

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# (*E,E*)-1-(2-Hydroxyimino-1-phenylethylidene)semicarbazide monohydrate

 Aslı Öztürk,<sup>a</sup> İlknur Babahan,<sup>b</sup> Nursabah Sarıkavaklı<sup>b</sup> and Tuncer Hökelek<sup>a\*</sup>
<sup>a</sup>Hacettepe University, Department of Physics, 06800 Beytepe, Ankara, Turkey, and

<sup>b</sup>Adnan Menderes University, Department of Chemistry, 09010 Aydın, Turkey

Correspondence e-mail: merzifon@hacettepe.edu.tr

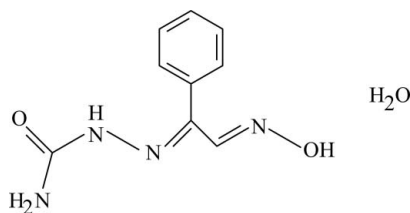
Received 23 March 2009; accepted 13 April 2009

 Key indicators: single-crystal X-ray study;  $T = 294$  K; mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å;  $R$  factor = 0.060;  $wR$  factor = 0.185; data-to-parameter ratio = 10.2.

In the title compound,  $\text{C}_9\text{H}_{10}\text{N}_4\text{O}_2 \cdot \text{H}_2\text{O}$ , the oxime unit has an *E* configuration, and an intramolecular  $\text{N}-\text{H} \cdots \text{N}$  hydrogen bond results in the formation of a planar five-membered ring, which is oriented with respect to the aromatic ring at a dihedral angle of  $74.82$  ( $17$ )°. In the crystal structure, intermolecular  $\text{O}-\text{H} \cdots \text{O}$  and  $\text{N}-\text{H} \cdots \text{O}$  hydrogen bonds link the molecules and  $R_2^2(8)$  ring motifs are apparent.

## Related literature

For general background, see: Balsamo *et al.* (1990); Marsman *et al.* (1999); Karle *et al.* (1996); Etter *et al.* (1990). For related structures, see: Sarıkavaklı *et al.* (2007, 2008); Özel Güven *et al.* (2007); Hökelek, Batı *et al.* (2001); Hökelek, Zülfikaroğlu *et al.* (2001); Büyükgüngör *et al.* (2003); Hökelek *et al.* (2004); Hökelek *et al.* (2004a,b). For reference structural data, see: Allen *et al.* (1987). For ring motifs, see: Bernstein *et al.* (1995).



## Experimental

### Crystal data

 $\text{C}_9\text{H}_{10}\text{N}_4\text{O}_2 \cdot \text{H}_2\text{O}$ 
 $M_r = 224.23$ 

 Triclinic,  $P\bar{1}$ 
 $a = 5.5593$  (2) Å

 $b = 8.2701$  (3) Å

 $c = 12.6193$  (5) Å

 $\alpha = 71.900$  (3)°

 $\beta = 89.998$  (5)°

 $\gamma = 78.538$  (5)°

 $V = 539.29$  (4) Å<sup>3</sup>
 $Z = 2$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.11$  mm<sup>-1</sup>
 $T = 294$  K

 $0.40 \times 0.25 \times 0.20$  mm

### Data collection

 Enraf–Nonius TurboCAD-4 diffractometer  
 Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
 $T_{\min} = 0.968$ ,  $T_{\max} = 0.978$   
 1953 measured reflections

 1752 independent reflections  
 867 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: 1%

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.060$ 
 $wR(F^2) = 0.185$ 
 $S = 1.05$ 

1752 reflections

171 parameters

5 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.19$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.34$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N3}-\text{H3A} \cdots \text{N1}$	0.88 (3)	2.32 (6)	2.647 (8)	102 (5)
$\text{O3}-\text{H31} \cdots \text{O1}$	0.88 (7)	1.92 (8)	2.776 (9)	164 (8)
$\text{O3}-\text{H32} \cdots \text{O3}^i$	0.90 (3)	2.17 (7)	2.909 (11)	140 (7)
$\text{N2}-\text{H22} \cdots \text{O3}^{ii}$	0.82 (3)	2.10 (4)	2.901 (10)	162 (5)
$\text{N3}-\text{H3B} \cdots \text{O1}^{iii}$	0.96 (7)	1.96 (6)	2.909 (8)	169 (6)

 Symmetry codes: (i)  $-x, -y, -z + 2$ ; (ii)  $-x + 1, -y, -z + 2$ ; (iii)  $-x + 1, -y - 1, -z + 2$ .

