3705 reflections
253 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 2.4171P]$
where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.210 (3)	O3—C18	1.217 (3)
O2—C8	1.219 (3)		
C5—C1—C2—C3	12.6 (3)	C6—C7—C8—C9	170.7 (2)
C1—C2—C3—C4	−30.9 (3)	C7—C8—C9—C10	−169.4 (2)
C2—C3—C4—C5	38.3 (3)	C4—C5—C16—C17	−58.7 (3)
C2—C1—C5—C4	10.5 (3)	C5—C16—C17—C18	−173.3 (2)
C3—C4—C5—C1	−29.6 (3)	C16—C17—C18—O3	0.8 (4)
C3—C2—C6—C7	−67.5 (3)	C16—C17—C18—C19	−179.4 (3)
C2—C6—C7—C8	−171.2 (2)	C17—C18—C19—C20	7.2 (5)
C6—C7—C8—O2	−7.2 (4)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C7—H7$A\cdots$O2$^{\rm i}$	0.97	2.41	3.113 (3)	129
C10—H10$A\cdots\pi$(ring B)$^{\rm ii}$	0.93	3.05	3.863 (3)	146
C15—H15$F\cdots\pi$(ring C)$^{\rm iii}$	0.96	2.72	3.637 (3)	160
C17—H17$B\cdots\pi$(ring B)$^{\rm iv}$	0.97	3.16	3.986 (3)	143
C25—H25$E\cdots\pi$(ring C)$^{\rm v}$	0.96	3.10	3.970 (3)	152

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

All H atoms were located from a difference electron-density map. Both methyl groups were found to be disordered, with two positions rotated from each other by 60°, and they were treated as idealized disordered methyl groups. The remaining H atoms were also placed at geometrically calculated positions and a riding model was used for their refinement. The $U_{\rm iso}$ values for the methyl H atoms were set at $1.5U_{\rm eq}$ of the attached atom and those for the remaining H atoms were set at $1.2U_{\rm eq}$.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1083). Services for accessing these data are described at the back of the journal.

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1-(4-Benzylpiperidin-1-yl)glyoxal dioxime

Sema Öztürk,[a] Şamil Işık,[b] Mustafa Macit[c] and Hoong-Kun Fun[d]

[a]*Department of Physics, Faculty of Arts and Sciences, Erciyes University, Kayseri 38039, Turkey,* [b]*Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, Kurupelit, 55139 Samsun, Turkey,* [c]*Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, Kurupelit, 55139 Samsun, Turkey, and* [d]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: samili@samsun.omu.edu.tr*

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Abstract

The title compound, $C_{14}H_{19}N_3O_2$, contains two independent molecules in the asymmetric unit. The piperidine rings assume chair conformations. The dihedral angle between the piperidine and benzyl ring planes is 86.6 (1)° in molecule *A* and 77.2 (1)° in molecule *B*. Both O—H$\cdots$O and O—H$\cdots$N intermolecular hydrogen bonds are present.

Comment

Oximes and dioximes are of much analytical and medicinal interest, and for this reason their chemistry has been investigated extensively (Hamilton, 1961; Calleri *et al.*, 1966; Chakravorty, 1974). The interesting property they possess of forming complexes with transition metals has also been studied (Bekaroğlu *et al.*, 1978; Endres, 1978). Structural information on oximes and dioximes and their derivatives is therefore useful, and so the structure of the title compound, (I), has been determined.

