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(2*E*)-2-(2-Phenylhydrazin-1-ylidene)-propanoic acid

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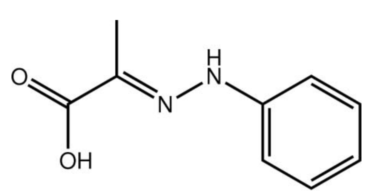
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.114; data-to-parameter ratio = 15.1.

The 13 non-H atoms comprising the title compound, $C_9H_{10}N_2O_2$, are close to planar (r.m.s. deviation = 0.140 Å), with maximum deviations of 0.292 (1) and 0.210 (1) Å to either side of the least-squares plane exhibited by the hydroxy and carbonyl O atoms, respectively. The observed conformation is stabilized by an intramolecular O—H···N hydrogen bond. The conformation about the N=C double bond [1.2909 (16) Å] is *E*. The hydroxy OH group also forms an intermolecular hydrogen bond to a carbonyl O atom, and the amine H atom similarly forms an N—H···O hydrogen bond to a second carbonyl O atom. The result is the formation of a double layer with a flat topology. Layers stack along the *a*-axis direction connected by C—H···π interactions.

Related literature

For background and recent studies on the biological activity of tin/organotin compounds, see: Gielen & Tiekink (2005); Affan *et al.* (2009).



Experimental

Crystal data

$C_9H_{10}N_2O_2$
M_r = 178.19
Monoclinic, $P2_1/c$
a = 7.3239 (3) Å
b = 12.0837 (7) Å
c = 9.6836 (4) Å
β = 99.119 (4)°
V = 846.17 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.20 × 0.15 × 0.10 mm

Data collection

Agilent Supernova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.734, T_{max} = 1.000
7879 measured reflections
1920 independent reflections
1544 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.114
S = 1.03
1920 reflections
127 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C4–C9 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.86 (2)	2.12 (2)	2.6169 (16)	115.9 (16)
O1—H1···O2^{i}	0.86 (2)	2.18 (2)	2.9039 (14)	141.5 (19)
N2—H2···O2^{ii}	0.916 (18)	2.199 (19)	3.0579 (15)	155.9 (15)
C3—H3c···*Cg*1^{iii}	0.98	2.92	3.5830 (16)	126

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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 *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5024).

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(2*E*)-2-(2-Phenylhydrazin-1-ylidene)propanoic acid

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S1. Comment

The title compound, (I), was prepared as a potential ligand for tin (Affan *et al.*, 2009), motivated by the wide range of biological activities displayed by organotin compounds (Gielen & Tiekink, 2005). The r.m.s. for the 13 non-hydrogen atoms comprising (I), Fig. 1, is 0.140 Å. The maximum deviations are found for the carboxylic acid-O atoms with the O1 atom being 0.292 (1) Å out of the least-squares plane and the O2 lying 0.210 (1) Å to the other side. The planarity in the molecule is readily explained in terms of an intramolecular O—H···N hydrogen bond as the hydroxy H is directed toward the centre of the molecule, Table 1. The conformation about the N1═C2 double bond [1.2909 (16) Å] is *E*. In the crystal packing, the carbonyl-O2 atom accepts hydrogen bonds from both the hydroxy-O1—H and amine-H atoms, derived from different molecules, Table 1. The result is a supramolecular double layer as illustrated in Fig. 2. Layers stack along the *a* direction and are connected by C—H···π interactions, Fig. 3 and Table 1.

S2. Experimental

Pyruvic acid (0.440 g, 5 mmol) was dissolved in 10 ml absolute ethanol with constant stirring. An ethanolic solution of phenylhydrazine (0.540 g, 5 mmol) was then added to the solution drop-wise. The resulting reaction mixture was refluxed for 5 h. On cooling the solution to room temperature, a light-orange powder separated, which was filtered and washed with ethanol. The powder was recrystallized from ethanol and dried *in vacuo* over silica gel. (*M*.pt. 460–462 K. Yield 0.724 g (73.8%). Anal. Calc. for $C_9H_{10}N_2O_2$: C, 60.66; H, 5.65; N, 15.72%. Found: C, 60.61; H, 5.59; N, 15.68%. FT—IR (KBr, cm^{-1}) ν_{max}: 3333 (m, OH), 3285 (s, NH), 1709 (m, C=O), 1595 (w, C=N), 991 (m, N—N).

