

2-Hydroxyimino-1-phenylethanone thiosemicarbazone monohydrate

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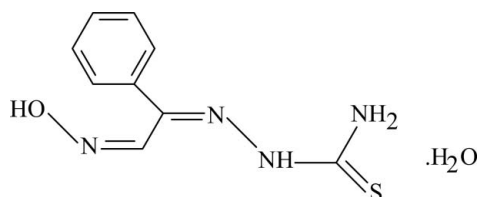
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.062; wR factor = 0.155; data-to-parameter ratio = 18.7.

In the title thiosemicarbazone derivative, $\text{C}_9\text{H}_{10}\text{N}_4\text{OS}\cdot\text{H}_2\text{O}$, intramolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds result in the formation of two nearly coplanar five- and six-membered rings, which are also almost coplanar with the adjacent phenyl ring. The oxime group has an E configuration and is involved in intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding as a donor. In the crystal structure, intramolecular $\text{O}-\text{H}\cdots\text{S}$ and $\text{N}-\text{H}\cdots\text{N}$ and intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds generate edge-fused $R_2^2(8)$ and $R_4^1(11)$ ring motifs. The hydrogen-bonded motifs are linked to each other to form a three-dimensional supramolecular network.

Related literature

For general background, see: Lukevics *et al.* (1995); Liberta & West (1992); Hagenbach & Gysin (1952); Jones *et al.* (1965); Brockman & Thomson (1956); Klayman *et al.* (1979); Petering & van Giesen (1966); Sevagapandian *et al.* (2000); Forman (1964); Holan *et al.* (1984); Balsamo *et al.* (1990); Marsman *et al.* (1999); Karle *et al.* (1996); Etter *et al.* (1990); Chertanova *et al.* (1994); Bernstein *et al.* (1995). For related structures, see: Sarıkavaklı *et al.* (2007); Özel Güven *et al.* (2007); Hökelek, Batı *et al.* (2001); Hökelek, Zülfikaroğlu & Batı (2001); Büyükgüngör *et al.* (2003); Hökelek *et al.* (2004a,b); Hökelek *et al.* (2004). For the synthesis, see: El-Shazly *et al.* (2005). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_9\text{H}_{10}\text{N}_4\text{OS}\cdot\text{H}_2\text{O}$

$M_r = 240.29$

Monoclinic, $C2/c$

$a = 28.5615$ (3) Å

$b = 4.6805$ (3) Å

$c = 22.0977$ (4) Å

$\beta = 127.24$ (2)°

$V = 2351.8$ (6) Å³

$Z = 8$

Mo $K\alpha$ radiation

$\mu = 0.27$ mm⁻¹

$T = 294$ (2) K

$0.30 \times 0.20 \times 0.15$ mm

Data collection

Rigaku R-Axis RAPID-S diffractometer

Absorption correction: multi-scan (Blessing, 1995)

$T_{\min} = 0.940$, $T_{\max} = 0.960$

31269 measured reflections

3607 independent reflections

2146 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.090$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.062$

$wR(F^2) = 0.154$

$S = 1.04$

3607 reflections

193 parameters

8 restraints

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.14$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1A}\cdots\text{O2}^i$	0.90 (3)	1.83 (3)	2.728 (3)	174 (3)
$\text{O2}-\text{H21}\cdots\text{S1}$	0.94 (3)	2.32 (3)	3.250 (3)	171 (3)
$\text{O2}-\text{H22}\cdots\text{O2}^i$	0.91 (3)	1.98 (3)	2.886 (3)	172 (4)
$\text{N3}-\text{H3A}\cdots\text{N1}$	0.92 (3)	1.91 (2)	2.604 (3)	130 (2)
$\text{N4}-\text{H41}\cdots\text{S1}^{ii}$	0.92 (2)	2.53 (2)	3.434 (2)	169 (3)
$\text{N4}-\text{H42}\cdots\text{N2}$	0.93 (3)	2.24 (3)	2.643 (3)	105 (2)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2403).

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., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Brockman, R. W. & Thomson, J. R. (1956). *Cancer Res.* **16**, 167–170.
- Büyükgüngör, O., Hökelek, T., Taş, M. & Batı, H. (2003). *Acta Cryst.* **E59**, o883–o885.

