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N'-(2-Hydroxybenzylidene)-2-(hydroxyimino)propanohydrazide

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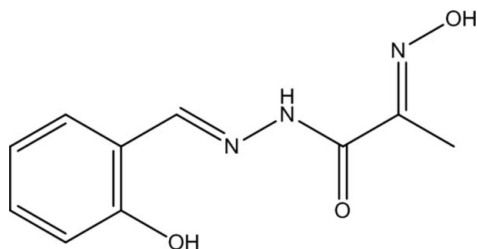
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.029; wR factor = 0.086; data-to-parameter ratio = 16.7.

The molecule of the title compound, $\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}_3$, adopts an all-*trans* conformation and is approximately planar, the largest deviation from the least-squares plane through all non-H atoms being 0.261 (1) Å. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond occurs. In the crystal, the molecules are packed into layers lying parallel to the *ab* plane by π -stacking interactions between the benzene ring of one molecule and the C—N bond of the oxime group of another molecule; the shortest intermolecular C \cdots C separation within the layer is 3.412 (1) Å. The layers are connected by $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the preparation and characterization of 3d metal complexes with related oxime derivatives, see: Kandal et al. (2005); Moroz et al. (2010). For the crystal structures of similar oxime derivatives, see: Świątek-Kozłowska et al. (2000); Mokhir et al. (2002); Sachse et al. (2008). For 2-hydroxyiminopropanamide and amide derivatives of 2-hydroxyiminopropanoic acid, see: Onindo et al. (1995); Duda et al. (1997); Sliva et al. (1997). For the synthesis of 2-(hydroxyimino)propanehydrazide, see: Fritsky et al. (1998). For related structures, see: Krämer & Fritsky (2000); Wörl et al. (2005).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}_3$
 $M_r = 221.22$
Monoclinic, *Cc*
 $a = 11.2296$ (4) Å
 $b = 8.1905$ (4) Å
 $c = 11.1000$ (5) Å
 $\beta = 102.223$ (2)°
 $V = 997.79$ (8) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 100$ K
 $0.61 \times 0.47 \times 0.34$ mm

Data collection

Bruker Kappa APEXII DUO CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008)
 $T_{\min} = 0.935$, $T_{\max} = 0.964$
16456 measured reflections
2465 independent reflections
2383 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.086$
 $S = 1.07$
2465 reflections
148 parameters
2 restraints
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.42$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.84	1.85	2.5808 (10)	144
O3—H3 \cdots O2 ⁱ	0.84	1.82	2.6518 (9)	171
N2—H2 \cdots O1 ⁱⁱ	0.88	2.32	3.1535 (9)	157

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

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2011); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YK2025).

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supporting information

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***N'*-(2-Hydroxybenzylidene)-2-(hydroxyimino)propanohydrazide**

Maxym O. Plutenko, Rostyslav D. Lampeka, Yurii S. Moroz, Matti Haukka and Svetlana V. Pavlova

S1. Comment

Polynucleative oxime ligands attract considerable interest because of their ability to act as efficient chelating agents with respect to 3 d-metal ions and their tendency to form polynuclear metal complexes (Kanderal *et al.* 2005; Moroz *et al.* 2010). In the present work we present the synthesis and structure of the title compound (1) (Fig. 1), which comprises several donor groups: oxime, hydrazone, azomethine, and phenolic.

In the structure 1 the *N'*-(2-hydroxybenzylidene)-2-(hydroxyimino)propanehydrazide molecules are connected by extensive system of hydrogen bonds. The bond lengths N—O and C—N in the oxime group are 1.3838 (9) and 1.2854 (9) Å respectively, which is typical for protonated moieties of this type (Świątek-Kozłowska *et al.*, 2000; Mokhir *et al.*, 2002; Sachse *et al.*, 2008). 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).

Bond lengths N—N', N—C and C—O of the hydrazone group are 1.3643 (9), 1.3544 (9) and 1.2353 (9) Å, respectively, and are typical for the protonated hydrazone groups (Moroz *et al.*, 2010). The oxime and the hydrazide groups are situated in *trans*-position around the C(8)—C(9) bond. The CH₃C(NOHC(O)NH) fragment is almost planar (deviations of the non-hydrogen atoms from the moiety's mean plane are less than 0.2 Å).

The C—C (1.3814 (11) – 1.4115 (9) Å) bond lengths in the benzene ring have their typical values (Krämer *et al.*, 2000; Wörl *et al.*, 2005). The angles C—C'-C'', C—N—C' and N—C—C' are near 120°.