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the purchase of the CAD-4 diffractometer under grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2932).

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Balsamo, A., Macchia, B., Martinelli, A., Orlandini, E., Rossello, A., Macchia, F., Bocelli, G. & Domiano, P. (1990). *Eur. J. Med. Chem.* **25**, 227–233.
- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Büyükgüngör, O., Hökelek, T., Taş, M. & Batı, H. (2003). *Acta Cryst.* **E59**, o883–o885.
- Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- Hökelek, T., Batı, H., Bekdemir, Y. & Kütük, H. (2001). *Acta Cryst.* **E57**, o663–o665.
- Hökelek, T., Büyükgüngör, O., Taş, M. & Batı, H. (2004a). *Acta Cryst.* **E60**, o109–o111.
- Hökelek, T., Büyükgüngör, O., Taş, M. & Batı, H. (2004b). *Acta Cryst.* **E60**, o406–o408.
- Hökelek, T., Taş, M. & Batı, H. (2004). *Cryst. Res. Technol.* **39**, 363–367.
- Hökelek, T., Zülfikaroğlu, A. & Batı, H. (2001). *Acta Cryst.* **E57**, o1247–o1249.

- Karle, I. L., Ranganathan, D. & Haridas, V. (1996). *J. Am. Chem. Soc.* **118**, 7128–7133.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Marsman, A. W., Leussing, E. D., Zwikker, J. W. & Jenneskens, L. W. (1999). *Chem. Mater.* **11**, 1484–1491.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Özel Güven, Ö., Erdoğan, T., Çaylak, N. & Hökelek, T. (2007). *Acta Cryst.* **E63**, o3463–o3464.
- Sarıkavaklı, N., Babahan, İ., Şahin, E. & Hökelek, T. (2008). *Acta Cryst.* **E64**, o623–o624.
- Sarıkavaklı, N., Şahin, E. & Hökelek, T. (2007). *Acta Cryst.* **E63**, o3601.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

**supplementary materials**

*Acta Cryst.* (2009). E65, o1059-o1060 [ doi:10.1107/S1600536809013865 ]

## (*E,E*)-1-(2-Hydroxyimino-1-phenylethylidene)semicarbazide monohydrate

A. Öztürk, I. Babahan, N. Sarıkavaklı and T. Hökelek

### Comment

Oxime and dioxime derivatives have a broad pharmacological activity spectrum, encompassing antibacterial, antidepressant and antifungal activities (e.g. Balsamo *et al.*, 1990). The oxime ( $-C=N-OH$ ) moiety is potentially ambidentate, with possibilities of coordination to metal ions through nitrogen and/or oxygen atoms. Oxime groups possess stronger hydrogen-bonding capabilities than alcohols, phenols, and carboxylic acids (Marsman *et al.*, 1999), in which intermolecular hydrogen bonding combines moderate strength and directionality (Karle *et al.*, 1996) in linking molecules to form supramolecular structures; this has received considerable attention with respect to directional noncovalent intermolecular interactions (Etter *et al.*, 1990).

The structures of some oxime and dioxime derivatives have been determined in our laboratory, including those of 2,3-dimethylquinoxaline-dimethyl-glyoxime (I/1), [(II) Hökelek, Batu *et al.*, 2001], 1-(2,6-dimethylphenylamino) propane-1,2-dione dioxime, [(III) (Hökelek, Zülfikaroğlu *et al.*, 2001), *N*-hydroxy-2-oxo-2,*N'*-diphenylacetamide, [(IV) (Büyükgüngör *et al.*, 2003), *N*-(3,4-dichlorophenyl)-*N'*-hydroxy-2-oxo-2-phenylacetamide, [(V) Hökelek *et al.*, 2004], *N*-hydroxy-*N'*-(1-naphthyl)-2-phenylacetamidin-2-one [(VI) Hökelek *et al.*, 2004a], *N*-(3-chloro-4-methylphenyl)-*N'*-hydroxy-2-oxo-2-phenylacetamide [(VII) Hökelek *et al.*, 2004b], 2-(1*H*-benzimidazol-1-yl)-1-phenylethanone oxime [(VIII) Özel Güven *et al.*, 2007], (1*Z*,2*E*)-1-(3,5-dimethyl-1*H*-pyrazole-1-yl)ethane-1,2-dione dioxime [(IX) Sarıkavaklı *et al.*, 2007] and 2-hydroxyimino-1-phenylethanone thiosemicarbazone monohydrate [(X) Sarıkavaklı *et al.*, 2008].