- Chertanova, L., Pascard, C. & Sheremetev, A. (1994). *Acta Cryst.* **B50**, 708–716.
- El-Shazly, R. M., Al-Hazmi, G. A. A., Ghazy, S. E., El-Shahawi, M. S. & El-Asmy, A. A. (2005). *Spectrochim. Acta*, **A61**, 243–252.
- 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.
- Forman, S. E. (1964). *J. Org. Chem.* **29**, 3323–3327.
- Hagenbach, R. E. & Gysin, H. (1952). *Experientia*, **8**, 184–185.
- Hökelek, T., Batu, H., Bekdemir, Y. & Kütük, H. (2001). *Acta Cryst.* **E57**, o663–o665.
- Hökelek, T., Büyükgüngör, O., Taş, M. & Batu, H. (2004a). *Acta Cryst.* **E60**, o109–o111.
- Hökelek, T., Büyükgüngör, O., Taş, M. & Batu, H. (2004b). *Acta Cryst.* **E60**, o406–o408.
- Hökelek, T., Taş, M. & Batu, H. (2004). *Cryst. Res. Technol.* **39**, 363–367.
- Hökelek, T., Zülfikaroğlu, A. & Batu, H. (2001). *Acta Cryst.* **E57**, o1247–o1249.
- Holan, G., Johnson, W. M. P., Rihs, K. & Virgona, C. T. (1984). *Pestic. Sci.* **15**, 361–368.
- Jones, D. H., Slack, R., Squires, S. & Woolridge, K. R. H. (1965). *J. Med. Chem.* **8**, 676–680.
- Karle, I. L., Ranganathan, D. & Haridas, V. (1996). *J. Am. Chem. Soc.* **118**, 7128–7133.
- Klayman, D. L., Bartoserich, J. F., Griffin, T. S., Manson, C. J. & Scovill, J. P. (1979). *J. Med. Chem.* **22**, 885–893.
- Liberta, A. E. & West, D. X. (1992). *Biometals*, **5**, 121–125.
- Lukevics, E., Jansone, D., Rubina, K., Abele, E., Germane, S., Leite, L., Shymaska, M. & Popelis, J. (1995). *Eur. J. Med. Chem.* **30**, 983–986.
- Marsman, A. W., Leussing, E. D., Zwikker, J. W. & Jenneskens, L. W. (1999). *Chem. Mater.* **11**, 1484–1491.
- Özel Güven, Ö., Erdoğan, T., Çaylak, N. & Hökelek, T. (2007). *Acta Cryst.* **E63**, o3463–o3464.
- Petering, H. G. & van Giesen, G. J. (1966). *The Biochemistry of Copper*, pp. 197–208. New York: Harriman.
- Rigaku/MS (2005). *CrystalClear*. Rigaku/MS, The Woodlands, Texas, USA.
- Sarikavaklı, N., Şahin, E. & Hökelek, T. (2007). *Acta Cryst.* **E63**, o3601.
- Sevagapandian, S., Rjagopal, G., Nehru, K. & Athappan, P. (2000). *Transition Met. Chem.* **25**, 388–393.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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Acta Cryst. (2008). E64, o623-o624 [doi:10.1107/S1600536808004947]

2-Hydroxyimino-1-phenylethanone thiosemicarbazone monohydrate

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Comment

Thiosemicarbazones are derivatives of carbonyl compounds and they have a wide range of biological activities, depending on the parent aldehyde or ketone (Lukevics *et al.*, 1995; Liberta & West, 1992). Some of the thiosemicarbazone derivatives have antitumour (Hagenbach & Gysin, 1952), antiviral (Jones *et al.*, 1965), antileukaemic (Brockman & Thomson, 1956) and antimalarial (Klayman *et al.*, 1979) activities. Thus, some of them have been used as drugs and have the ability to form complexes (Petering & van Giesen, 1966).

Oxime and dioxime derivatives are very important compounds in the chemical industry and medicine (Sevagapandian *et al.*, 2000). They have a broad pharmacological activity spectrum, encompassing antibacterial, antidepressant and antifungal activities (Forman, 1964; Holan *et al.*, 1984; Balsamo *et al.*, 1990). The oxime ($-C=N-OH$) moiety is potentially ambidentate, with possibilities of coordination through nitrogen and/or oxygen atoms. It is a functional group that has not been extensively explored in crystal engineering. In the solid state, oximes are usually associated *via* $O-H\cdots N$ hydrogen bonds of length 2.8 Å.