There are three hydrogen bonds in structure of 1 (Table 2). The O1—H1···N1 is an intramolecular hydrogen bond, where the phenolic oxygen atom acts as donor and the azomethine nitrogen atom acts as receptor. The O3—H3···O2 and N2—H2···O1 hydrogen bonds are intermolecular, the oximic oxygen and the hydrazone nitrogen atoms act as donors and the hydrazone oxygen and the phenolic oxygen atoms act as acceptors.

In the crystal packing, molecules of 1 form layers parallel to *ab* plane. The molecules in the layer are connected by π -stacking between the benzene ring of one molecule and C—N bond of the oxime group of another molecule. The distance between two planes formed by neighboring molecules is 3.3493 (7) Å. The layers are connected by extensive system of hydrogen bonds.

S2. Experimental

A mixture of 2-(hydroxyimino)propanehydrazide synthesized according to (Fritsky *et al.*, 1998) (0.117 g, 0.1 mmol) and salicylic aldehyde (0.122 g, 0.1 mmol) in 10 ml of methanol was heated to reflux for 2 h. On cooling to room temperature, a solid precipitate was formed. The solid was filtered and then recrystallized from methanol. Yellowish needle crystals of 1 were obtained by slow evaporation of the methanolic solution. Yield: 2 g (90%).

S3. Refinement

All hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.95–0.98 Å, N—H = 0.88 Å, O—H = 0.84 Å, and $U_{\text{iso}} = 1.2\text{--}1.5 U_{\text{eq}}(\text{parent atom})$. The highest peak is located 0.64 Å from atom N3 and the deepest hole is located 0.30 Å from atom H3.

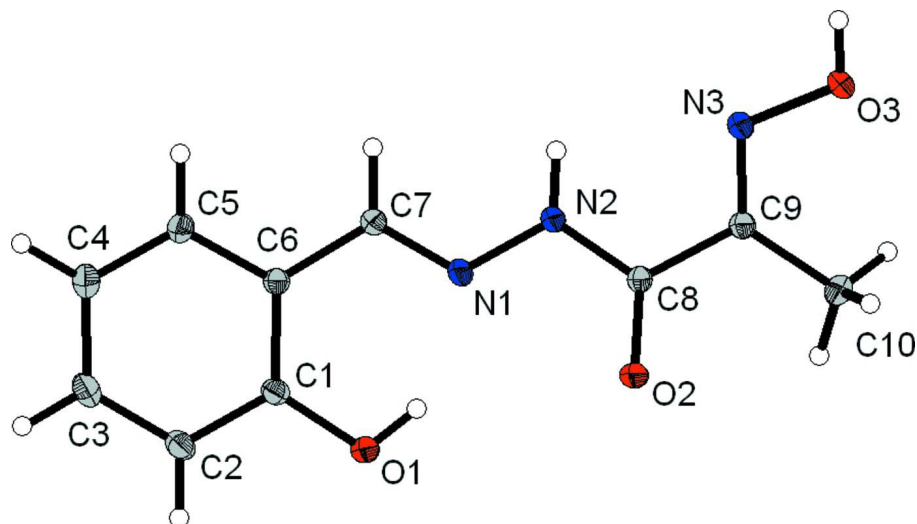


Figure 1

The molecular structure of the title compound, with displacement ellipsoids shown at the 50% probability level and atom labelling.

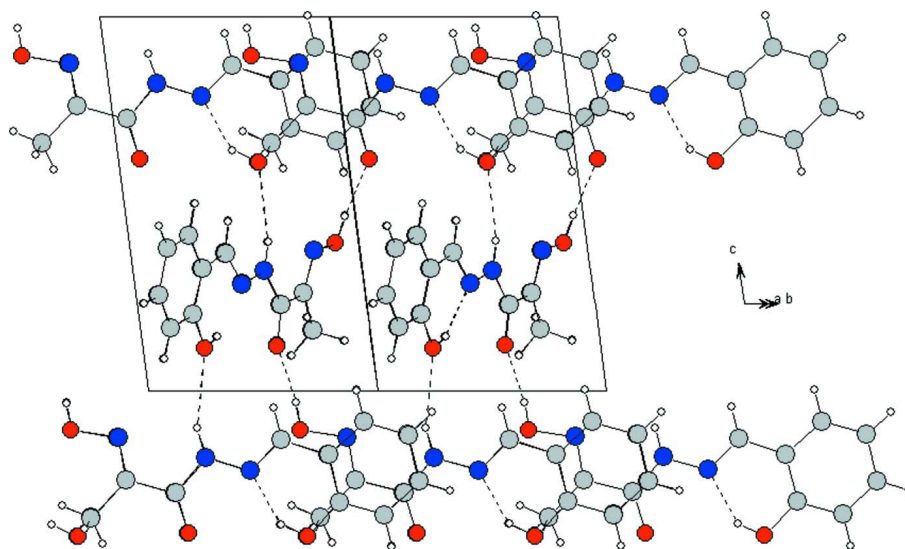


Figure 2

Crystal packing of the title compound.