As part of our ongoing studies in this area, the structure determination of the title compound, (I), an oxime derivative with one semicarbazide, one phenylacetaldehyde oxime moieties and one uncoordinated water molecule, was carried out in order to investigate the strength of the hydrogen bonding capability of the oxime and semicarbazide groups and to compare the geometry of the oxime moiety with the previously reported ones.

In the molecule of the title compound, (I), (Fig. 1) the bond lengths (Allen *et al.*, 1987) and angles are generally within normal ranges. Ring A (C1—C6) is, of course, planar. The intramolecular N—H $\cdots$ N hydrogen bond (Table 1) results in the formation of a planar five-membered ring B (N1—N3/C8/H3A). The dihedral angle between the planar rings is A/B = 74.82 (17)°.

In the crystal structure, intramolecular O—H $\cdots$ O and N—H $\cdots$ N and intermolecular O—H $\cdots$ O and N—H $\cdots$ O hydrogen bonds (Table 1) link the molecules through  $R_2^2(8)$  ring motifs (Bernstein *et al.*, 1995) (Fig. 2).

### Experimental

Semicarbazide hydrochloride (1.12 g, 10 mmol) and sodium acetate (0.82 g, 10 mmol) were dissolved in double distilled water in the molar ratio 1:1. Then, the solution was mixed with a solution of 2-isonitrosoacetophenone (1.49 g, 10 mmol) in ethanol (10 ml) yielding a turbid mixture. The excess ethanol was added to get a clear solution and was stirred in a magnetic stirrer at room temperature for 4 h. The precipitate formed was filtered, washed with water and dried at room temperature

## supplementary materials

in vacuum desiccator. It was recrystallized from ethanol/water (2:1) solution to yield colourless prisms of (I) (yield; 1.80 g, 85%, m.p. 409 K).

### Refinement

Atoms H9 (for CH), H21 (for OH), H22 (for NH), H3A, H3B (for NH<sub>2</sub>) and H31, H32 (for H<sub>2</sub>O) were located in difference Fourier maps and refined isotropically, with restrains of O3—H31 = 0.88 (7), O3—H32 = 0.90 (3), N2—H22 = 0.82 (3), N3—H3A = 0.88 (3) Å and H31—O3—H32 = 105 (4)° [ $U_{\text{iso}}(\text{H}) = 0.064$  (19) Å<sup>2</sup> (for CH), 0.09 (3) Å<sup>2</sup> (for OH), 0.040 (17) Å<sup>2</sup> (for NH), 0.08 (2) Å<sup>2</sup> (for NH<sub>2</sub>) and 0.125 Å<sup>2</sup> (for H<sub>2</sub>O)]. The remaining H atoms were positioned geometrically with C—H = 0.93 Å and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

### Figures

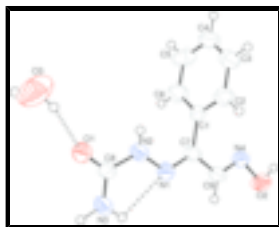


Fig. 1. The molecular structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

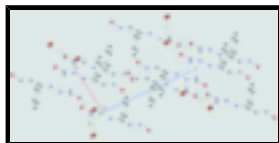


Fig. 2. A partial packing diagram of (I). Hydrogen bonds are shown as dotted lines.

