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(2*E*)-2-Hydroxyimino-*N*′-[(*E*)-2-pyridylmethylene]propanohydrazide

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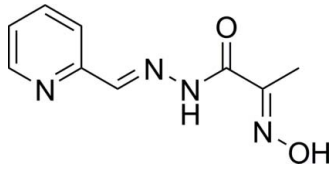
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.106; data-to-parameter ratio = 15.2.

In the title compound, $C_9H_{10}N_4O_2$, the pyridine ring is twisted by 16.5 (1)° from the mean plane defined by the remaining non-H atoms. An intramolecular N—H···N interaction is present. In the crystal, intermolecular O—H···N and N—H···O hydrogen bonds link molecules into layers parallel to the *bc* plane. The crystal packing exhibits π–π interactions indicated by the short distance of 3.649 (1) Å between the centroids of the pyridine rings of neighbouring molecules.

Related literature

For the crystal structures of related oxime derivatives, see: Mokhir *et al.* (2002); Moroz *et al.* (2009). For 2-hydroxyiminopropanamide and amide derivatives of 2-hydroxyiminopropanoic acid, see: Onindo *et al.* (1995); Duda *et al.* (1997); Sliva *et al.* (1997*a*). For the preparation and characterization of 3*d*-metal complexes with the structural analog of the title compound, see: Moroz *et al.* (2008*a*,*b*). For the synthesis of 2-(hydroxyimino)propanehydrazide, see Fritsky *et al.* (1998).



Experimental

Crystal data

$C_9H_{10}N_4O_2$
M_r = 206.21
Monoclinic, $P2_1/c$
a = 10.274 (4) Å
b = 9.717 (4) Å
c = 10.136 (4) Å
β = 109.28 (4)°
V = 955.2 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.4 × 0.4 × 0.3 mm

Data collection

Kuma KM-4-CCD diffractometer
Absorption correction: none
10648 measured reflections
2680 independent reflections
2449 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.106
S = 1.08
2680 reflections
176 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H21···N1^{i}	0.881 (17)	1.897 (18)	2.7667 (17)	168.6 (15)
N3—H31···O1^{ii}	0.879 (16)	2.217 (16)	2.9656 (15)	142.9 (14)
N3—H31···N4	0.879 (16)	2.240 (16)	2.6059 (15)	104.7 (12)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; 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); 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: CV2608).

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(2*E*)-2-Hydroxyimino-*N*′-[(*E*)-2-pyridylmethylene]propanohydrazide

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S1. Comment

As a part of our research study we present the structure of the title compound, **1** (Fig. 1), which comprises several donor groups: oxime, hydrazone, azomethine, and pyridine. It has been shown previously that structurally similiar strand ligand forms mono- and tetranuclear grid-like assemblies with 3d-metal ions (Moroz *et al.*, 2008*a*,*b*).

The C—N and N—O bond lengths in the oxime group, *i.e.* 1.285 (1) and 1.387 (1) Å, respectively, adopt typical values (Mokhir *et al.*, 2002; Moroz *et al.*, 2009). The oxime group is in a *trans* position with respect to the amide group, in accordance with the structures of 2-hydroxyiminopropanamide and other amide derivatives of 2-hydroxyiminopropanoic acid (Onindo *et al.*, 1995; Duda *et al.*, 1997; Sliva *et al.*, 1997*a*). This conformation is stabilized by an N3—H···N4 intramolecular interaction (Table 1). The $CH_3C(=NOH)C(O)NH$ fragment deviates from planarity because of a twist between the oxime and amide groups about the C7—C8 bond; the O1—C7—C8—N4 torsion angle is 173.3 (1)°. The C—N bond length in the azomethine group is 1.283 (1) Å, and the N2—C6—C5 angle has almost ideal value 120.5 (1)°. The pyridine nitrogen atom is situated in an *anti*- position with respect to the azomethine group.

In the crystal packing, molecules are connected by O2—H···N1 and N3—H···O1 hydrogen bonds (Table 1), where the oximic oxygen and hydrazone nitrogen atoms act as donors and the hydrazone oxygen and pyridine nitrogen atoms act as acceptors. Due to the presence of the system of O2—H···N1 and N3—H···O1 hydrogen bonds, layers parallel to *bc* plane are formed. The layers are connected in three-dimensional structure by π-π contacts with centroid-to-centroid distance of 3.649 (2) Å, which arise between the pyridine rings of neighbour molecules.