N'-(2-Hydroxybenzylidene)-2-(hydroxyimino)propanohydrazide

Crystal data

$\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}_3$
 $M_r = 221.22$

Monoclinic, *Cc*
Hall symbol: *C* -2yc

$a = 11.2296$ (4) Å
 $b = 8.1905$ (4) Å
 $c = 11.1000$ (5) Å
 $\beta = 102.223$ (2)°
 $V = 997.79$ (8) Å³
 $Z = 4$
 $F(000) = 464$
 $D_x = 1.473$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 9875 reflections
 $\theta = 3.1\text{--}36.5^\circ$
 $\mu = 0.11$ mm⁻¹
 $T = 100$ K
 Block, yellow
 $0.61 \times 0.47 \times 0.34$ mm

Data collection

Bruker Kappa APEXII DUO CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Curved graphite crystal monochromator
 Detector resolution: 16 pixels mm⁻¹
 φ scans and ω scans with κ offset
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2008)
 $T_{\min} = 0.935$, $T_{\max} = 0.964$

16456 measured reflections
 2465 independent reflections
 2383 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 $\theta_{\max} = 36.6^\circ$, $\theta_{\min} = 3.1^\circ$
 $h = -18 \rightarrow 18$
 $k = -13 \rightarrow 13$
 $l = -17 \rightarrow 18$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.086$
 $S = 1.07$
 2465 reflections
 148 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-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.0678P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.42$ e Å⁻³
 $\Delta\rho_{\min} = -0.22$ e Å⁻³

Special details

Geometry. All s.u.'s (except the s.u.'s in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.43931 (7)	-0.17009 (9)	0.11307 (6)	0.01652 (13)
H1	0.4154	-0.0863	0.1446	0.025*
O2	0.34012 (7)	0.24663 (9)	0.12079 (6)	0.01761 (13)
O3	0.27014 (7)	0.63005 (9)	0.39602 (7)	0.01675 (13)
H3	0.2975	0.6608	0.4688	0.025*
N1	0.43375 (7)	0.03499 (9)	0.28832 (7)	0.01324 (13)
N2	0.39288 (7)	0.18082 (9)	0.32287 (6)	0.01328 (12)
H2	0.3983	0.2071	0.4007	0.016*

N3	0.32114 (7)	0.48105 (9)	0.37696 (7)	0.01365 (13)
C1	0.50374 (8)	-0.26684 (11)	0.20397 (8)	0.01297 (13)
C2	0.54689 (9)	-0.41516 (12)	0.16937 (8)	0.01686 (15)
H2A	0.5302	-0.4465	0.0851	0.020*
C3	0.61440 (9)	-0.51730 (12)	0.25817 (9)	0.01803 (16)
H3A	0.6450	-0.6173	0.2337	0.022*
C4	0.63779 (9)	-0.47511 (11)	0.38250 (9)	0.01695 (15)
H4	0.6840	-0.5455	0.4427	0.020*
C5	0.59273 (8)	-0.32933 (11)	0.41698 (8)	0.01476 (14)
H5	0.6073	-0.3011	0.5019	0.018*
C6	0.52600 (8)	-0.22227 (10)	0.32956 (7)	0.01217 (13)
C7	0.48236 (8)	-0.06904 (11)	0.37043 (7)	0.01343 (14)
H7	0.4897	-0.0466	0.4557	0.016*
C8	0.34332 (8)	0.28258 (10)	0.22951 (7)	0.01258 (13)
C9	0.29200 (7)	0.43934 (10)	0.26290 (7)	0.01256 (13)
C10	0.21336 (9)	0.53605 (12)	0.16288 (8)	0.01674 (15)
H10A	0.1476	0.5877	0.1948	0.025*
H10B	0.1784	0.4635	0.0943	0.025*
H10C	0.2625	0.6204	0.1339	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0240 (3)	0.0136 (3)	0.0116 (2)	0.0019 (2)	0.0029 (2)	0.0001 (2)
O2	0.0277 (3)	0.0146 (3)	0.0108 (2)	0.0026 (2)	0.0044 (2)	-0.0002 (2)
O3	0.0226 (3)	0.0130 (3)	0.0146 (2)	0.0040 (2)	0.0037 (2)	-0.0023 (2)
N1	0.0175 (3)	0.0093 (3)	0.0132 (3)	0.0009 (2)	0.0040 (2)	-0.0002 (2)
N2	0.0185 (3)	0.0099 (3)	0.0115 (2)	0.0019 (2)	0.0034 (2)	-0.0002 (2)
N3	0.0167 (3)	0.0111 (3)	0.0132 (3)	0.0007 (2)	0.0034 (2)	-0.0006 (2)
C1	0.0156 (3)	0.0117 (3)	0.0121 (3)	-0.0010 (2)	0.0040 (2)	-0.0008 (2)
C2	0.0217 (4)	0.0137 (3)	0.0161 (3)	0.0011 (3)	0.0062 (3)	-0.0024 (3)
C3	0.0201 (4)	0.0137 (3)	0.0215 (4)	0.0026 (3)	0.0071 (3)	-0.0018 (3)
C4	0.0176 (4)	0.0136 (3)	0.0198 (3)	0.0023 (3)	0.0042 (3)	0.0016 (3)
C5	0.0169 (3)	0.0125 (3)	0.0142 (3)	0.0007 (3)	0.0019 (3)	0.0011 (2)
C6	0.0149 (3)	0.0102 (3)	0.0115 (3)	-0.0006 (2)	0.0030 (2)	0.0002 (2)
C7	0.0169 (3)	0.0113 (3)	0.0117 (3)	0.0007 (3)	0.0023 (2)	-0.0006 (2)
C8	0.0155 (3)	0.0102 (3)	0.0121 (3)	-0.0001 (2)	0.0028 (2)	0.0004 (2)
C9	0.0145 (3)	0.0109 (3)	0.0123 (3)	0.0000 (2)	0.0029 (2)	0.0001 (2)
C10	0.0178 (4)	0.0173 (4)	0.0145 (3)	0.0039 (3)	0.0019 (3)	0.0022 (3)