### (*E,E*)-1-(2-Hydroxyimino-1-phenylethylidene)semicarbazide monohydrate

#### Crystal data

C<sub>9</sub>H<sub>10</sub>N<sub>4</sub>O<sub>2</sub>·H<sub>2</sub>O

$M_r = 224.23$

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

$a = 5.5593$  (2) Å

$b = 8.2701$  (3) Å

$c = 12.6193$  (5) Å

$\alpha = 71.900$  (3)°

$\beta = 89.998$  (5)°

$\gamma = 78.538$  (5)°

$V = 539.29$  (4) Å<sup>3</sup>

$Z = 2$

$F_{000} = 236$

$D_x = 1.381$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 25 reflections

$\theta = 8.6$ – $17.3$ °

$\mu = 0.11$  mm<sup>-1</sup>

$T = 294$  K

Prism, colorless

$0.40 \times 0.25 \times 0.20$  mm

#### Data collection

Enraf–Nonius TurboCAD-4  
diffractometer

$R_{\text{int}} = 0.048$

Radiation source: fine-focus sealed tube	$\theta_{\max} = 24.3^\circ$
Monochromator: graphite	$\theta_{\min} = 3.4^\circ$
$T = 294$ K	$h = -6 \rightarrow 0$
Non-profiled $\omega$ scans	$k = -9 \rightarrow 9$
Absorption correction: $\psi$ scan (North <i>et al.</i> , 1968)	$l = -14 \rightarrow 14$
$T_{\min} = 0.968$ , $T_{\max} = 0.978$	3 standard reflections
1953 measured reflections	every 120 min
1752 independent reflections	intensity decay: 1%
867 reflections with $I > 2\sigma(I)$	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.060$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.185$	$w = 1/[\sigma^2(F_o^2) + (0.0859P)^2]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
1752 reflections	$(\Delta/\sigma)_{\max} < 0.001$
171 parameters	$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$
5 restraints	$\Delta\rho_{\min} = -0.34 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.5650 (8)	-0.2847 (5)	0.9749 (4)	0.0635 (13)
O2	1.6136 (8)	-0.2215 (5)	0.5421 (4)	0.0583 (13)
H21	1.658 (12)	-0.131 (10)	0.490 (6)	0.09 (3)*
O3	0.2334 (12)	-0.0741 (10)	1.0657 (7)	0.132 (3)
H31	0.344 (10)	-0.122 (11)	1.028 (6)	0.125*
H32	0.088 (7)	-0.084 (10)	1.041 (7)	0.125*

## supplementary materials

---

N1	1.0377 (8)	-0.2812 (6)	0.7948 (4)	0.0456 (13)
N2	0.8573 (9)	-0.2253 (6)	0.8554 (4)	0.0498 (14)
H22	0.823 (9)	-0.128 (4)	0.863 (4)	0.040 (17)*
N3	0.7601 (11)	-0.4938 (7)	0.9070 (5)	0.0594 (16)
H3A	0.884 (8)	-0.531 (8)	0.872 (5)	0.08 (2)*
H3B	0.665 (11)	-0.578 (9)	0.942 (5)	0.08 (2)*
N4	1.4358 (8)	-0.1346 (5)	0.5950 (4)	0.0435 (13)
C1	1.0910 (10)	0.0218 (7)	0.7132 (5)	0.0389 (14)
C2	1.2415 (11)	0.0996 (7)	0.7590 (5)	0.0538 (17)
H2	1.3800	0.0317	0.8044	0.065*
C3	1.1895 (12)	0.2758 (8)	0.7384 (6)	0.0646 (19)
H3	1.2905	0.3257	0.7719	0.077*
C4	0.9938 (13)	0.3786 (8)	0.6700 (6)	0.0602 (18)
H4	0.9626	0.4987	0.6543	0.072*
C5	0.8426 (12)	0.3024 (8)	0.6244 (5)	0.065 (2)
H5	0.7064	0.3716	0.5778	0.078*
C6	0.8891 (12)	0.1243 (8)	0.6466 (5)	0.0591 (18)
H6	0.7829	0.0742	0.6162	0.071*
C7	1.1479 (10)	-0.1688 (6)	0.7314 (4)	0.0387 (14)
C8	0.7189 (11)	-0.3364 (7)	0.9167 (5)	0.0455 (15)
C9	1.3383 (11)	-0.2382 (8)	0.6696 (5)	0.0444 (15)
H9	1.374 (10)	-0.359 (9)	0.687 (5)	0.064 (19)*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.066 (3)	0.051 (3)	0.078 (3)	-0.018 (2)	0.036 (3)	-0.024 (2)
O2	0.062 (3)	0.044 (3)	0.064 (3)	-0.005 (2)	0.026 (2)	-0.014 (2)
O3	0.106 (5)	0.148 (6)	0.173 (7)	0.002 (5)	0.008 (5)	-0.111 (5)
N1	0.049 (3)	0.041 (3)	0.048 (3)	-0.012 (2)	0.017 (3)	-0.013 (2)
N2	0.058 (3)	0.036 (3)	0.057 (3)	-0.015 (3)	0.019 (3)	-0.014 (3)
N3	0.065 (4)	0.043 (3)	0.076 (4)	-0.022 (3)	0.030 (3)	-0.019 (3)
N4	0.046 (3)	0.037 (3)	0.046 (3)	-0.006 (2)	0.009 (2)	-0.012 (2)
C1	0.038 (3)	0.035 (3)	0.041 (3)	-0.007 (3)	0.011 (3)	-0.010 (3)
C2	0.050 (4)	0.042 (4)	0.066 (4)	-0.005 (3)	-0.008 (3)	-0.014 (3)
C3	0.062 (4)	0.046 (4)	0.089 (5)	-0.016 (4)	-0.003 (4)	-0.024 (4)
C4	0.074 (5)	0.035 (3)	0.071 (5)	-0.013 (4)	0.013 (4)	-0.016 (3)
C5	0.068 (5)	0.045 (4)	0.067 (5)	0.010 (4)	-0.011 (4)	-0.007 (4)
C6	0.060 (4)	0.045 (4)	0.065 (4)	-0.008 (3)	-0.014 (4)	-0.010 (3)
C7	0.043 (3)	0.032 (3)	0.037 (3)	-0.009 (3)	0.002 (3)	-0.005 (3)
C8	0.049 (4)	0.035 (3)	0.046 (4)	-0.009 (3)	0.012 (3)	-0.005 (3)
C9	0.051 (4)	0.031 (3)	0.051 (4)	-0.011 (3)	0.007 (3)	-0.012 (3)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

