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[(2,4-Dinitrophenyl)hydrazono](phenyl)-acetaldehyde oxime monohydrate

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Key indicators

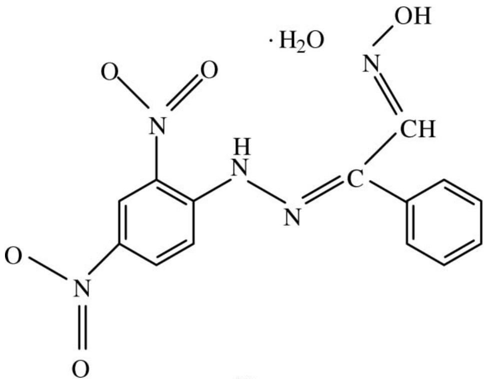
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
R factor = 0.070
wR factor = 0.134
Data-to-parameter ratio = 11.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{14}H_{11}N_5O_5 \cdot H_2O$, the organic and water molecules are linked by C—H···O and O—H···O hydrogen bonds into a three-dimensional framework. A centrosymmetric $R_4^4(12)$ ring is centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$); these rings are linked to each other by H atoms of the oxime groups. Besides these interactions, there are also intramolecular N—H···O and N—H···N hydrogen bonds.

Comment

Oximes and hydrazones are interesting because of their wide application in medicine, industry and analytical chemistry. These compounds are used as analytical reagents for the detection and determination of some metal ions (Gup & Giziroğlu, 2006; Tezcan *et al.*, 2004). Hydrazones have been utilized for the determination of carbonyl compounds (Tezcan *et al.*, 2004; Townshend & Wheatly, 1998). Hydrazones and hydrazone oximes exhibit biological activity. Hydrazone derivatives have been synthesized in order to investigate the relationship between structure and biological activity (Tsapkov, 2002; Ghosh & Bandyopadhyay, 1985; Shan *et al.*, 2002). In this study, we present the crystal structure of the title compound, (I), which is a product of the condensation reaction of 2,4-dinitrophenylhydrazine with isonitrosoacetophenone.



(I)

The molecular structure of (I) is shown in Fig. 1. Compound (I) contains three units, namely dinitrophenyl, monoxime and hydrazone. The dinitrophenyl and hydrazone units are approximately planar. The dihedral angle between the oxime plane and the plane of the dinitrophenylhydrazone is 12.4 (4)°. The dihedral angle between the phenyl ring and the oxime plane is 45.9 (2)°. The phenyl and dinitrophenyl groups are linked through the hydrazone and oxime units. The bond lengths and angles of the oxime and hydrazone are given in Table 1. Both the oxime and hydrazone units in (I) have an *E*

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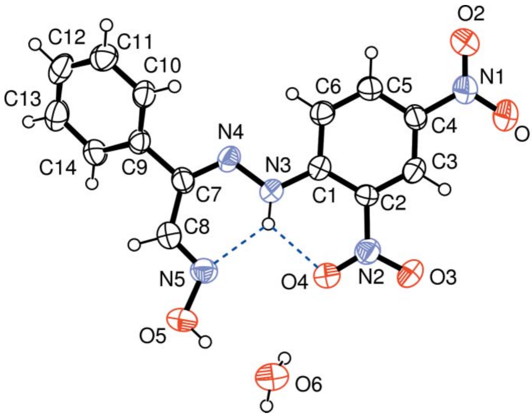

Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

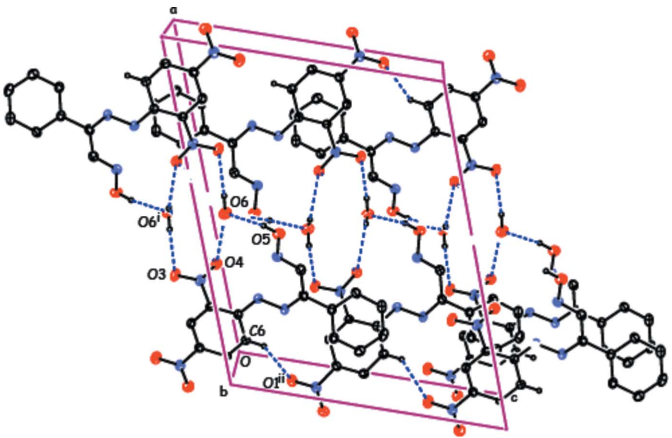

Figure 2
The crystal structure of (I). H atoms have been omitted except for those involved in hydrogen bonds (dashed lines). [Symmetry codes: (i) $1 - x, 2 - y, -z$; (ii) $x, \frac{3}{2} - y, \frac{1}{2} + z$.]

configuration [O5—N5═C8—C7 = 174.8 (4)° and N3—N4═C7—C9 = 177.9 (4)°]. The N3—N4 bond distance of 1.379 (5) Å is appreciably shorter than a typical N—N single bond, such as that found in free 2,4-dinitrophenylhydrazine [1.405 (6)Å; Okabe *et al.*, 1993]; this suggests the existence of a delocalized double bond.

