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2-Hydroxyimino-*N*′-[1-(2-pyridyl)ethylidene]propanohydrazide

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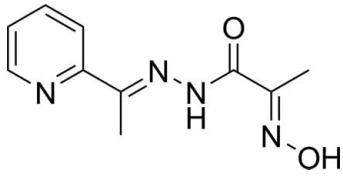
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.046; wR factor = 0.097; data-to-parameter ratio = 6.2.

The title compound, $C_{10}H_{12}N_4O_2$, features an intramolecular N—H···N hydrogen bond formed between the imine NH and oxime N atoms. The oxime group and the amide C=O bond are *anti* to each other. In the crystal, molecules are connected by O—H···O hydrogen bonds into supramolecular zigzag chains along the *c* axis.

Related literature

For oxime and pyridine derivatives, see: Sliva *et al.* (1997*b*); Mokhir *et al.* (2002); Krämer *et al.* (2002); Kovbasyuk *et al.* (2004). 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 2-hydroxyimino-*N*′-[1-(2-pyridyl)ethylidene]propanohydrazone, see: Moroz *et al.* (2008*a*,*b*). For typical bond lengths, see: Bürgi & Dunitz (1994).



Experimental

Crystal data

$C_{10}H_{12}N_4O_2$
M_r = 220.24
Monoclinic, *Cc*
a = 4.4498 (11) Å
b = 22.833 (7) Å
c = 10.955 (3) Å
β = 97.47 (2)°
V = 1103.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.15 × 0.10 × 0.05 mm

Data collection

Oxford Diffraction KM-4/Xcalibur diffractometer with a Sapphire3 detector
Absorption correction: multi-scan (*CrysAlis CCD*; Oxford Diffraction, 2006)
T_{min} = 0.986, T_{max} = 0.995
3899 measured reflections
958 independent reflections
793 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.097
S = 1.10
958 reflections
155 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*OA*···O1^{i}	0.84 (5)	1.88 (5)	2.709 (4)	170 (5)
N3—H3*NA*···N4	0.87 (4)	2.30 (4)	2.640 (4)	104 (3)

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

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: TK2523).

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2-Hydroxyimino-*N*′-[1-(2-pyridyl)ethylidene]propanohydrazide

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S1. Comment

As a part of our on-going work, we would like to report the structure of the title compound (I), Fig. 1, which comprises several groups capable of forming hydrogen bonding interactions: oxime, hydrazone, azomethine, and pyridine. Molecule (I) has been shown previously to form mono- and tetra-nuclear grid-like complexes with 3d-metals (Moroz *et al.*, 2008*a*,*b*).

The C—N and N—O bond lengths in the oxime group, i.e. 1.285 (5) and 1.388 (4) Å, respectively, adopt typical values (Sliva *et al.*, 1997*b*; Mokhir *et al.*, 2002). The oxime group is in an *anti*- position with respect to the amide group, an observation consistent 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 stabilised by an N3—H···N4 intramolecular interaction, Table 1. The $CH_3C(=NOH)C(O)NH$ fragment deviates from planarity as seen in a twist between the oxime and amide groups about the C8—C9 bond; the O1-C8-C9-N4 torsion angle is -164.0 (4)°. The flattened geometry of molecule results in the appearance of short intramolecular contacts H10···O2 is 2.34 Å and H7C···H3N 2.28 Å. The C—N bond distance in the azomethine group is 1.277 (4) Å, and the N2—C6—C1 angle is 115.7 (3)°. The pyridine-N atom is situated in an *anti*- position with respect to the azomethine group. Finally, the C—N and C—C bond lengths within the pyridine ring are normal for 2-substituted pyridine derivatives (Krämer *et al.*, 2002; Kovbasyuk *et al.*, 2004).

In the crystal packing molecules are united by O2—H···O1 hydrogen bonds, Table 1, where oximic-oxygen atom acts as donor and the hydrazone-oxygen atom acts as an acceptor (Fig. 2). This interaction probably results in the elongation of the C8—O1 bond to 1.233 (4) Å as compared with its mean value 1.210 Å (Bürgi & Dunitz, 1994). Due to the presence of the O2—H···O1 hydrogen bonds, zig-zag supramolecular chains are formed along the *c* axis.

S2. Experimental

Compound (I) was prepared according to the reported procedure (Moroz *et al.*, 2008*b*).

S3. Refinement

All H atoms were observed in a difference Fourier map, but C—H hydrogen atoms were placed at calculated positions and treated as riding on their parent atoms [C—H = 0.93-0.96 Å and U_{iso}(H) = 1.2- 1.5U_{eq}(C)]. The N—H and O—H hydrogen atoms were fully refined; O-H = 0.84 (5) Å and N-H = 0.87 (4) Å. In the absence of significant anomalous scattering effects, 766 Friedel pairs were averaged in the final refinement.