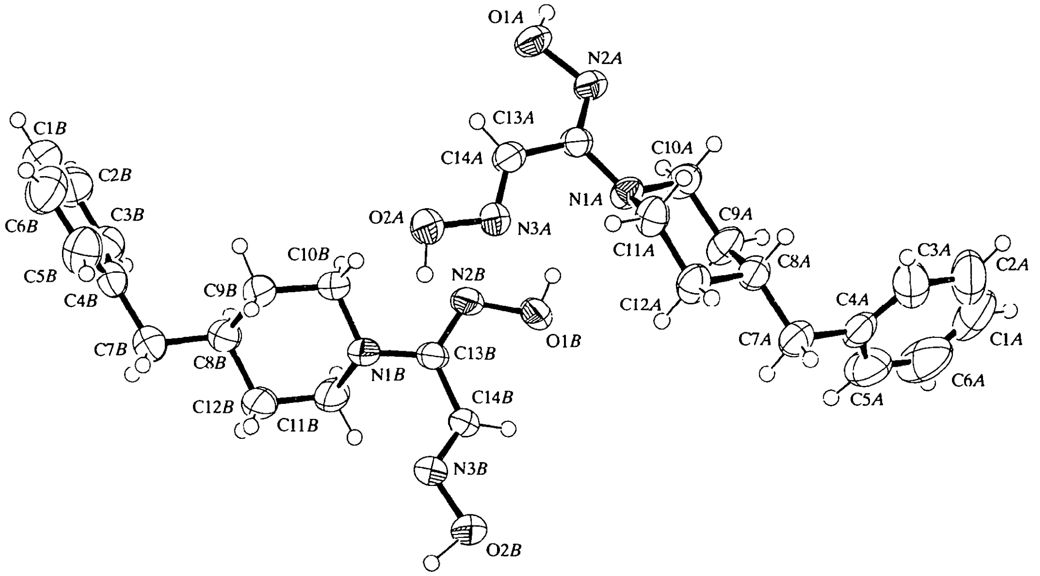
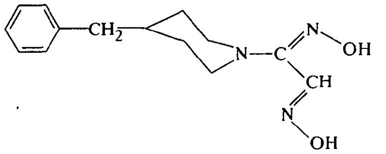


Fig. 1. The structures of the two independent molecules in the asymmetric unit of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.



(I)

The conformations of the two independent molecules of the title compound, as found in the crystal structure, are shown in Fig. 1. The benzyl ring is planar, with the largest deviations from the least-squares plane being those of atoms C6*A* [−0.006 (4) Å] and C5*B* [0.005 (2) Å] in molecules *A* and *B*, respectively. The average N—C distance in the piperazine ring is 1.475 (2) Å, in agreement with the value of 1.470 (2) Å found in 4-hydroxy-2,6-bis-[(4-methylpiperazinyl)methyl]benzonitrile monohydrate (Gunasekaran *et al.*, 1997). The piperidine ring adopts the chair conformation, with the corresponding puckering parameters (Cremer & Pople, 1975) Q = 0.578 (2) Å and θ = 179.5 (2)° in molecule *A*, and Q = 0.560 (2) Å and θ = 177.7 (2)° in molecule *B*. In the glyoxime groups, the average N—O bond distance [1.405 (2) Å] is comparable with those found in X-ray investigations of glyoxime derivatives [1.403 (2) Å; Chertanova *et al.*, 1994]. The glyoxime has the *E*,*E*-configuration.

All bond distances and angles are in the normal ranges (Allen *et al.*, 1987; Olszak *et al.*, 1993, 1995). In the crystal, O—H···O and O—H···N intermolecular hydrogen bonds form an infinite chain along the *c* axis. The details of these hydrogen bonds are given in Table 2. Molecules *A* and *B* are linked to each other as mixed crosslinking chains along the [001] direction. There is only one bifurcated hydrogen bond [O1*A*—H1O*A*···O1*B*^{i} = 2.40 (3) and O1*A*—H1O*A*···N3*A*^{i} = 2.47 (2) Å; symmetry code: (i) $x, \frac{1}{2}-y, z-\frac{1}{2}$]. Three OH groups are donors in *A* [O1*A*—H1O*A*···O1*B*^{i} = 3.089 (2), O1*A*—H1O*A*···N3*A*^{i} = 3.156 (2) and O2*A*—

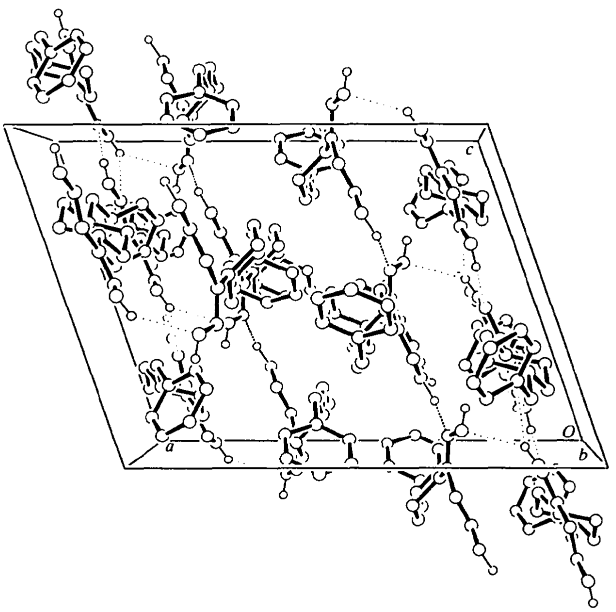


Fig. 2. A perspective drawing of the packing arrangement in (I) along the *b* axis. Dashed lines indicate O—H···O and O—H···N interactions. Only the H atoms that participate in these interactions are shown.

