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(2Z,3Z)-Quinoxaline-2,3(1H,4H)-dione dioxime

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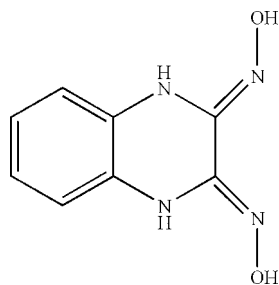
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.060; wR factor = 0.118; data-to-parameter ratio = 14.7.

The asymmetric unit of the title compound, $\text{C}_8\text{H}_8\text{N}_4\text{O}_2$, contains one half-molecule; a twofold rotation axis bisects the molecule. An intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond results in the formation of a five-membered ring, which displays an envelope conformation. In the crystal structure, intermolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules.

Related literature

For related literature, see: Kakanejadifard, Niknam & Zabardasti (2007); Kakanejadifard, Saniei *et al.* (2007); Kakanejadifard & Niknam (2006); For general background, see: Jones *et al.* (1961); Schrauzer & Kohnle (1964); Yari *et al.* (2006); Hashemi *et al.* (2006); Ghiasvand *et al.* (2004, 2005); Kakanejadifard, Niknam, Ranjbar *et al.* (2007); Gok & Kantekin (1997).



Experimental

Crystal data

$\text{C}_8\text{H}_8\text{N}_4\text{O}_2$
 $M_r = 192.18$
 Orthorhombic, $Fddd$
 $a = 9.831$ (2) Å
 $b = 13.609$ (3) Å
 $c = 24.344$ (5) Å

$V = 3256.9$ (11) Å³
 $Z = 16$
 Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 120$ (2) K
 $0.4 \times 0.2 \times 0.2$ mm

Data collection

Bruker SMART 1000 CCD
 area-detector diffractometer
 Absorption correction: none
 7925 measured reflections

983 independent reflections
 708 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.065$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.118$
 $S = 1.01$
 983 reflections
 67 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.43$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}^i$	0.852 (13)	1.971 (10)	2.763 (2)	154
$\text{N2}-\text{H2A}\cdots\text{O1}$	0.86	2.25	2.566 (3)	102

Symmetry code: (i) $-x + 1, -y, -z + 1$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2493).

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supplementary materials

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(2Z,3Z)-Quinoxaline-2,3(1H,4H)-dione dioxime

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Comment

Recently, we have reported the syntheses and chemical behaviours of some vic-dioximes. In our investigations, the reactions of amines with dichloro- glyoxime or cyanogendi-N-oxide resulted in various symmetrically substituted diaminoglyoxime derivatives, in which some of them were quite suitable to act, as donor species, towards some transition metal ions (Kakanejadifard, Niknam & Zabardasti, 2007; Kakanejadifard, Saniei *et al.*, 2007; Kakanejadifard & Niknam, 2006). Some oximes are widely used for various purposes in organic, inorganic, bioinorganic, pigment, analytical, dyes and medical chemistry (Jones *et al.*, 1961; Schrauzer & Kohnle, 1964; Yari *et al.*, 2006; Hashemi *et al.*, 2006; Ghiasvand *et al.*, 2004, 2005; Kakanejadifard, Niknam, Ranjbar *et al.*, 2007). vic-Dioximes, containing mildly acidic hydroxyl groups and slightly basic nitrogen atoms, are amphoteric and their transition metal complexes have been widely investigated as analytical reagents (Gok & Kantekin, 1997). We report herein the synthesis and crystal structure of the title compound.

The asymmetric unit of the title compound contains one-half molecule (Fig. 1). The intramolecular N-H...O hydrogen bond (Table 1) results in the formation of a five-membered ring: (O1/N1/N2/C1/H2A), having envelope conformation, with H2A atom displaced by -0.132 Å from the plane of the other ring atoms.

In the crystal structure, intermolecular O-H...N hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, a solution of Na₂CO₃ (0.2 g, 1.9 mmol) in MeCN (30 ml) was added to a magnetically stirred solution of dichloroglyoxime (1.57 g, 10 mmol) in MeCN (20 ml) and a solution of 1,2-phenyldiamine (1.08 g, 10 mmol) at 278 K. After 2 h stirring at room temperature, the mixture was filtered and the brown precipitate was washed with cold MeCN. It was recrystallized from EtOH (70% aq) in one week (yield; 93.0%, m.p. 512 K).

Refinement

H1 atom (for OH) was located in difference syntheses and refined [O-H = 0.852 (13) Å, U_{iso}(H) = 0.039 Å²]. The remaining H atoms were positioned geometrically, with N-H = 0.86 Å (for NH) and C-H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C,N).

