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## Structure Reports

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

Ali Kakanejadifard<sup>a\*</sup> and Vahid Amani<sup>b</sup><sup>a</sup>Department of Chemistry, Faculty of Science, Lorestan University, Khorramabad, Iran, and <sup>b</sup>Department of Chemistry, Islamic Azad University, Shahr-e-Rey Branch, Tehran, Iran

Correspondence e-mail: alikakanejadifard@yahoo.com

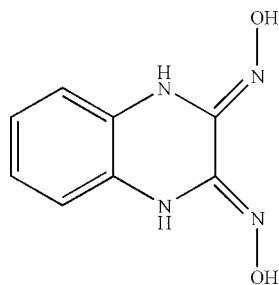
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Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.060;  $wR$  factor = 0.118; data-to-parameter ratio = 14.7.

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) Å<sup>3</sup>  
 $Z = 16$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.12$  mm<sup>-1</sup>  
 $T = 120$  (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<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.25$  e Å<sup>-3</sup>

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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## supporting information

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**(2Z,3Z)-Quinoxaline-2,3(1H,4H)-dione dioxime****Ali Kakanejadifard and Vahid Amani****S1. 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 di-aminoglyoxime 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.

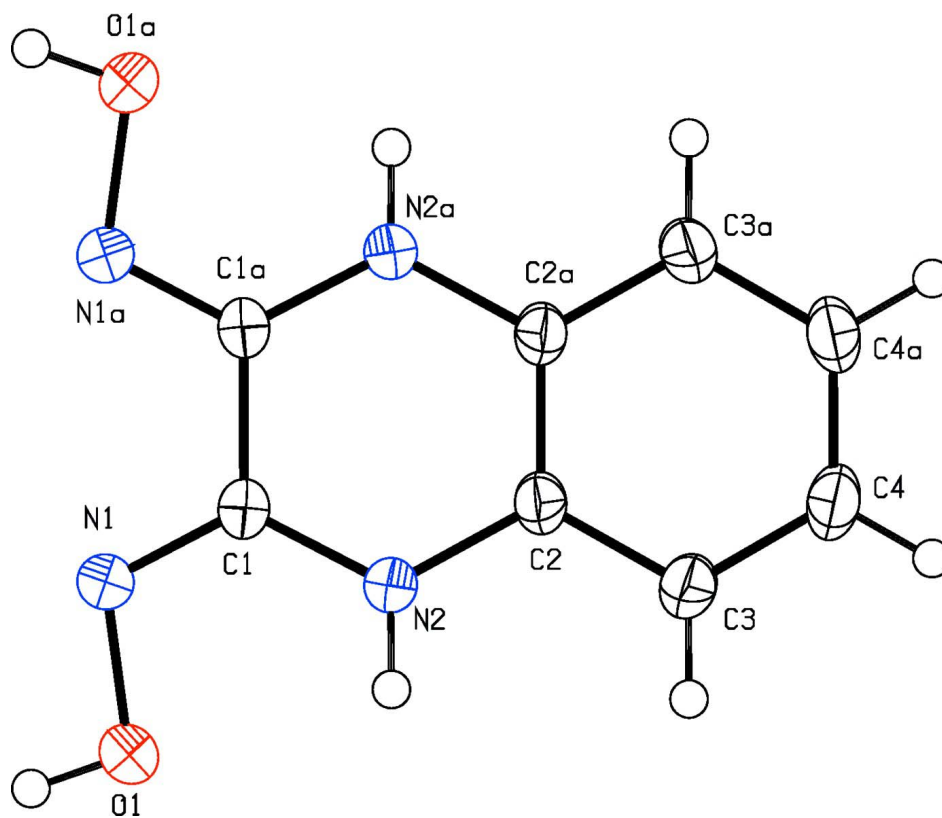
In the crystal structure, intermolecular O-H...N hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure.