O1—C8	1.226 (6)	C2—H2	0.9300
O2—N4	1.399 (5)	C3—H3	0.9300
O2—H21	0.91 (8)	C4—C3	1.352 (9)
O3—H31	0.88 (7)	C4—C5	1.368 (9)

O3—H32	0.90 (3)	C4—H4	0.9300
N1—C7	1.281 (6)	C5—C6	1.381 (8)
N2—N1	1.357 (6)	C5—H5	0.9300
N2—C8	1.369 (7)	C6—H6	0.9300
N2—H22	0.82 (3)	C7—C1	1.489 (7)
N3—H3A	0.88 (3)	C8—N3	1.320 (7)
N3—H3B	0.96 (7)	C9—N4	1.264 (7)
C1—C2	1.376 (8)	C9—C7	1.447 (7)
C1—C6	1.365 (8)	C9—H9	0.94 (6)
C2—C3	1.368 (8)		
N4—O2—H21	101 (4)	C3—C4—C5	118.6 (6)
H31—O3—H32	105 (4)	C3—C4—H4	120.7
C7—N1—N2	118.1 (5)	C5—C4—H4	120.7
N1—N2—C8	120.5 (5)	C4—C5—C6	121.0 (6)
N1—N2—H22	126 (4)	C4—C5—H5	119.5
C8—N2—H22	113 (4)	C6—C5—H5	119.5
C8—N3—H3A	122 (4)	C1—C6—C5	120.0 (6)
C8—N3—H3B	123 (4)	C1—C6—H6	120.0
H3A—N3—H3B	115 (6)	C5—C6—H6	120.0
C9—N4—O2	112.3 (4)	N1—C7—C1	126.4 (5)
C2—C1—C7	121.7 (5)	N1—C7—C9	114.9 (5)
C6—C1—C2	118.6 (5)	C9—C7—C1	118.7 (5)
C6—C1—C7	119.7 (5)	O1—C8—N2	119.1 (5)
C1—C2—H2	119.7	O1—C8—N3	124.4 (6)
C3—C2—C1	120.7 (6)	N3—C8—N2	116.5 (5)
C3—C2—H2	119.7	N4—C9—C7	119.3 (5)
C2—C3—H3	119.5	N4—C9—H9	125 (4)
C4—C3—C2	121.1 (6)	C7—C9—H9	116 (3)
C4—C3—H3	119.5		
N2—N1—C7—C1	-2.8 (8)	C5—C4—C3—C2	2.2 (10)
N2—N1—C7—C9	179.9 (5)	C3—C4—C5—C6	-0.6 (10)
C8—N2—N1—C7	174.2 (5)	C4—C5—C6—C1	-1.2 (10)
N1—N2—C8—O1	176.6 (5)	N1—C7—C1—C2	106.3 (7)
N1—N2—C8—N3	-4.4 (8)	N1—C7—C1—C6	-75.8 (8)
C6—C1—C2—C3	0.1 (9)	C9—C7—C1—C2	-76.5 (7)
C7—C1—C2—C3	178.0 (6)	C9—C7—C1—C6	101.4 (6)
C2—C1—C6—C5	1.4 (9)	N4—C9—C7—N1	171.4 (5)
C7—C1—C6—C5	-176.5 (6)	N4—C9—C7—C1	-6.1 (8)
C1—C2—C3—C4	-2.0 (10)	C7—C9—N4—O2	-179.2 (5)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3A $\cdots$ N1	0.88 (3)	2.32 (6)	2.647 (8)	102 (5)
O3—H31 $\cdots$ O1	0.88 (7)	1.92 (8)	2.776 (9)	164 (8)
O3—H32 $\cdots$ O3 <sup>i</sup>	0.90 (3)	2.17 (7)	2.909 (11)	140 (7)
N2—H22 $\cdots$ O3 <sup>ii</sup>	0.82 (3)	2.10 (4)	2.901 (10)	162 (5)
N3—H3B $\cdots$ O1 <sup>iii</sup>	0.96 (7)	1.96 (6)	2.909 (8)	169 (6)