Figures

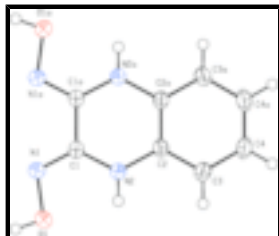


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level [symmetry code: (a) $5/4 - x, 1/4 - y, z$].

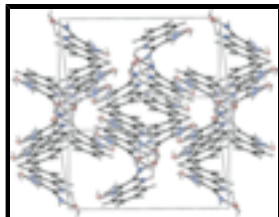


Fig. 2. A packing diagram of the title molecule. Hydrogen bonds are shown as dashed lines.

(2Z,3Z)-Quinoxaline-2,3(1H,4H)-dione dioxime

Crystal data

$C_8H_8N_4O_2$

$M_r = 192.18$

Orthorhombic, $Fddd$

Hall symbol: $-F\ 2uv\ 2vw$

$a = 9.831\ (2)\ \text{\AA}$

$b = 13.609\ (3)\ \text{\AA}$

$c = 24.344\ (5)\ \text{\AA}$

$V = 3256.9\ (11)\ \text{\AA}^3$

$Z = 16$

$F_{000} = 1600$

$D_x = 1.568\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 744 reflections

$\theta = 3\text{--}30^\circ$

$\mu = 0.12\ \text{mm}^{-1}$

$T = 120\ (2)\ \text{K}$

Prism, light-brown

$0.4 \times 0.2 \times 0.2\ \text{mm}$

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 120\ (2)\ \text{K}$

φ and ω scans

Absorption correction: none

7925 measured reflections

983 independent reflections

708 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.065$

$\theta_{\text{max}} = 28.0^\circ$

$\theta_{\text{min}} = 2.7^\circ$

$h = -12 \rightarrow 12$

$k = -17 \rightarrow 17$

$l = -32 \rightarrow 31$

Refinement

Refinement on F^2

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

$$R[F^2 > 2\sigma(F^2)] = 0.060$$

$$wR(F^2) = 0.118$$

$$S = 1.01$$

983 reflections

67 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.005P)^2 + 25P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Special details

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.47468 (17)	-0.04564 (12)	0.43944 (6)	0.0324 (4)
H1	0.454 (3)	-0.0611 (18)	0.4723 (4)	0.039*
N1	0.54273 (19)	0.04548 (13)	0.44726 (7)	0.0261 (4)
N2	0.5691 (2)	0.03307 (14)	0.35156 (7)	0.0308 (5)
H2A	0.5385	-0.0261	0.3513	0.037*
C1	0.5867 (2)	0.07818 (15)	0.40084 (8)	0.0242 (4)
C2	0.5981 (2)	0.07776 (16)	0.30119 (8)	0.0268 (5)
C3	0.5745 (2)	0.03025 (19)	0.25164 (9)	0.0358 (6)
H3A	0.5413	-0.0337	0.2514	0.043*
C4	0.6000 (3)	0.0776 (2)	0.20291 (9)	0.0412 (6)
H4A	0.5835	0.0457	0.1698	0.049*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0423 (10)	0.0304 (8)	0.0244 (7)	-0.0098 (7)	0.0009 (7)	0.0025 (7)
N1	0.0314 (9)	0.0243 (9)	0.0226 (8)	-0.0053 (8)	0.0002 (7)	0.0004 (7)
N2	0.0447 (11)	0.0279 (9)	0.0198 (8)	-0.0126 (8)	0.0022 (8)	-0.0015 (7)
C1	0.0282 (11)	0.0261 (10)	0.0184 (9)	-0.0014 (9)	-0.0009 (8)	-0.0002 (8)
C2	0.0282 (11)	0.0334 (11)	0.0187 (9)	-0.0055 (9)	0.0016 (8)	-0.0002 (8)
C3	0.0422 (13)	0.0422 (13)	0.0230 (10)	-0.0154 (11)	0.0041 (10)	-0.0059 (10)
C4	0.0440 (14)	0.0596 (17)	0.0199 (10)	-0.0188 (13)	0.0004 (10)	-0.0054 (10)

supplementary materials

Geometric parameters (\AA , $^\circ$)