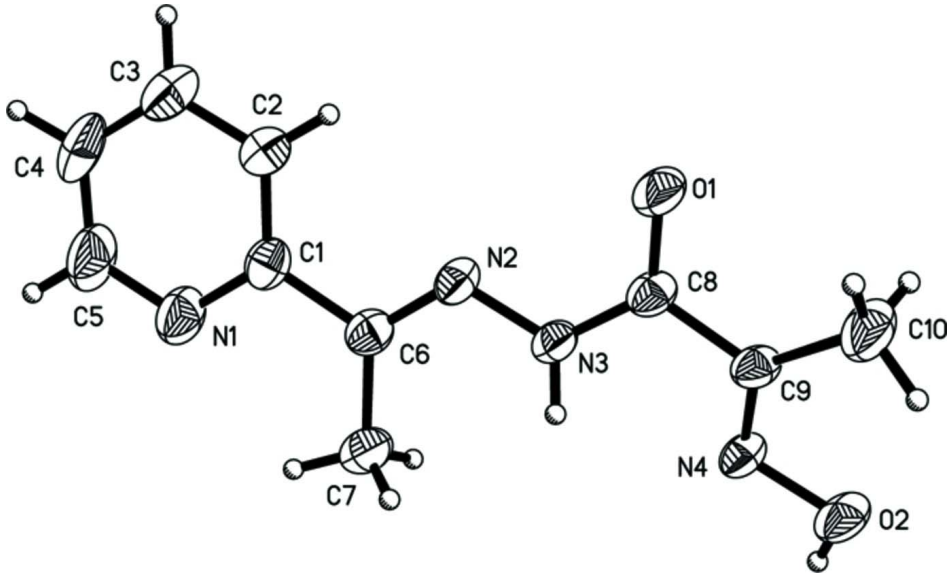

Figure 1

A view of (I), with displacement ellipsoids shown at the 40% probability level and atom labelling.

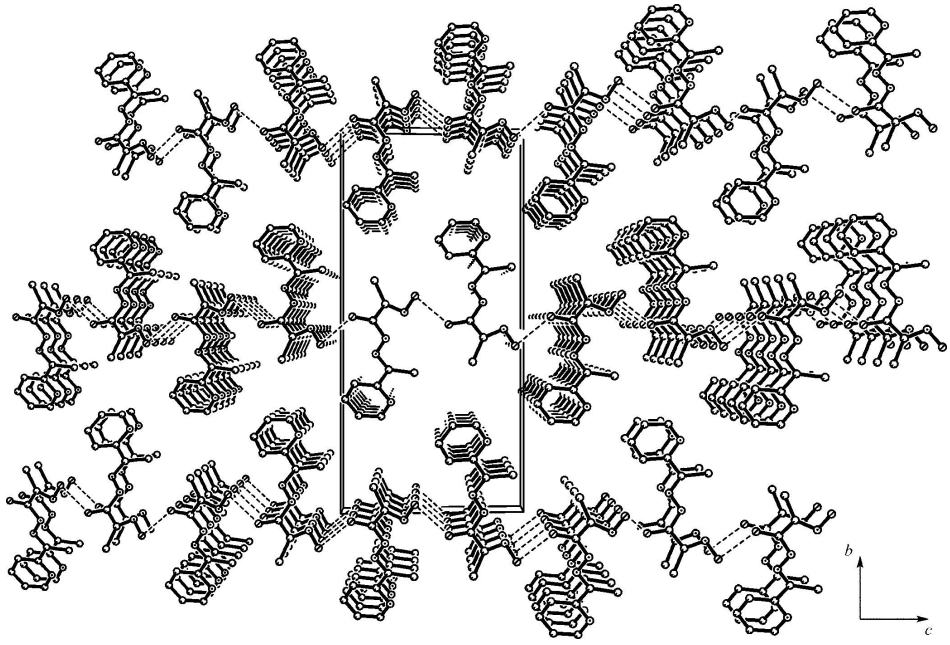

Figure 2

A packing diagram for (I) viewed in projection down the a axis. Hydrogen bonds are indicated by dashed lines; H atoms are omitted for clarity.