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, Batı *et al.*, 2001], 1-(2,6-dimethylphenylamino)-propane-1,2-dione dioxime, [(III) Hökelek, Zülfikaroğlu & Batı, 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-phenylacetamide-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] and (1*Z*,2*E*)-1-(3,5-dimethyl-1*H*-pyrazole-1-yl)ethane-1,2-dione dioxime [(IX) Sarikavaklı *et al.*, 2007]. The structure determination of the title compound, (I), a thiosemicarbazone derivative with one 2-hydroxyimino-1-phenyl-ethanone, one thiosemicarbazone moieties and one uncoordinated water molecule, was carried out in order to investigate the strength of the hydrogen bonding capability of the oxime and thiosemicarbazone 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 bonds (Table 1) result in the formation of two more planar five- and six-membered rings B (C9/N2—N4/H42) and C (C7/C8/N1—N3/H3A). The rings A, B and C are also nearly coplanar with dihedral angles of A/B = 3.47 (10)°, A/C = 2.84 (10) and B/C = 5.94 (10)°.

Some significant changes in the geometry of the oxime moiety are evident when the bond lengths and angles are compared with the corresponding values in compounds (II)-(VII) (Table 2). The oxime moiety has an E configuration

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[C7—C8—N1—O1 177.1 (2)°; Chertanova *et al.*, 1994]. In this configuration, the oxime groups are involved as donors in O—H···O intermolecular hydrogen bondings (Table 1).

In the crystal structure, intramolecular O—H···S and N—H···N and intermolecular O—H···O and N—H···S hydrogen bonds (Table 1) generate edge-fused $R_2^2(8)$ and $R_4^1(11)$ ring motifs (Fig. 2) (Bernstein *et al.*, 1995). The hydrogen bonded motifs are linked to each other to form a three dimensional network (Fig. 3). The intra- and intermolecular hydrogen bonds seem to be effective in the stabilization of the crystal structure.

Experimental

The title compound was prepared according to the literature method (El-Shazly *et al.*, 2005). 2-Isonitrosoacetophenone (149 mg, 1 mmol) was reacted with thiosemicarbazide (91 mg, 1 mmol) in ethanol-water mixture (1:1) by refluxing for 24 h. Then, a few drops of glacial acetic acid were added. The formed precipitate was filtered and recrystallized from ethanol to obtain yellow crystals (yield: 155 mg, 70%).

Refinement

H atoms were located in difference syntheses and refined isotropically [O—H = 0.903 (18)–0.940 (17) Å; $U_{\text{iso}}(\text{H}) = 0.100(10)$ – $0.133(14)$ Å², N—H = 0.915 (17)–0.930 (17) Å; $U_{\text{iso}}(\text{H}) = 0.077(8)$ – $0.084(9)$ Å² and C—H = 0.92 (3)–0.97 (3) Å; $U_{\text{iso}}(\text{H}) = 0.071(8)$ – $0.092(9)$ Å²]. The restrains on the O—H (for OH) and N—H (for NH and NH₂) bonds and O—H bond lengths and H—O—H bond angle of water molecule were applied.

Figures

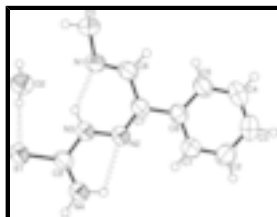


Fig. 1. The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

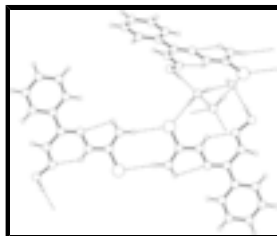


Fig. 2. A part of the crystal structure of (I), showing the formation of $R_2^2(8)$ and $R_4^1(11)$ ring motifs. Hydrogen bonds are shown as dashed lines.