S2. Experimental

The compound **1** has beeen prepared accourding to following procedure: picolinaldehyde (1.1 ml, 0.012 mol) was added to 20 ml of a stirred warm ethanol/water solution of 2-(hydroxyimino)propanehydrazide (1.17 g, 0.01 mol) prepared according to early reported method (Fritsky *et al.*, 1998). After 20 minutes of stirring at 60°C a yellowish precipitate was formed. It was filtered off, washed with water and acetone and recrystalized from methanol. Yield: 1.65 g, 80%. ^{1}H NMR, 400.13 MHz, (DMSO-d_6): 1.983 (s, 3H, CH_3), 7.393 (dd, 1H, py-5, J = 5.1 Hz, J = 7.2 Hz, py—H), 7.854 (t, 1H, py-4, J = 7.2 Hz, py—H), 7.932 (d, 1H, py-3, J = 7.2 Hz, py—H), 8.494 (s, 1H, CH), 8.594 (d, 1H, py-6, J = 5.1 Hz, py—H), 11.703 (s, 1H, NH), 11.932 (s, 1H, NOH); IR (KBr, cm^{-1}): 1670 $\nu(CO_{amid\ I})$, 1040, 895 $\nu(NO_{oxim})$, 3345 $\nu(NH_{as})$. Anal. calc. for $C_9H_{10}N_4O_2$ C, 52.42; H, 4.89; N, 27.17. Found: C, 52.30; H, 4.98; N, 26.98.

S3. Refinement

All H atoms were located in a difference Fourier map and refined isotropically.

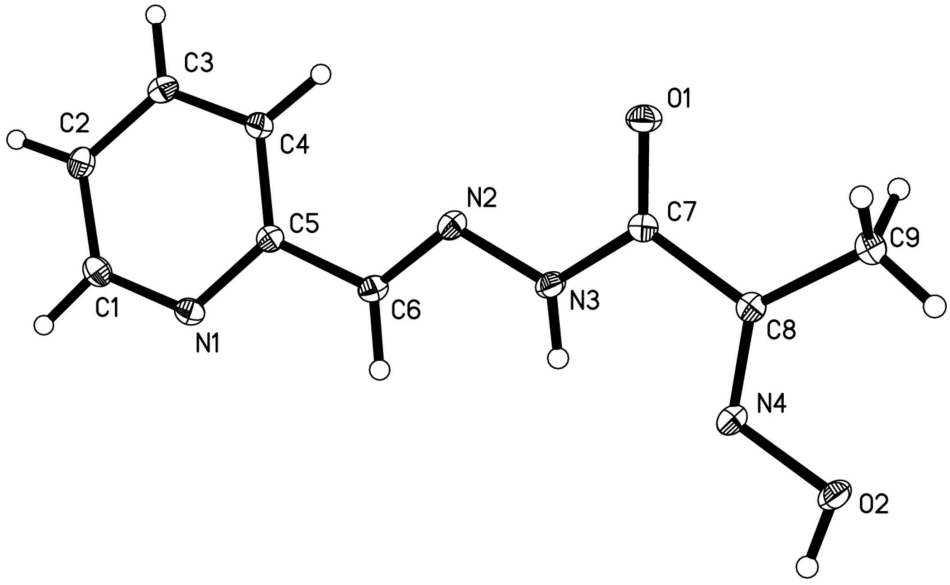

Figure 1

A view of **1**, with displacement ellipsoids shown at the 40% probability level and atom labelling.

(2*E*)-2-Hydroxyimino-*N*′-[(*E*)-2- pyridylmethylene]propanohydrazide

Crystal data

$C_9H_{10}N_4O_2$
$M_r = 206.21$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.274$ (4) Å
$b = 9.717$ (4) Å
$c = 10.136$ (4) Å
$\beta = 109.28$ (4)°
$V = 955.2$ (7) Å^3
$Z = 4$
$F(000) = 432$
$D_x = 1.434$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 11569 reflections
$\theta = 3$–37°
$\mu = 0.11$ mm^{-1}
$T = 100$ K
Block, white
0.4 × 0.4 × 0.3 mm