# supplementary materials

---

Symmetry codes: (i)  $-x, -y, -z+2$ ; (ii)  $-x+1, -y, -z+2$ ; (iii)  $-x+1, -y-1, -z+2$ .

Fig. 1

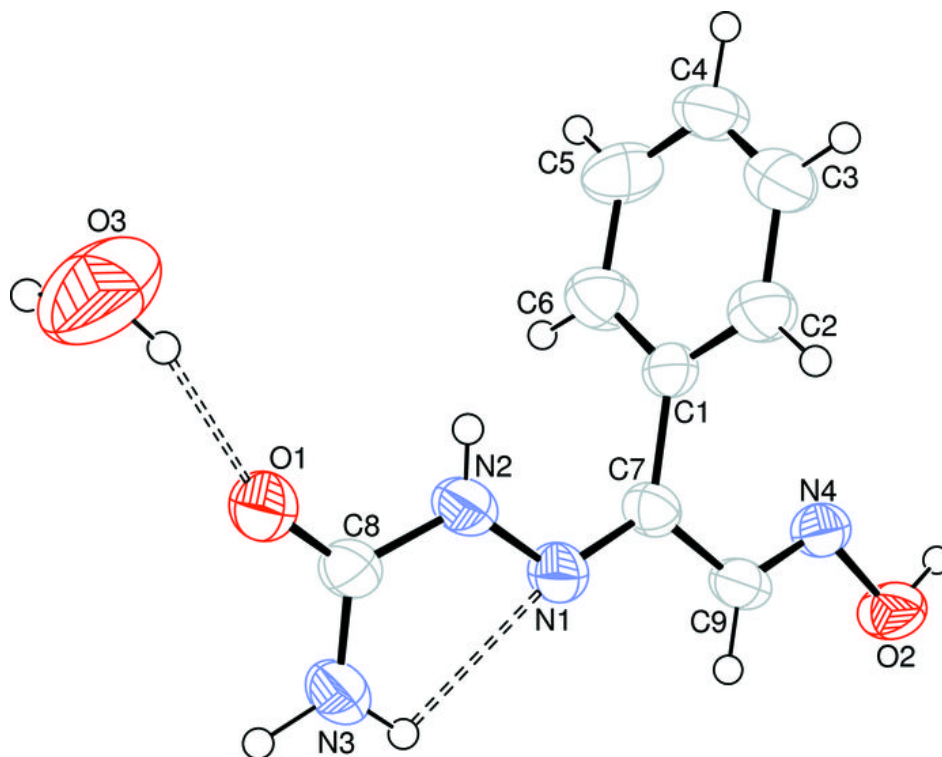


Fig. 2