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H = 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2–1.5U_{eq}(C). The O—H and N—H hydrogen atoms were freely refined; see Table 1 for bond distances.

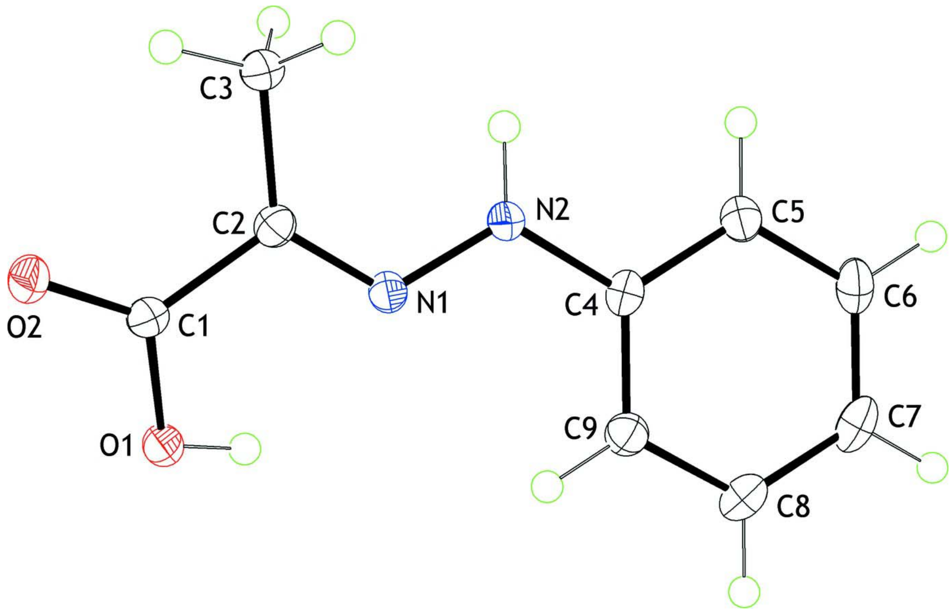

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

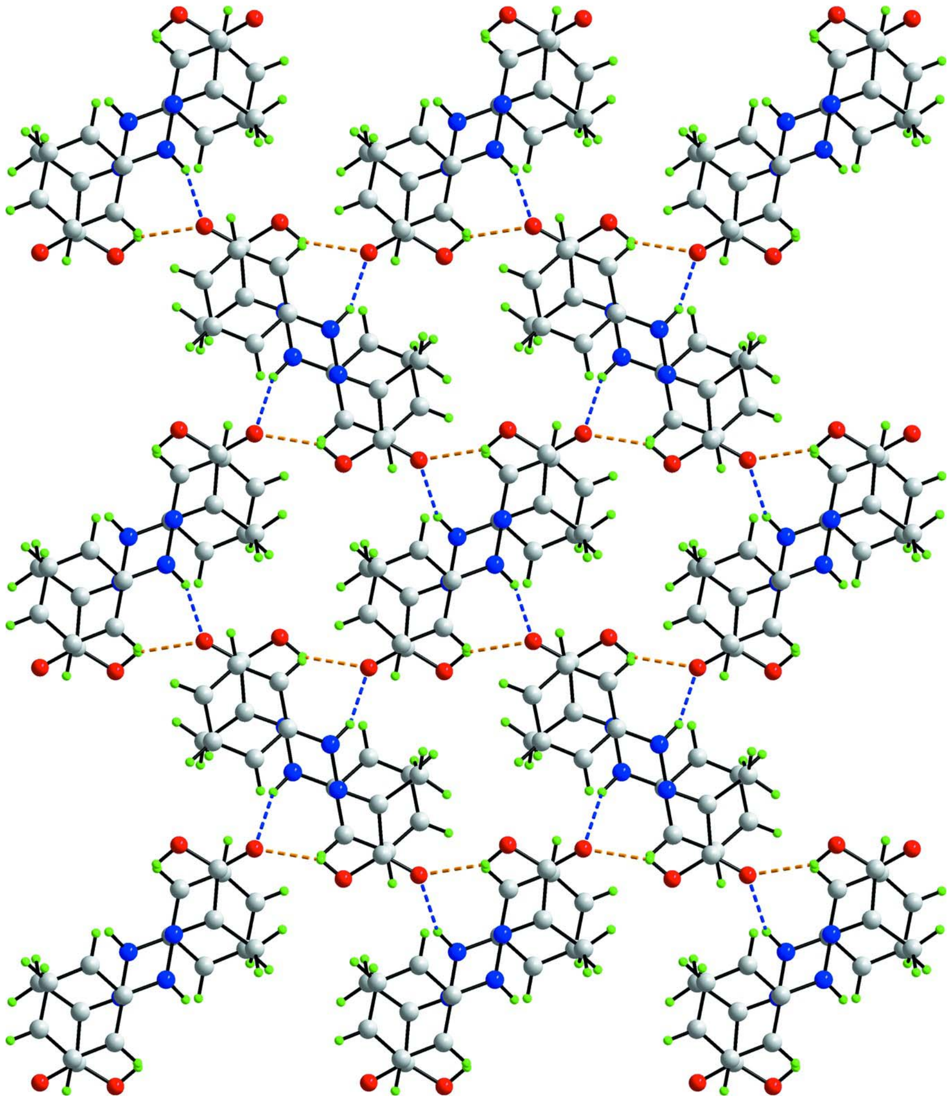

Figure 2

A view in projection down the *a* axis of the supramolecular double layer in (I). The O—H···O and N—H···O hydrogen bonds are shown as orange and blue dashed lines, respectively.

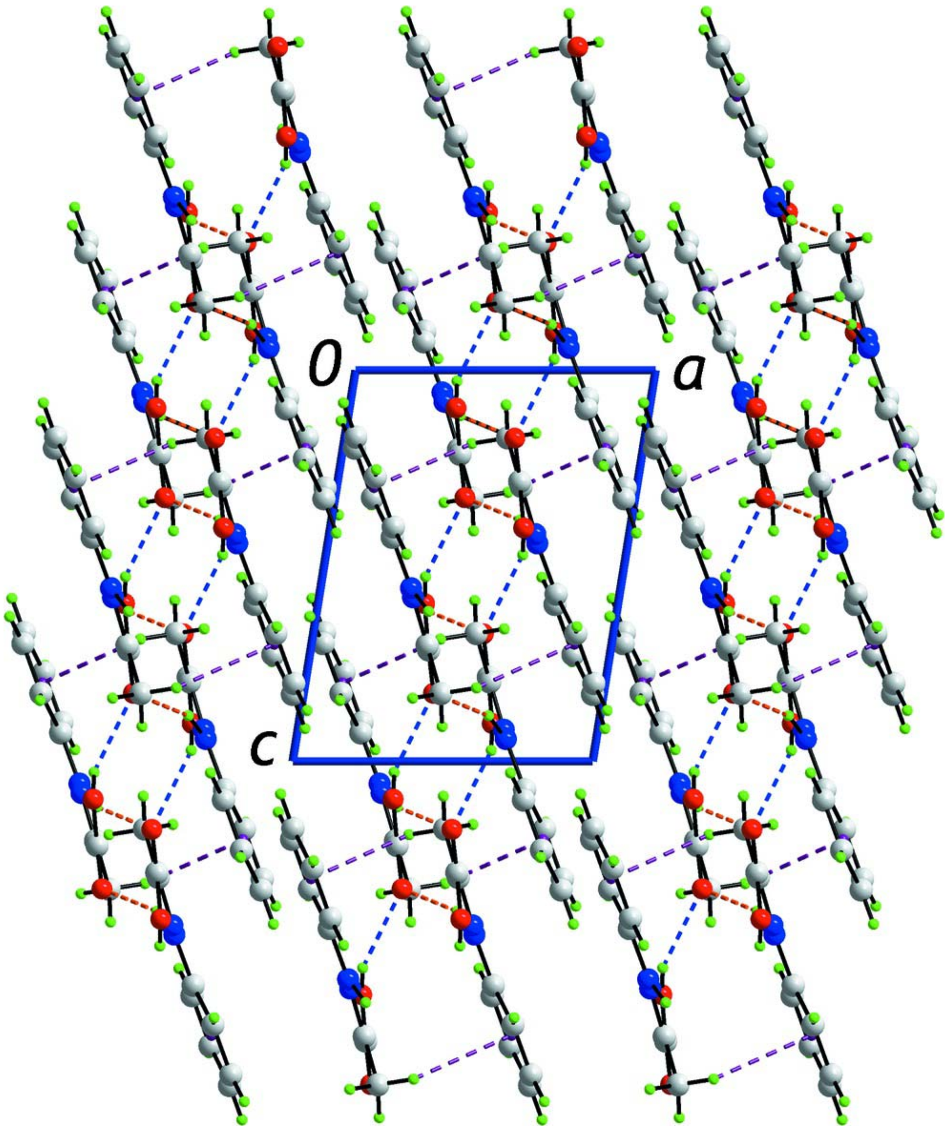

Figure 3

A view in projection down the *b* axis of the crystal packing in (I) showing the connection between layers *via* C—H···π interactions. The O—H···O and N—H···S hydrogen bonds are shown as orange and blue dashed lines, respectively, and the C—H···π contacts are shown as purple dashed lines.