**S2. Experimental**

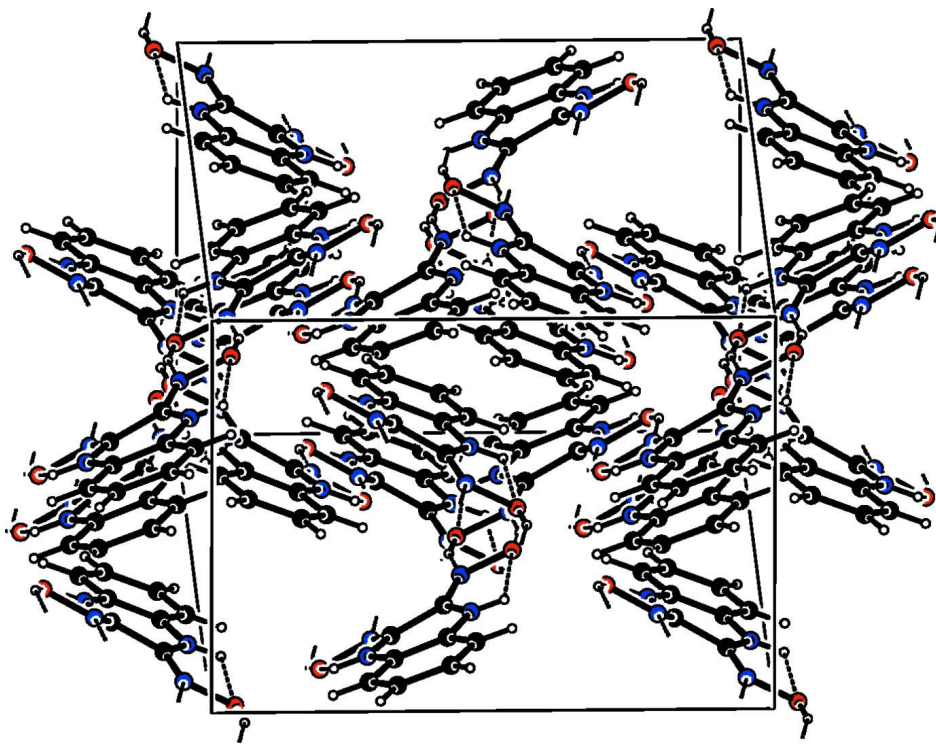
For the preparation of the title compound, a solution of Na<sub>2</sub>CO<sub>3</sub> (0.2 g, 1.9 mmol) in MeCN (30 ml) was added to a magnetically stirred solution of dichloroglyoxime (1.57 g, 10 mmol) in MeCN (20 ml) and a solution of 1,2-phenylenediamine (1.08 g, 10 mmol) at 278 K. After 2 h stirring 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).

**S3. Refinement**

H1 atom (for OH) was located in difference syntheses and refined [O-H = 0.852 (13) Å, U<sub>iso</sub>(H) = 0.039 Å<sup>2</sup>]. 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<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(C,N).

**Figure 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)  $5/4 - x, 1/4 - y, z$ ].



**Figure 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

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

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

$T = 120\ \text{K}$

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

Graphite monochromator

$\varphi$  and  $\omega$  scans

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  
 $R[F^2 > 2\sigma(F^2)] = 0.060$   
 $wR(F^2) = 0.118$   
 $S = 1.01$   
 983 reflections  
 67 parameters  
 1 restraint  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: difference Fourier map  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.005P)^2 + 25P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

Geometric parameters (Å, °)

O1—H1	0.852 (13)	C2—C2 <sup>i</sup>	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 <sup>i</sup>	1.380 (5)
N2—H2A	0.8600	C4—H4A	0.9300
C1—C1 <sup>i</sup>	1.481 (4)		
N1—O1—H1	101.8 (17)	C3—C2—N2	121.7 (2)
C1—N1—O1	109.96 (16)	C2 <sup>i</sup> —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 <sup>i</sup>	120.25 (14)
N1—C1—C1 <sup>i</sup>	117.89 (12)	C3—C4—H4A	119.9
N2—C1—C1 <sup>i</sup>	116.98 (12)	C4 <sup>i</sup> —C4—H4A	119.9
C3—C2—C2 <sup>i</sup>	119.62 (13)		
C1—N2—C2—C3	-177.8 (2)	C2—N2—C1—C1 <sup>i</sup>	-11.4 (4)
C1—N2—C2—C2 <sup>i</sup>	2.2 (4)	C2 <sup>i</sup> —C2—C3—C4	-2.1 (4)
O1—N1—C1—N2	0.8 (3)	N2—C2—C3—C4	177.9 (2)
O1—N1—C1—C1 <sup>i</sup>	-178.7 (2)	C2—C3—C4—C4 <sup>i</sup>	0.4 (5)
C2—N2—C1—N1	169.1 (2)		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1...N1 <sup>ii</sup>	0.85 (1)	1.97 (1)	2.763 (2)	154
N2—H2A...O1	0.86	2.25	2.566 (3)	102

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