K\alpha$ radiation
$M_r = 193.21$	Cell parameters from 7310
Orthorhombic, $Pbca$	reflections
$a = 11.6618\ (9)$ Å	$\theta = 1.8–25.4°$
$b = 8.8584\ (7)$ Å	$\mu = 0.10$ mm^{-1}
$c = 19.163\ (2)$ Å	$T = 293\ (2)$ K
$V = 1979.6\ (3)$ Å^3	Prism, yellow
$Z = 8$	$0.50 \times 0.28 \times 0.11$ mm
$D_x = 1.297$ Mg m^{-3}	

Data collection

Stoe IPDS-II diffractometer	$R_{int} = 0.074$
ω scans	$\theta_{max} = 26.0°$
18 058 measured reflections	$h = -13 \rightarrow 14$
1935 independent reflections	$k = -10 \rightarrow 10$
1037 reflections with $I > 2\sigma(I)$	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.034$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.085$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.80$	$\Delta\rho_{max} = 0.14$ e Å^{-3}
1935 reflections	$\Delta\rho_{min} = -0.11$ e Å^{-3}
128 parameters	Extinction correction: *SHELXL*97
H-atom parameters constrained	Extinction coefficient: 0.0139 (14)

Table 1
Selected geometric parameters (Å, °).

O2—N2	1.3912 (16)	C8—N1	1.3400 (19)
N3—C8	1.2986 (18)	N2—C9	1.2532 (19)
N3—O1	1.4075 (16)	N1—C7	1.456 (2)
C8—N3—O1	110.59 (12)	C8—N1—C7	127.94 (13)
C9—N2—O2	111.75 (13)		
O1—N3—C8—N1	1.5 (2)	O2—N2—C9—C8	179.56 (15)
O1—N3—C8—C9	−179.26 (13)	C8—N1—C7—C6	174.75 (15)
C9—C8—N1—C7	7.2 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$O2^i	0.82	2.05	2.7805 (16)	148
O2—H2$\cdots$N3^{ii}	0.82	1.85	2.6705 (17)	174
N1—H1$A\cdots$O1	0.86	2.16	2.5314 (17)	106

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

H atoms were positioned geometrically and treated using a riding model, with C—H distances of 0.93 (aromatic CH) and 0.97 Å (CH$_2$) and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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