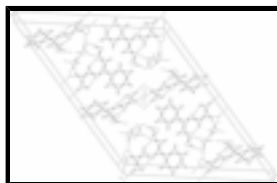


Fig. 3. A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

2-Hydroxyimino-1-phenylethanone thiosemicarbazone monohydrate

Crystal data

$C_9H_{10}N_4OS \cdot H_2O$	$F_{000} = 1008$
$M_r = 240.29$	$D_x = 1.357 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Melting point: 443 K
Hall symbol: $-C 2yc$	Mo $K\alpha$ radiation
$a = 28.5615 (3) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 4.6805 (3) \text{ \AA}$	Cell parameters from 2893 reflections
$c = 22.0977 (4) \text{ \AA}$	$\theta = 2.3\text{--}30.5^\circ$
$\beta = 127.24 (2)^\circ$	$\mu = 0.27 \text{ mm}^{-1}$
$V = 2351.8 (6) \text{ \AA}^3$	$T = 294 (2) \text{ K}$
$Z = 8$	Prism, yellow
	$0.30 \times 0.20 \times 0.15 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID-S diffractometer	3607 independent reflections
Radiation source: fine-focus sealed tube	2146 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.090$
$T = 294(2) \text{ K}$	$\theta_{\text{max}} = 30.5^\circ$
ω scans	$\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (Blessing, 1995)	$h = -40 \rightarrow 40$
$T_{\text{min}} = 0.940, T_{\text{max}} = 0.960$	$k = -6 \rightarrow 5$
31269 measured reflections	$l = -31 \rightarrow 31$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.062$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.154$	$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 0.9027P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
3607 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
193 parameters	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
8 restraints	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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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	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.08587 (3)	0.69943 (16)	0.58554 (3)	0.0693 (2)
O1	0.24428 (8)	0.9359 (4)	0.88734 (10)	0.0740 (5)
H1A	0.2527 (12)	1.022 (6)	0.8584 (15)	0.100 (10)*
O2	0.22848 (8)	0.6577 (4)	0.70204 (11)	0.0720 (5)
H21	0.1878 (8)	0.692 (7)	0.6673 (16)	0.133 (14)*
H22	0.2445 (13)	0.817 (5)	0.7313 (16)	0.113 (12)*
N1	0.19953 (8)	0.7460 (4)	0.83853 (10)	0.0576 (5)
N2	0.09978 (8)	0.3607 (4)	0.75639 (10)	0.0577 (5)
N3	0.10929 (9)	0.5206 (5)	0.71329 (10)	0.0619 (5)
H3A	0.1412 (9)	0.641 (5)	0.7347 (14)	0.077 (8)*
N4	0.02448 (9)	0.3422 (5)	0.60650 (11)	0.0654 (6)
H41	-0.0011 (10)	0.316 (5)	0.5550 (10)	0.076 (8)*
H42	0.0210 (12)	0.238 (5)	0.6394 (14)	0.084 (9)*
C1	0.07415 (11)	0.0295 (6)	0.83508 (14)	0.0644 (6)
H1	0.0517 (11)	0.012 (5)	0.7826 (15)	0.074 (8)*
C2	0.06113 (12)	-0.1405 (6)	0.87378 (17)	0.0721 (7)
H2	0.0280 (11)	-0.257 (5)	0.8455 (15)	0.071 (8)*
C3	0.09577 (13)	-0.1362 (6)	0.95201 (17)	0.0724 (7)
H3	0.0859 (12)	-0.245 (6)	0.9780 (16)	0.083 (9)*
C4	0.14393 (14)	0.0388 (6)	0.99041 (16)	0.0742 (7)
H4	0.1666 (12)	0.036 (6)	1.0426 (16)	0.092 (9)*
C5	0.15737 (13)	0.2112 (6)	0.95211 (14)	0.0639 (6)
H5	0.1913 (13)	0.330 (6)	0.9810 (17)	0.089 (9)*
C6	0.12248 (9)	0.2111 (5)	0.87338 (12)	0.0521 (5)
C7	0.13487 (9)	0.3927 (5)	0.82959 (11)	0.0516 (5)
C8	0.18351 (10)	0.5985 (5)	0.87101 (13)	0.0580 (6)
H8	0.2005 (11)	0.621 (5)	0.9241 (15)	0.080 (8)*
C9	0.07134 (10)	0.5057 (5)	0.63645 (11)	0.0556 (5)

Atomic displacement parameters (\AA^2)