2-Hydroxyimino-*N*′-[1-(2-pyridyl)ethylidene]propanohydrazide

Crystal data

$C_{10}H_{12}N_4O_2$
M_r = 220.24
Monoclinic, *Cc*
Hall symbol: C -2yc
a = 4.4498 (11) Å
b = 22.833 (7) Å

c = 10.955 (3) Å
β = 97.47 (2)°
V = 1103.7 (5) Å^3
Z = 4
F(000) = 464
D_x = 1.325 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 5860 reflections
θ = 3.6–32.0°
μ = 0.10 mm^{-1}
T = 293 K
Needles, white
0.15 × 0.10 × 0.05 mm

Data collection

Oxford Diffraction KM-4/Xcalibur diffractometer with a Sapphire3 detector
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1827 pixels mm^{-1}
φ scans and ω scans with κ offset
Absorption correction: multi-scan (*CrysAlis CCD*; Oxford Diffraction, 2006)
T_{min} = 0.986, T_{max} = 0.995

3899 measured reflections
958 independent reflections
793 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 25.0°, θ_{min} = 3.6°
h = −5→5
k = −25→26
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.097
S = 1.10
958 reflections
155 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.2337P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: nd

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}
N1	−0.4385 (8)	0.29326 (15)	0.1660 (4)	0.0601 (10)
N2	−0.0269 (7)	0.41668 (13)	0.0900 (3)	0.0411 (8)
N3	0.1840 (7)	0.45542 (13)	0.1431 (3)	0.0418 (8)
N4	0.6418 (7)	0.52027 (13)	0.2434 (3)	0.0402 (8)
O1	0.1686 (7)	0.50958 (13)	−0.0301 (3)	0.0624 (9)
O2	0.8544 (6)	0.55993 (14)	0.2979 (3)	0.0559 (8)
C1	−0.3464 (9)	0.33542 (16)	0.0958 (4)	0.0439 (10)
C2	−0.4547 (11)	0.3399 (2)	−0.0272 (4)	0.0624 (13)

H2	−0.3891	0.3701	−0.0743	0.075*
C3	−0.6589 (11)	0.2998 (2)	−0.0793 (5)	0.0763 (16)
H3	−0.7310	0.3019	−0.1628	0.092*
C4	−0.7553 (11)	0.2573 (2)	−0.0090 (5)	0.0706 (15)
H4	−0.8962	0.2296	−0.0423	0.085*
C5	−0.6403 (13)	0.2558 (2)	0.1128 (6)	0.0750 (15)
H5	−0.7085	0.2265	0.1614	0.090*
C6	−0.1176 (8)	0.37676 (17)	0.1582 (3)	0.0425 (10)
C7	−0.0053 (11)	0.3692 (2)	0.2922 (4)	0.0607 (13)
H7A	−0.0416	0.4044	0.3357	0.091*
H7B	0.2082	0.3611	0.3022	0.091*
H7C	−0.1103	0.3372	0.3246	0.091*
C8	0.2702 (8)	0.50043 (16)	0.0783 (3)	0.0405 (9)
C9	0.4981 (9)	0.54051 (15)	0.1430 (3)	0.0404 (10)
C10	0.5493 (13)	0.59805 (19)	0.0889 (5)	0.0755 (16)
H10A	0.3634	0.6119	0.0440	0.113*
H10B	0.7001	0.5945	0.0342	0.113*
H10C	0.6177	0.6253	0.1534	0.113*
H3NA	0.239 (8)	0.4565 (15)	0.222 (4)	0.035 (10)*
H2OA	0.932 (11)	0.5383 (19)	0.355 (5)	0.061 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.063 (2)	0.045 (2)	0.069 (3)	−0.013 (2)	−0.0028 (19)	0.0015 (19)
N2	0.0370 (18)	0.0396 (16)	0.0439 (18)	−0.0042 (15)	−0.0056 (14)	−0.0027 (15)
N3	0.0436 (19)	0.0436 (19)	0.0353 (19)	−0.0083 (16)	−0.0062 (15)	−0.0009 (14)
N4	0.0361 (18)	0.0403 (17)	0.0408 (18)	−0.0022 (15)	−0.0078 (14)	−0.0055 (14)
O1	0.071 (2)	0.0603 (18)	0.0478 (17)	−0.0208 (16)	−0.0238 (15)	0.0105 (14)
O2	0.0555 (18)	0.0570 (18)	0.0478 (17)	−0.0045 (15)	−0.0220 (13)	−0.0013 (14)
C1	0.036 (2)	0.041 (2)	0.054 (2)	0.0004 (18)	0.0032 (19)	−0.0047 (18)
C2	0.068 (3)	0.071 (3)	0.046 (2)	−0.025 (3)	0.002 (2)	−0.007 (2)
C3	0.072 (4)	0.089 (4)	0.063 (3)	−0.026 (3)	−0.007 (3)	−0.020 (3)
C4	0.051 (3)	0.058 (3)	0.100 (4)	−0.020 (2)	0.000 (3)	−0.028 (3)
C5	0.078 (4)	0.052 (3)	0.093 (4)	−0.028 (3)	0.004 (3)	−0.003 (3)
C6	0.045 (3)	0.038 (2)	0.043 (2)	0.0000 (18)	0.0022 (19)	−0.0043 (17)
C7	0.075 (3)	0.061 (3)	0.044 (2)	−0.017 (2)	−0.005 (2)	−0.0039 (19)
C8	0.040 (2)	0.038 (2)	0.039 (2)	0.0029 (18)	−0.0085 (17)	0.0004 (17)
C9	0.047 (3)	0.0368 (19)	0.0342 (19)	0.0021 (18)	−0.0066 (17)	−0.0001 (16)
C10	0.087 (4)	0.059 (3)	0.069 (3)	−0.024 (3)	−0.034 (3)	0.015 (3)