H2OA···N2A^{ii} = 2.826 (2) Å; symmetry code: (ii) $x, \frac{1}{2}-y, z+\frac{1}{2}$], but only one in *B* [O2*B*—H2O*B*···N2*B*^{ii} = 2.738 (2) Å].

Experimental

To a stirred solution of *anti*-chloroglyoxime (20 mmol) in absolute ethanol (40 ml) at 263 K, 1-benzyl-4-piperidineglyoxime (20 mmol) in absolute ethanol (40 ml) was added dropwise over a period of 1 h. The reaction mixture was stirred for a further 4 h at a temperature below 273 K. After adjusting the pH of the mixture to 6–7 with 1% KOH solution at 273 K and stirring for a further 4 h, the mixture was filtered and washed with diethyl ether to give (I). The product was recrystallized from an ethanol–water mixture (2:1) by slow evaporation at room temperature.

Crystal data

$C_{14}H_{19}N_3O_2$
$M_r = 261.32$
Monoclinic
$P2_1/c$
$a = 17.0422(3)$ Å
$b = 13.5414(2)$ Å
$c = 13.1573(2)$ Å
$\beta = 109.060(1)^\circ$
$V = 2869.92(8)$ Å^3
$Z = 8$
$D_x = 1.210$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6266 reflections
$\theta = 2.81$–33.17°
$\mu = 0.083$ mm^{-1}
$T = 293(2)$ K
Block
0.60 × 0.48 × 0.34 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
18 111 measured reflections
6542 independent reflections
5092 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.019$
$\theta_{\rm max} = 27.5^\circ$
$h = -22 \rightarrow 20$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 17$
Intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.122$
$S = 1.028$
6542 reflections
495 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.6450P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.33$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1*A*—N2*A*	1.4160 (15)	O1*B*—N2*B*	1.431 (2)
O2*A*—N3*A*	1.3890 (14)	O2*B*—N3*B*	1.3837 (15)
N1*A*—C13*A*	1.401 (2)	N1*B*—C13*B*	1.383 (2)
N2*A*—C13*A*	1.287 (2)	N2*B*—C13*B*	1.289 (2)
N3*A*—C14*A*	1.267 (2)	N3*B*—C14*B*	1.274 (2)
C4*A*—C7*A*	1.509 (2)	C4*B*—C7*B*	1.510 (2)
C7*A*—C8*A*	1.530 (2)	C7*B*—C8*B*	1.531 (2)
C13*A*—N2*A*—O1*A*	112.22 (11)	C13*B*—N2*B*—O1*B*	111.71 (11)
C14*A*—N3*A*—O2*A*	112.56 (11)	C14*B*—N3*B*—O2*B*	111.01 (11)
C4*A*—C7*A*—C8*A*	114.94 (12)	C4*B*—C7*B*—C8*B*	114.06 (13)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1O*A*···O1*B*^{i}	0.84 (3)	2.40 (3)	3.089 (2)	139 (2)
O1*A*—H1O*A*···N3*A*^{i}	0.84 (3)	2.47 (2)	3.156 (2)	139 (2)
O2*A*—H2O*A*···N2*A*^{ii}	0.92 (3)	1.93 (2)	2.826 (2)	163 (2)
O2*B*—H2O*B*···N2*B*^{ii}	1.12 (3)	1.63 (3)	2.738 (2)	170 (3)

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

All H atoms were located from a difference Fourier map and were refined isotropically, with $U_{\rm iso}$ in the range 0.049 (4)–0.138 (10) Å^2 and C—H distances in the range 0.92 (2)–1.02 (2) Å.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *SHELXTL* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1184). Services for accessing these data are described at the back of the journal.

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