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

S1	0.0632 (4)	0.0943 (5)	0.0452 (3)	-0.0096 (3)	0.0301 (3)	0.0036 (3)
O1	0.0703 (11)	0.0827 (12)	0.0573 (10)	-0.0268 (9)	0.0324 (9)	-0.0116 (9)
O2	0.0630 (11)	0.0814 (13)	0.0698 (12)	0.0040 (10)	0.0393 (10)	-0.0022 (10)
N1	0.0518 (10)	0.0649 (12)	0.0491 (10)	-0.0074 (9)	0.0269 (9)	-0.0074 (8)
N2	0.0588 (11)	0.0704 (12)	0.0439 (10)	-0.0070 (9)	0.0311 (9)	-0.0025 (8)
N3	0.0611 (12)	0.0780 (14)	0.0419 (9)	-0.0147 (10)	0.0287 (9)	-0.0037 (9)
N4	0.0628 (12)	0.0832 (15)	0.0449 (11)	-0.0118 (11)	0.0299 (10)	-0.0060 (10)
C1	0.0567 (13)	0.0777 (17)	0.0544 (14)	-0.0031 (12)	0.0313 (12)	0.0037 (12)
C2	0.0633 (15)	0.0771 (18)	0.0776 (18)	-0.0037 (14)	0.0436 (15)	0.0078 (14)
C3	0.0841 (19)	0.0761 (18)	0.0785 (19)	0.0119 (15)	0.0603 (17)	0.0164 (14)
C4	0.093 (2)	0.0816 (18)	0.0567 (15)	0.0102 (16)	0.0496 (15)	0.0081 (14)
C5	0.0737 (16)	0.0693 (15)	0.0500 (13)	-0.0021 (13)	0.0381 (12)	-0.0023 (11)
C6	0.0534 (12)	0.0566 (12)	0.0491 (11)	0.0061 (10)	0.0325 (10)	0.0012 (9)
C7	0.0503 (11)	0.0596 (13)	0.0429 (11)	0.0026 (10)	0.0272 (9)	-0.0009 (9)
C8	0.0563 (13)	0.0668 (14)	0.0435 (11)	-0.0018 (11)	0.0263 (10)	-0.0028 (10)
C9	0.0549 (12)	0.0657 (14)	0.0438 (11)	-0.0008 (11)	0.0286 (10)	-0.0044 (10)

Geometric parameters (Å, °)

S1—C9	1.681 (2)	C2—H2	0.93 (3)
O1—H1A	0.903 (18)	C3—C2	1.378 (4)
O2—H21	0.940 (17)	C3—C4	1.368 (4)
O2—H22	0.907 (18)	C3—H3	0.93 (3)
N1—O1	1.385 (2)	C4—H4	0.92 (3)
N1—C8	1.264 (3)	C5—C4	1.381 (4)
N2—N3	1.362 (3)	C5—H5	0.95 (3)
N2—C7	1.297 (3)	C6—C1	1.389 (3)
N3—H3A	0.923 (17)	C6—C5	1.386 (3)
N4—C9	1.320 (3)	C6—C7	1.483 (3)
N4—H41	0.915 (17)	C7—C8	1.469 (3)
N4—H42	0.930 (17)	C8—H8	0.97 (3)
C1—C2	1.374 (3)	C9—N3	1.355 (3)
C1—H1	0.93 (2)		
N1—O1—H1A	104.6 (19)	C3—C4—C5	121.2 (3)
H21—O2—H22	106 (3)	C3—C4—H4	116.6 (19)
C8—N1—O1	112.88 (19)	C5—C4—H4	122.2 (19)
C7—N2—N3	118.58 (19)	C4—C5—C6	120.9 (3)
C9—N3—N2	120.1 (2)	C4—C5—H5	118.5 (18)
C9—N3—H3A	117.9 (16)	C6—C5—H5	120.6 (18)
N2—N3—H3A	122.0 (17)	C1—C6—C7	119.6 (2)
C9—N4—H41	120.3 (17)	C5—C6—C1	117.3 (2)
C9—N4—H42	117.9 (17)	C5—C6—C7	123.0 (2)
H41—N4—H42	121 (2)	N2—C7—C8	125.4 (2)
C2—C1—C6	121.2 (2)	N2—C7—C6	116.00 (19)
C2—C1—H1	118.9 (16)	C8—C7—C6	118.60 (18)
C6—C1—H1	119.7 (16)	N1—C8—C7	122.4 (2)
C1—C2—C3	120.9 (3)	N1—C8—H8	122.8 (16)
C1—C2—H2	118.0 (16)	C7—C8—H8	114.7 (16)
C3—C2—H2	121.1 (16)	N4—C9—N3	117.2 (2)