(2*E*)-2-(2-Phenylhydrazin-1-ylidene)propanoic acid

Crystal data

$C_9H_{10}N_2O_2$
M_r = 178.19
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.3239 (3) Å
b = 12.0837 (7) Å
c = 9.6836 (4) Å
β = 99.119 (4)°
V = 846.17 (7) Å^3
Z = 4
F(000) = 376
D_x = 1.399 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2940 reflections
θ = 2.7–29.2°
μ = 0.10 mm^{-1}

T = 100 K
Block, yellow
0.20 × 0.15 × 0.10 mm

Data collection

Agilent Supernova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.734, T_{max} = 1.000
7879 measured reflections
1920 independent reflections
1544 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°, θ_{min} = 2.7°
$h = -9\rightarrow9$
$k = -11\rightarrow15$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.114$
S = 1.03
1920 reflections
127 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.2722P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.64494 (15)	0.79085 (9)	0.40334 (10)	0.0218 (3)
O2	0.56711 (14)	0.78175 (8)	0.17390 (10)	0.0207 (3)
N2	0.69829 (16)	0.46664 (10)	0.44283 (12)	0.0175 (3)
N1	0.68674 (15)	0.57591 (10)	0.41916 (11)	0.0158 (3)
C1	0.60689 (18)	0.73282 (12)	0.28486 (13)	0.0168 (3)
C2	0.61419 (18)	0.61094 (12)	0.29678 (13)	0.0164 (3)
C3	0.5429 (2)	0.54090 (12)	0.17314 (14)	0.0196 (3)
H3A	0.6342	0.4838	0.1615	0.029*
H3B	0.5208	0.5872	0.0891	0.029*
H3C	0.4269	0.5057	0.1876	0.029*
C4	0.78824 (18)	0.42878 (12)	0.57266 (14)	0.0163 (3)
C5	0.7901 (2)	0.31539 (12)	0.59907 (15)	0.0205 (3)
H5	0.7321	0.2656	0.5297	0.025*

C6	0.87695 (19)	0.27559 (13)	0.72715 (16)	0.0242 (4)
H6	0.8773	0.1984	0.7455	0.029*
C7	0.9634 (2)	0.34750 (14)	0.82876 (15)	0.0248 (4)
H7	1.0229	0.3200	0.9163	0.030*
C8	0.96165 (19)	0.45980 (13)	0.80084 (15)	0.0228 (3)
H8	1.0207	0.5094	0.8700	0.027*
C9	0.87518 (19)	0.50122 (13)	0.67369 (15)	0.0193 (3)
H9	0.8753	0.5785	0.6557	0.023*
H1	0.656 (3)	0.7447 (18)	0.472 (2)	0.044 (6)*
H2	0.628 (2)	0.4184 (16)	0.3834 (19)	0.032 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0340 (6)	0.0160 (6)	0.0142 (5)	0.0001 (4)	−0.0003 (4)	−0.0001 (4)
O2	0.0272 (5)	0.0187 (6)	0.0157 (5)	0.0014 (4)	0.0018 (4)	0.0027 (4)
N2	0.0218 (6)	0.0138 (6)	0.0157 (6)	0.0001 (5)	−0.0007 (5)	0.0008 (5)
N1	0.0166 (6)	0.0149 (6)	0.0158 (6)	0.0009 (4)	0.0026 (4)	0.0007 (4)
C1	0.0175 (7)	0.0172 (8)	0.0153 (6)	−0.0006 (5)	0.0014 (5)	−0.0006 (5)
C2	0.0159 (6)	0.0180 (8)	0.0153 (6)	−0.0003 (5)	0.0027 (5)	0.0006 (5)
C3	0.0240 (7)	0.0175 (8)	0.0162 (6)	−0.0013 (6)	0.0002 (6)	−0.0008 (5)
C4	0.0146 (6)	0.0195 (8)	0.0153 (6)	0.0022 (5)	0.0043 (5)	0.0032 (5)
C5	0.0209 (7)	0.0181 (8)	0.0223 (7)	0.0016 (6)	0.0024 (6)	0.0010 (6)
C6	0.0226 (7)	0.0211 (8)	0.0288 (8)	0.0042 (6)	0.0034 (6)	0.0088 (6)
C7	0.0195 (7)	0.0345 (9)	0.0194 (7)	0.0039 (6)	0.0005 (6)	0.0093 (6)
C8	0.0189 (7)	0.0302 (9)	0.0182 (7)	−0.0008 (6)	−0.0003 (6)	0.0014 (6)
C9	0.0188 (7)	0.0195 (8)	0.0196 (7)	−0.0013 (5)	0.0025 (6)	0.0010 (6)