Data collection

Kuma KM-4-CCD diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 8.3359 pixels mm^{-1}
ω scans
10648 measured reflections
2680 independent reflections
2449 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 30.0°$, $\theta_{min} = 3.2°$
$h = -14 \rightarrow 13$
$k = -13 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.106$
$S = 1.08$
2680 reflections
176 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
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2 + 0.2439P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ 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	0.59592 (7)	0.23238 (7)	0.23853 (7)	0.01751 (17)
O2	0.24735 (8)	0.10483 (8)	−0.16272 (8)	0.01954 (17)
H21	0.2245 (17)	0.1328 (17)	−0.2501 (18)	0.032 (4)*
N1	0.85431 (9)	0.66853 (8)	−0.05564 (8)	0.01561 (18)
N2	0.70574 (8)	0.39826 (8)	0.08234 (8)	0.01477 (17)
N3	0.59208 (9)	0.31588 (9)	0.02612 (9)	0.01608 (18)
H31	0.5529 (16)	0.3104 (16)	−0.0650 (17)	0.030 (4)*
N4	0.36471 (8)	0.18113 (8)	−0.09566 (8)	0.01500 (17)
C1	0.95964 (11)	0.75821 (10)	−0.01987 (11)	0.0176 (2)
H1	0.9570 (16)	0.8267 (16)	−0.0903 (16)	0.029 (4)*
C2	1.06781 (10)	0.75321 (10)	0.10549 (10)	0.0168 (2)
H2	1.1433 (15)	0.8188 (14)	0.1263 (15)	0.023 (3)*
C3	1.06686 (10)	0.64989 (10)	0.20031 (10)	0.0167 (2)
H3	1.1409 (16)	0.6414 (16)	0.2873 (16)	0.026 (4)*
C4	0.95703 (10)	0.55828 (10)	0.16717 (10)	0.01493 (19)
H4	0.9544 (15)	0.4838 (15)	0.2317 (15)	0.024 (3)*
C5	0.85205 (10)	0.57099 (9)	0.03849 (9)	0.01315 (18)
C6	0.73225 (10)	0.47858 (10)	−0.00602 (10)	0.01499 (19)
H6	0.6756 (15)	0.4825 (15)	−0.1032 (15)	0.026 (3)*
C7	0.54344 (10)	0.23721 (9)	0.11115 (10)	0.01334 (18)
C8	0.41629 (10)	0.15545 (9)	0.03573 (10)	0.01366 (19)
C9	0.36358 (11)	0.05663 (11)	0.11943 (11)	0.0188 (2)
H9A	0.2794 (19)	0.0181 (19)	0.0675 (19)	0.044 (5)*
H9B	0.3462 (17)	0.1013 (17)	0.1967 (18)	0.037 (4)*
H9C	0.4325 (17)	−0.0149 (17)	0.1626 (17)	0.035 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0173 (3)	0.0225 (4)	0.0118 (3)	−0.0006 (3)	0.0036 (3)	0.0006 (2)
O2	0.0176 (4)	0.0249 (4)	0.0133 (3)	−0.0076 (3)	0.0014 (3)	−0.0023 (3)
N1	0.0163 (4)	0.0174 (4)	0.0136 (4)	0.0008 (3)	0.0055 (3)	0.0016 (3)

N2	0.0133 (4)	0.0166 (4)	0.0145 (4)	−0.0022 (3)	0.0046 (3)	−0.0024 (3)
N3	0.0160 (4)	0.0202 (4)	0.0109 (4)	−0.0054 (3)	0.0029 (3)	−0.0011 (3)
N4	0.0128 (4)	0.0168 (4)	0.0146 (4)	−0.0022 (3)	0.0036 (3)	−0.0028 (3)
C1	0.0192 (5)	0.0172 (4)	0.0180 (5)	−0.0002 (3)	0.0084 (4)	0.0029 (3)
C2	0.0155 (4)	0.0163 (4)	0.0198 (5)	−0.0018 (3)	0.0076 (4)	−0.0022 (3)
C3	0.0147 (4)	0.0188 (4)	0.0153 (4)	0.0002 (3)	0.0034 (3)	−0.0017 (3)
C4	0.0161 (4)	0.0153 (4)	0.0132 (4)	0.0001 (3)	0.0046 (3)	0.0007 (3)
C5	0.0139 (4)	0.0138 (4)	0.0128 (4)	0.0006 (3)	0.0058 (3)	−0.0011 (3)
C6	0.0147 (4)	0.0171 (4)	0.0126 (4)	−0.0005 (3)	0.0038 (3)	−0.0014 (3)
C7	0.0133 (4)	0.0139 (4)	0.0133 (4)	0.0011 (3)	0.0050 (3)	−0.0004 (3)
C8	0.0136 (4)	0.0139 (4)	0.0139 (4)	0.0001 (3)	0.0051 (3)	−0.0010 (3)
C9	0.0201 (5)	0.0190 (4)	0.0170 (4)	−0.0037 (4)	0.0058 (4)	0.0024 (3)