supplementary materials

C4—C3—C2	118.4 (3)	N4—C9—S1	124.28 (17)
C4—C3—H3	120.7 (18)	N3—C9—S1	118.46 (18)
C2—C3—H3	120.8 (18)		
O1—N1—C8—C7	177.1 (2)	C1—C6—C5—C4	-0.6 (4)
C7—N2—N3—C9	-175.9 (2)	C7—C6—C5—C4	179.8 (2)
N3—N2—C7—C8	2.8 (3)	C5—C6—C7—N2	177.2 (2)
N3—N2—C7—C6	-179.56 (19)	C1—C6—C7—N2	-2.4 (3)
C6—C1—C2—C3	-0.1 (4)	C5—C6—C7—C8	-5.0 (3)
C4—C3—C2—C1	-0.8 (4)	C1—C6—C7—C8	175.4 (2)
C2—C3—C4—C5	1.0 (4)	N2—C7—C8—N1	-7.6 (4)
C6—C5—C4—C3	-0.3 (4)	C6—C7—C8—N1	174.8 (2)
C5—C6—C1—C2	0.8 (4)	S1—C9—N3—N2	-178.23 (16)
C7—C6—C1—C2	-179.6 (2)	N4—C9—N3—N2	3.0 (3)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots O2 ⁱ	0.90 (3)	1.83 (3)	2.728 (3)	174 (3)
O2—H21 \cdots S1	0.94 (3)	2.32 (3)	3.250 (3)	171 (3)
O2—H22 \cdots O2 ⁱ	0.91 (3)	1.98 (3)	2.886 (3)	172 (4)
N3—H3A \cdots N1	0.92 (3)	1.91 (2)	2.604 (3)	130 (2)
N4—H41 \cdots S1 ⁱⁱ	0.92 (2)	2.53 (2)	3.434 (2)	169 (3)
N4—H42 \cdots N2	0.93 (3)	2.24 (3)	2.643 (3)	105 (2)

Symmetry codes: (i) $-x+1/2, y+1/2, -z+3/2$; (ii) $-x, -y+1, -z+1$.

Table 2

Table 2. Comparison of the bond lengths and angles (\AA , $^\circ$) in the oxime moieties of the title compound, (I), with the corresponding values in the related compounds (II)–(VII)

Bond/Angle	(I)	(II)	(III)	(IV)	(V)	(VI)	(VII)
N1-O1	1.385 (2)	1.403 (2)	1.423 (3)	1.417 (1)	1.429 (4)	1.424 (2)	1.416 (3)
		1.396 (2)	1.396 (3)				1.397 (3)
N1-C8	1.264 (3)	1.281 (2)	1.290 (3)	1.290 (1)	1.241 (6)	1.289 (2)	1.282 (3)
		1.281 (2)	1.282 (3)				1.289 (3)
C7-C8	1.469 (3)	1.477 (3)	1.489 (3)	1.510 (1)	1.551 (7)	1.513 (2)	1.501 (4)
		1.473 (3)					1.502 (4)
C7-C8-N1	122.4 (2)	115.2 (2)	116.6 (2)	114.3 (1)	118.3 (5)	113.2 (1)	114.4 (2)
		115.0 (2)	115.0 (2)				113.4 (2)
C8-N1-O1	112.9 (2)	112.4 (1)	109.4 (2)	110.7 (1)	112.2 (4)	110.6 (1)	110.7 (2)
		112.2 (1)	111.5 (2)				111.1 (2)

Notes: (II): 2,3-dimethylquinoxaline-dimethylglyoxime (1/1) (Hökelek, Batı *et al.*, 2001), (III): 1-(2,6-dimethylphenylamino)propane-1,2-dione dioxime (Hökelek, Zülfikaroğlu & Batı, 2001), (IV): *N*-hydroxy-2-oxo-2,2'-diphenylacetamide (Büyükgüngör *et al.*, 2003), (V): *N*-(3,4-dichlorophenyl)-*N*'-hydroxy-2-oxo-2-phenylacetamide (Hökelek *et al.*, 2004), (VI): *N*-hydroxy-*N*'-(1-naphthyl)-2-phenylacetamide-2-one (Hökelek *et al.*, 2004a), (VII): *N*-(3-chloro-4-methylphenyl)-*N*'-hydroxy-2-oxo-2-phenylacetamide-2,3-dimethylquinoxaline-dimethylglyoxime (1/1) (Hökelek *et al.*, 2004b).