O1—H1	0.852 (13)	C2—C2 ⁱ	1.390 (4)
N1—C1	1.289 (3)	C2—C3	1.388 (3)
N1—O1	1.422 (2)	C3—C4	1.373 (3)
N2—C1	1.359 (2)	C3—H3A	0.9300
N2—C2	1.398 (3)	C4—C4 ⁱ	1.380 (5)
N2—H2A	0.8600	C4—H4A	0.9300
C1—C1 ⁱ	1.481 (4)		
N1—O1—H1	101.8 (17)	C3—C2—N2	121.7 (2)
C1—N1—O1	109.96 (16)	C2 ⁱ —C2—N2	118.68 (11)
C1—N2—C2	123.50 (18)	C4—C3—C2	120.1 (2)
C1—N2—H2A	118.3	C4—C3—H3A	120.0
C2—N2—H2A	118.3	C2—C3—H3A	120.0
N1—C1—N2	125.13 (19)	C3—C4—C4 ⁱ	120.25 (14)
N1—C1—C1 ⁱ	117.89 (12)	C3—C4—H4A	119.9
N2—C1—C1 ⁱ	116.98 (12)	C4 ⁱ —C4—H4A	119.9
C3—C2—C2 ⁱ	119.62 (13)		
C1—N2—C2—C3	-177.8 (2)	C2—N2—C1—C1 ⁱ	-11.4 (4)
C1—N2—C2—C2 ⁱ	2.2 (4)	C2 ⁱ —C2—C3—C4	-2.1 (4)
O1—N1—C1—N2	0.8 (3)	N2—C2—C3—C4	177.9 (2)
O1—N1—C1—C1 ⁱ	-178.7 (2)	C2—C3—C4—C4 ⁱ	0.4 (5)
C2—N2—C1—N1	169.1 (2)		

Symmetry codes: (i) $-x+5/4, -y+1/4, z$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots N1 ⁱⁱ	0.852 (13)	1.971 (10)	2.763 (2)	154
N2—H2A \cdots O1	0.86	2.25	2.566 (3)	102

Symmetry codes: (ii) $-x+1, -y, -z+1$.

Fig. 1

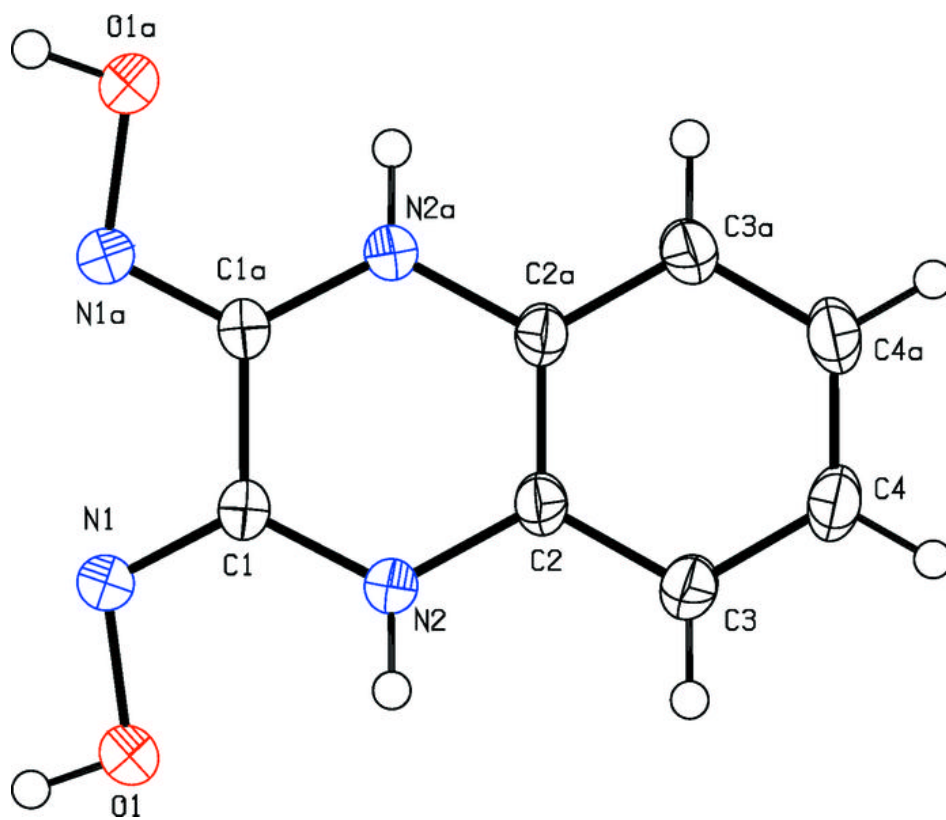


Fig. 2