Geometric parameters (Å, º)

O1—C1	1.3358 (16)	C4—C9	1.390 (2)
O1—H1	0.86 (2)	C4—C5	1.393 (2)
O2—C1	1.2205 (16)	C5—C6	1.3871 (19)
N2—N1	1.3405 (16)	C5—H5	0.9500
N2—C4	1.4005 (17)	C6—C7	1.388 (2)
N2—H2	0.916 (18)	C6—H6	0.9500
N1—C2	1.2909 (16)	C7—C8	1.383 (2)
C1—C2	1.478 (2)	C7—H7	0.9500
C2—C3	1.4913 (18)	C8—C9	1.3858 (19)
C3—H3A	0.9800	C8—H8	0.9500
C3—H3B	0.9800	C9—H9	0.9500
C3—H3C	0.9800		
C1—O1—H1	107.9 (14)	C9—C4—N2	121.60 (13)
N1—N2—C4	118.90 (11)	C5—C4—N2	118.39 (12)
N1—N2—H2	120.4 (11)	C6—C5—C4	119.65 (14)
C4—N2—H2	119.5 (11)	C6—C5—H5	120.2
C2—N1—N2	119.05 (12)	C4—C5—H5	120.2

O2—C1—O1	119.34 (13)	C5—C6—C7	120.65 (14)
O2—C1—C2	123.52 (12)	C5—C6—H6	119.7
O1—C1—C2	117.13 (11)	C7—C6—H6	119.7
N1—C2—C1	113.77 (12)	C8—C7—C6	119.11 (13)
N1—C2—C3	126.28 (13)	C8—C7—H7	120.4
C1—C2—C3	119.95 (11)	C6—C7—H7	120.4
C2—C3—H3A	109.5	C7—C8—C9	121.11 (14)
C2—C3—H3B	109.5	C7—C8—H8	119.4
H3A—C3—H3B	109.5	C9—C8—H8	119.4
C2—C3—H3C	109.5	C8—C9—C4	119.46 (14)
H3A—C3—H3C	109.5	C8—C9—H9	120.3
H3B—C3—H3C	109.5	C4—C9—H9	120.3
C9—C4—C5	120.01 (13)		

C4—N2—N1—C2	175.73 (12)	C9—C4—C5—C6	0.8 (2)
N2—N1—C2—C1	179.08 (12)	N2—C4—C5—C6	−179.52 (13)
N2—N1—C2—C3	−1.5 (2)	C4—C5—C6—C7	−0.6 (2)
O2—C1—C2—N1	169.64 (13)	C5—C6—C7—C8	0.1 (2)
O1—C1—C2—N1	−10.90 (18)	C6—C7—C8—C9	0.1 (2)
O2—C1—C2—C3	−9.8 (2)	C7—C8—C9—C4	0.2 (2)
O1—C1—C2—C3	169.66 (12)	C5—C4—C9—C8	−0.6 (2)
N1—N2—C4—C9	−4.0 (2)	N2—C4—C9—C8	179.71 (13)
N1—N2—C4—C5	176.34 (12)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C4–C9 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.86 (2)	2.12 (2)	2.6169 (16)	115.9 (16)
O1—H1···O2^{i}	0.86 (2)	2.18 (2)	2.9039 (14)	141.5 (19)
N2—H2···O2^{ii}	0.916 (18)	2.199 (19)	3.0579 (15)	155.9 (15)
C3—H3c···*Cg*1^{iii}	0.98	2.92	3.5830 (16)	126

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) −*x*+1, −*y*+1, −*z*+1.