Fig. 1

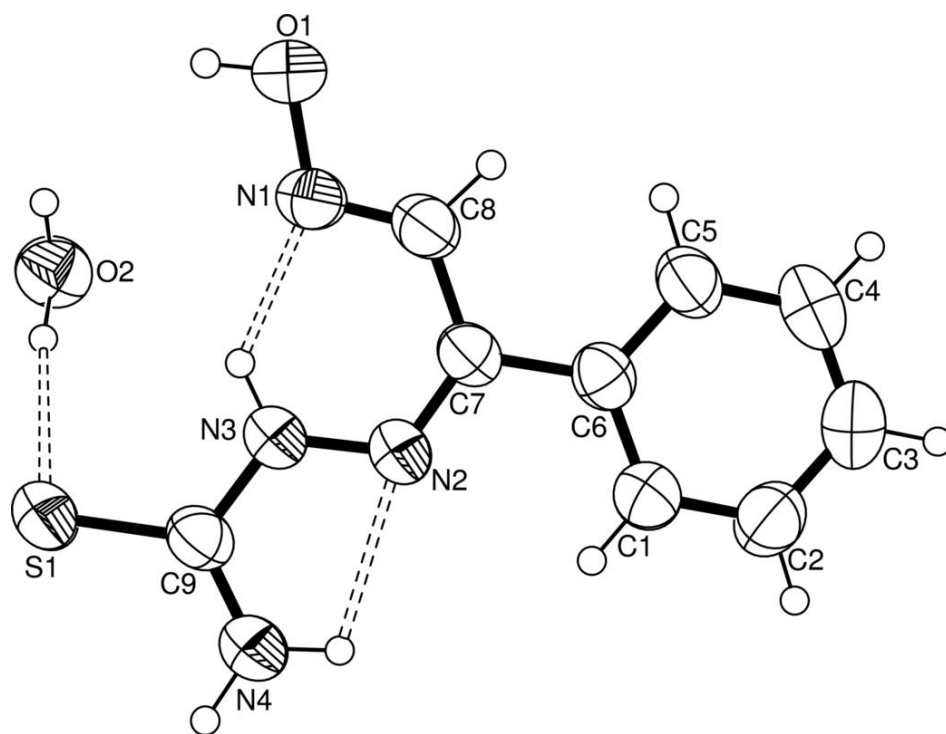


Fig. 2

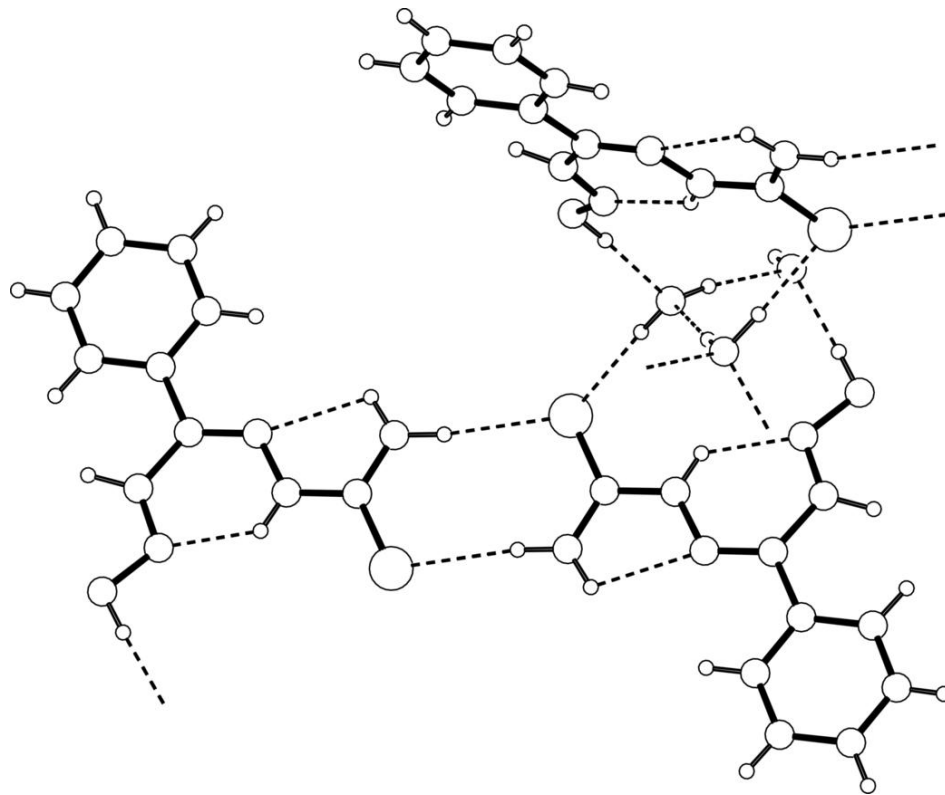


Fig. 3