Geometric parameters (Å, °)

O1—C1	1.3640 (11)	C3—C4	1.3926 (14)
O1—H1	0.8400	C3—H3A	0.9500
O2—C8	1.2352 (10)	C4—C5	1.3822 (13)
O3—N3	1.3832 (10)	C4—H4	0.9500
O3—H3	0.8400	C5—C6	1.4014 (11)
N1—C7	1.2822 (10)	C5—H5	0.9500

N1—N2	1.3634 (10)	C6—C7	1.4543 (12)
N2—C8	1.3543 (10)	C7—H7	0.9500
N2—H2	0.8800	C8—C9	1.4861 (12)
N3—C9	1.2850 (11)	C9—C10	1.4916 (12)
C1—C2	1.3917 (12)	C10—H10A	0.9800
C1—C6	1.4113 (11)	C10—H10B	0.9800
C2—C3	1.3891 (14)	C10—H10C	0.9800
C2—H2A	0.9500		
C1—O1—H1	109.5	C4—C5—H5	119.3
N3—O3—H3	109.5	C6—C5—H5	119.3
C7—N1—N2	120.02 (7)	C5—C6—C1	118.62 (7)
C8—N2—N1	115.60 (7)	C5—C6—C7	119.34 (7)
C8—N2—H2	122.2	C1—C6—C7	122.03 (7)
N1—N2—H2	122.2	N1—C7—C6	118.21 (7)
C9—N3—O3	110.98 (7)	N1—C7—H7	120.9
O1—C1—C2	117.65 (7)	C6—C7—H7	120.9
O1—C1—C6	122.40 (7)	O2—C8—N2	121.54 (8)
C2—C1—C6	119.95 (8)	O2—C8—C9	121.13 (7)
C3—C2—C1	119.97 (8)	N2—C8—C9	117.32 (7)
C3—C2—H2A	120.0	N3—C9—C8	116.37 (7)
C1—C2—H2A	120.0	N3—C9—C10	125.40 (8)
C2—C3—C4	120.92 (9)	C8—C9—C10	118.22 (7)
C2—C3—H3A	119.5	C9—C10—H10A	109.5
C4—C3—H3A	119.5	C9—C10—H10B	109.5
C5—C4—C3	119.05 (8)	H10A—C10—H10B	109.5
C5—C4—H4	120.5	C9—C10—H10C	109.5
C3—C4—H4	120.5	H10A—C10—H10C	109.5
C4—C5—C6	121.48 (8)	H10B—C10—H10C	109.5

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.84	1.85	2.5808 (10)	144
O3—H3 \cdots O2 ⁱ	0.84	1.82	2.6518 (9)	171
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