The C1—C2 and C1—C6 bonds close to the imine group are appreciably longer than the other C—C bonds in the same benzene ring (Table 1). This agrees with the situation found in 3-chloroacetophenone 2,4-dinitrophenylhydrazone (Fan *et al.*, 2004). The N4═C7 and N5═C8 distances of 1.309 (5) and 1.281 (6) Å, respectively, are typical of a double bond. The C═N and N—O distances of the free oximes are close to those commonly found in oxime derivatives [C═N = 1.292 (13) Å and N—O = 1.396 (3) Å; Saraçoğlu *et al.*, 2004).

The organic molecules of (I) are linked to the water molecules by C—H···O and O—H···O hydrogen bonds (Table 2), forming a three-dimensional framework. Atom C6 at (x, y, z) acts as a donor to atom O1 at $(x, \frac{3}{2} - y, \frac{1}{2} + z)$, so producing a *C*(6) chain (Bernstein *et al.*, 1995) running parallel to the [001] direction and generated by a *c*-glide plane at $y = \frac{1}{4}$. In the O—H···O hydrogen bonds, atoms O3 and O4 at (x, y, z) act as acceptors to O6(H7*W*) at $(1 - x, 2 - y, -z)$ and O6(H6*W*) at (x, y, z), respectively. Similarly, atoms O3 and O4 at $(1 - x, 2 - y, -z)$ act as acceptors to atoms O6(H7*W*) at (x, y, z) and O6(H6*W*) at $(1 - x, 2 - y, -z)$, generating a centrosymmetric $R_4^4(12)$ ring centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. These rings are linked to each other by H atoms of the oxime units. The $R_4^4(12)$ rings formed by hydrogen bonds are centred at $[\frac{1}{2}, (1 - n)/2, (1 + n)/2]$ (*n* is zero or an integer). Besides these interactions, there are intramolecular N—H···O and N—H···N hydrogen bonds.

Experimental

Isonitrosoacetephenone (1 mmol, 0.20 g) dissolved in hot anhydrous ethanol (30 ml) was added dropwise to solution of 2,4-dinitrophenylhydrazine (1 mmol, 0.15 g) in hot anhydrous ethanol (30 ml). The reaction mixture was refluxed for 11 h. The resulting orange product was filtered off and washed with methanol. The solid product was recrystallized from acetonitrile to give crystals of the title compound (yield 60%; m.p. 513–517 K).

Crystal data

$C_{14}H_{11}N_5O_5 \cdot H_2O$
$M_r = 347.29$
Monoclinic, $P2_1/c$
$a = 17.2299$ (19) Å
$b = 7.0635$ (12) Å
$c = 13.5311$ (15) Å
$\beta = 109.629$ (8)°
$V = 1551.1$ (4) Å^3
$Z = 4$
$D_x = 1.487$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.12$ mm^{-1}
$T = 296$ K
Plate, red
0.60 × 0.30 × 0.02 mm

Data collection

Stoe IPDS 2 area-detector diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.958$, $T_{max} = 0.998$
11683 measured reflections
2689 independent reflections
1189 reflections with $I > 2\sigma(I)$
$R_{int} = 0.178$
$\theta_{max} = 25.0$°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.134$
$S = 0.97$
2689 reflections
232 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O5—N5	1.391 (4)	N4—C7	1.309 (5)
N1—C4	1.467 (6)	N5—C8	1.281 (6)
N2—C2	1.450 (5)	C1—C2	1.412 (6)
N3—C1	1.375 (5)	C1—C6	1.419 (6)
N3—N4	1.379 (5)	C7—C8	1.469 (6)
C1—N3—N4	117.4 (4)	N3—C1—C2	123.9 (4)
C7—N4—N3	118.2 (4)	N3—C1—C6	119.0 (4)
C8—N5—O5	111.3 (4)	N5—C8—C7	121.4 (5)
C1—N3—N4—C7	−174.4 (4)	O5—N5—C8—C7	174.8 (4)
N3—N4—C7—C9	179.7 (4)	C8—C7—C9—C14	−41.2 (7)

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Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O4	0.86	2.03	2.627 (5)	126
N3—H3*A*···N5	0.86	2.07	2.709 (5)	131
O5—H5*A*···O6	0.82	1.96	2.760 (5)	163
O6—H6*W*···O4	0.82 (3)	2.46 (6)	2.985 (5)	124 (4)
O6—H7*W*···O3^{i}	0.84 (3)	2.22 (3)	3.055 (5)	170
C6—H6···O1^{ii}	0.93	2.51	3.230 (7)	135

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

The high value of $R_{\rm int}$ indicates that the overall quality of the data may be poor due to the poor quality of the crystal. The water H atoms were located in difference maps and refined isotropically with bond restraints of O—H = 0.84 (3) Å. C-, N- and the remaining O-bound H atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å, N—H = 0.86 Å and O—H = 0.82 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N) or 1.5$U_{\rm eq}$(O) for the hydroxy group.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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