Geometric parameters (Å, º)

N1—C5	1.320 (6)	C3—C4	1.345 (7)
N1—C1	1.330 (5)	C3—H3	0.9300
N2—C6	1.277 (4)	C4—C5	1.366 (8)
N2—N3	1.364 (4)	C4—H4	0.9300
N3—C8	1.334 (5)	C5—H5	0.9300

N3—H3NA	0.87 (4)	C6—C7	1.498 (5)
N4—C9	1.285 (5)	C7—H7A	0.9600
N4—O2	1.388 (4)	C7—H7B	0.9600
O1—C8	1.233 (4)	C7—H7C	0.9600
O2—H2OA	0.84 (5)	C8—C9	1.477 (5)
C1—C2	1.374 (6)	C9—C10	1.471 (5)
C1—C6	1.488 (5)	C10—H10A	0.9600
C2—C3	1.362 (6)	C10—H10B	0.9600
C2—H2	0.9300	C10—H10C	0.9600

C5—N1—C1	117.2 (4)	N2—C6—C1	115.7 (3)
C6—N2—N3	117.7 (3)	N2—C6—C7	124.4 (4)
C8—N3—N2	120.1 (3)	C1—C6—C7	119.9 (4)
C8—N3—H3NA	116 (2)	C6—C7—H7A	109.5
N2—N3—H3NA	122 (2)	C6—C7—H7B	109.5
C9—N4—O2	111.7 (3)	H7A—C7—H7B	109.5
N4—O2—H2OA	97 (3)	C6—C7—H7C	109.5
N1—C1—C2	121.7 (4)	H7A—C7—H7C	109.5
N1—C1—C6	115.9 (3)	H7B—C7—H7C	109.5
C2—C1—C6	122.4 (4)	O1—C8—N3	123.3 (4)
C3—C2—C1	119.4 (5)	O1—C8—C9	120.0 (3)
C3—C2—H2	120.3	N3—C8—C9	116.7 (3)
C1—C2—H2	120.3	N4—C9—C10	125.5 (4)
C4—C3—C2	119.3 (5)	N4—C9—C8	115.0 (3)
C4—C3—H3	120.3	C10—C9—C8	119.5 (3)
C2—C3—H3	120.3	C9—C10—H10A	109.5
C3—C4—C5	118.1 (4)	C9—C10—H10B	109.5
C3—C4—H4	121.0	H10A—C10—H10B	109.5
C5—C4—H4	121.0	C9—C10—H10C	109.5
N1—C5—C4	124.2 (5)	H10A—C10—H10C	109.5
N1—C5—H5	117.9	H10B—C10—H10C	109.5
C4—C5—H5	117.9		

C6—N2—N3—C8	−175.2 (3)	C2—C1—C6—N2	−0.3 (6)
C5—N1—C1—C2	−0.3 (6)	N1—C1—C6—C7	0.2 (5)
C5—N1—C1—C6	−179.7 (4)	C2—C1—C6—C7	−179.2 (4)
N1—C1—C2—C3	−0.9 (7)	N2—N3—C8—O1	−0.1 (6)
C6—C1—C2—C3	178.5 (4)	N2—N3—C8—C9	179.6 (3)
C1—C2—C3—C4	1.3 (8)	O2—N4—C9—C10	0.7 (6)
C2—C3—C4—C5	−0.7 (8)	O2—N4—C9—C8	179.1 (3)
C1—N1—C5—C4	0.9 (8)	O1—C8—C9—N4	−164.0 (4)
C3—C4—C5—N1	−0.5 (9)	N3—C8—C9—N4	16.3 (5)
N3—N2—C6—C1	−179.7 (3)	O1—C8—C9—C10	14.5 (6)
N3—N2—C6—C7	−0.8 (5)	N3—C8—C9—C10	−165.2 (4)
N1—C1—C6—N2	179.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*OA*···O1^{i}	0.84 (5)	1.88 (5)	2.709 (4)	170 (5)
N3—H3*NA*···N4	0.87 (4)	2.30 (4)	2.640 (4)	104 (3)

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