Geometric parameters (Å, º)

O1—C7	1.2249 (13)	C2—H2	0.972 (14)
O2—N4	1.3872 (12)	C3—C4	1.3886 (14)
O2—H21	0.881 (17)	C3—H3	0.959 (15)
N1—C1	1.3427 (13)	C4—C5	1.3967 (15)
N1—C5	1.3505 (12)	C4—H4	0.982 (14)
N2—C6	1.2832 (13)	C5—C6	1.4691 (14)
N2—N3	1.3746 (12)	C6—H6	0.965 (14)
N3—C7	1.3643 (12)	C7—C8	1.5042 (14)
N3—H31	0.879 (16)	C8—C9	1.4962 (13)
N4—C8	1.2848 (14)	C9—H9A	0.930 (19)
C1—C2	1.3854 (16)	C9—H9B	0.962 (17)
C1—H1	0.970 (15)	C9—H9C	0.985 (17)
C2—C3	1.3921 (14)		
N4—O2—H21	103.1 (11)	N1—C5—C4	122.25 (9)
C1—N1—C5	117.71 (9)	N1—C5—C6	114.84 (9)
C6—N2—N3	114.32 (9)	C4—C5—C6	122.90 (9)
C7—N3—N2	120.18 (8)	N2—C6—C5	120.45 (9)
C7—N3—H31	119.6 (10)	N2—C6—H6	122.7 (9)
N2—N3—H31	120.2 (10)	C5—C6—H6	116.8 (9)
C8—N4—O2	113.47 (8)	O1—C7—N3	124.18 (9)
N1—C1—C2	123.77 (9)	O1—C7—C8	121.44 (9)
N1—C1—H1	115.0 (9)	N3—C7—C8	114.38 (8)
C2—C1—H1	121.2 (9)	N4—C8—C9	127.58 (9)
C1—C2—C3	118.17 (9)	N4—C8—C7	114.58 (9)
C1—C2—H2	121.1 (9)	C9—C8—C7	117.84 (9)
C3—C2—H2	120.7 (9)	C8—C9—H9A	112.3 (11)
C4—C3—C2	119.06 (10)	C8—C9—H9B	111.8 (10)
C4—C3—H3	120.1 (9)	H9A—C9—H9B	104.7 (15)
C2—C3—H3	120.8 (9)	C8—C9—H9C	111.3 (9)
C3—C4—C5	118.98 (9)	H9A—C9—H9C	111.4 (15)
C3—C4—H4	120.7 (8)	H9B—C9—H9C	104.8 (13)
C5—C4—H4	120.3 (8)		

C6—N2—N3—C7	−172.56 (9)	N1—C5—C6—N2	−167.65 (9)
C5—N1—C1—C2	2.31 (14)	C4—C5—C6—N2	13.31 (14)
N1—C1—C2—C3	−0.46 (15)	N2—N3—C7—O1	−1.03 (15)
C1—C2—C3—C4	−1.31 (14)	N2—N3—C7—C8	178.35 (8)
C2—C3—C4—C5	1.19 (14)	O2—N4—C8—C9	−0.54 (14)
C1—N1—C5—C4	−2.42 (14)	O2—N4—C8—C7	−179.80 (7)
C1—N1—C5—C6	178.54 (8)	O1—C7—C8—N4	173.25 (9)
C3—C4—C5—N1	0.71 (14)	N3—C7—C8—N4	−6.14 (12)
C3—C4—C5—C6	179.68 (8)	O1—C7—C8—C9	−6.08 (13)
N3—N2—C6—C5	−178.98 (8)	N3—C7—C8—C9	174.52 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H21···N1^{i}	0.881 (17)	1.897 (18)	2.7667 (17)	168.6 (15)
N3—H31···O1^{ii}	0.879 (16)	2.217 (16)	2.9656 (15)	142.9 (14)
N3—H31···N4	0.879 (16)	2.240 (16)	2.6059 (15)	104.7 (12)

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