

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(E)-2-Hydroxynaphthalene-1-carbaldehyde semicarbazone

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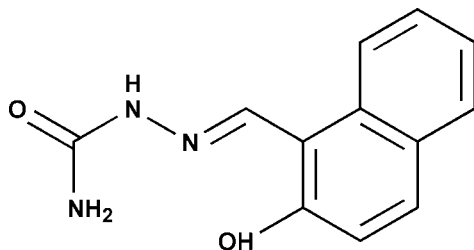
Received 23 March 2009; accepted 30 March 2009

 Key indicators: single-crystal X-ray study; $T = 283$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.037; wR factor = 0.107; data-to-parameter ratio = 15.0.

The title compound, $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_2$, adopts an *E* or *trans* configuration with respect to the $\text{C}=\text{N}$ bond. There is an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond involving the hydroxyl H atom and an N atom of the hydrazine group. In the crystal structure, molecules are connected *via* $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a three-dimensional network.

Related literature

For the potential pharmacological and antitumor properties of hydrazones and Schiff bases, see: Karthikeyan *et al.* (2006); Khattab (2005); Kucukguzel *et al.* (2006). For related structures, see: Okabe *et al.* (1993); Zhang *et al.* (1999); Xu *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_2$
 $M_r = 229.24$

 Monoclinic, $P2_1/c$
 $a = 16.091$ (3) Å

 $b = 4.7350$ (9) Å
 $c = 15.776$ (3) Å
 $\beta = 114.26$ (3)°
 $V = 1095.8$ (4) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 283$ K
 $0.20 \times 0.10 \times 0.10$ mm

Data collection

 Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.988$, $T_{\max} = 0.989$
 6629 measured reflections
 2324 independent reflections
 1550 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.107$
 $S = 1.05$
 2324 reflections

 155 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.15$ 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	0.82	1.83	2.5563 (15)	146
N2—H2 \cdots O2 ⁱ	0.86	2.01	2.8385 (15)	163
N3—H3A \cdots O1 ⁱⁱ	0.86	2.14	2.9871 (15)	171
N3—H3B \cdots O2 ⁱⁱⁱ	0.86	2.63	3.0957 (16)	115

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Key Project of Science and Technology of Anhui, People's Republic of China (grant No. 08010302218).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2105).

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supplementary materials

Acta Cryst. (2009). E65, o1047 [doi:10.1107/S160053680901174X]

(E)-2-Hydroxynaphthalene-1-carbaldehyde semicarbazone

H.-J. Xu, N.-N. Du, X.-Y. Jiang, L.-Q. Sheng and Y.-P. Tian

Comment

Hydrazones and Schiff bases have attracted much attention for their excellent biological properties, especially for their potential pharmacological and antitumor properties (Kucukguzel *et al.*, 2006; Khattab, 2005; Karthikeyan *et al.*, 2006; Okabe *et al.*, 1993). As we are interested in this field of research (Xu *et al.* 2009), we report herein on the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1 and geometrical parameters are given in the archived CIF. The molecule has a *trans* configuration about the C=N bond. There is an intramolecular O-H...N hydrogen bond involving the naphthalene hydroxyl substituent and atom N1 of the hydrazine group (Table 1).

In the crystal structure symmetry related molecules are connected *via* N-H...O hydrogen bonds so forming a three-dimensional structure (Table 1 and Fig. 2).

Experimental

The ligand H₂L was prepared according to the reported procedure (Zhang *et al.*, 1999). A solution of semicarbazide hydrochloride (0.112 g, 1 mmol) in 5 ml of ethanol was added slowly to a solution of 2-hydro-1-naphthaldehyde (0.172 g, 1 mmol) in 15 ml absolute ethanol, under heating and stirring. The mixture was refluxed for 3 h, then cooled to rt and left to stand in air for 5 days. Yellow block-shaped crystals were formed on slow evaporation of the solvent.

Refinement

All the H-atoms were placed in calculated positions [O—H = 0.82 Å, N—H = 0.86 Å, C—H = 0.93 Å] and treated as riding [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{parent O-atom})$ and $= 1.2U_{\text{eq}}(\text{parent C-atom and N-atom})$].

Figures

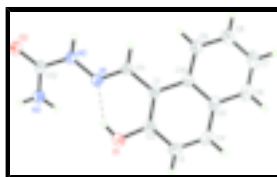


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids (H-atoms have been omitted for clarity).

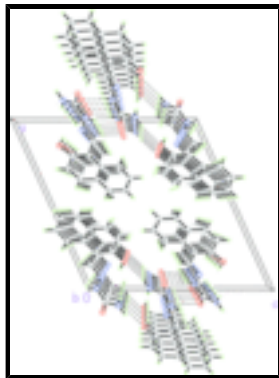


Fig. 2. Crystal packing of the title compound viewed along the b axis. The intra- and inter-molecular hydrogen bonds are shown as dashed lines (details are given in Table 1).

(E)-2-Hydroxynaphthalene-1-carbaldehyde semicarbazone

Crystal data

$C_{12}H_{11}N_3O_2$

$M_r = 229.24$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 16.091 (3) \text{ \AA}$

$b = 4.7350 (9) \text{ \AA}$

$c = 15.776 (3) \text{ \AA}$

$\beta = 114.26 (3)^\circ$

$V = 1095.8 (4) \text{ \AA}^3$

$Z = 4$

$F_{000} = 480$

$D_x = 1.389 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 628 reflections

$\theta = 2.5\text{--}15^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 283 \text{ K}$

Block, yellow

$0.20 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 283 \text{ K}$

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.988$, $T_{\max} = 0.989$

6629 measured reflections

2324 independent reflections

1550 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\max} = 27.0^\circ$

$\theta_{\min} = 2.8^\circ$

$h = -20 \rightarrow 20$

$k = -6 \rightarrow 4$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.107$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2]$

$S = 1.05$

2324 reflections

155 parameters

Primary atom site location: structure-invariant direct methods

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.23742 (8)	0.2085 (3)	0.15396 (8)	0.0367 (3)
C2	0.24256 (9)	0.1120 (3)	0.23930 (9)	0.0441 (3)
C3	0.30716 (10)	-0.0898 (3)	0.29109 (10)	0.0538 (4)
H3C	0.3094	-0.1497	0.3481	0.065*
C4	0.36618 (10)	-0.1978 (3)	0.25859 (10)	0.0530 (4)
H4A	0.4089	-0.3307	0.2939	0.064*
C5	0.36419 (8)	-0.1126 (3)	0.17185 (9)	0.0435 (3)
C6	0.42499 (10)	-0.2280 (3)	0.13721 (11)	0.0572 (4)
H6A	0.4674	-0.3622	0.1722	0.069*
C7	0.42258 (10)	-0.1462 (4)	0.05361 (12)	0.0623 (4)
H7A	0.4634	-0.2231	0.0320	0.075*
C8	0.35881 (11)	0.0531 (3)	0.00052 (11)	0.0571 (4)
H8A	0.3572	0.1088	-0.0567	0.068*
C9	0.29852 (9)	0.1678 (3)	0.03161 (9)	0.0477 (4)
H9A	0.2560	0.2989	-0.0053	0.057*
C10	0.29938 (8)	0.0915 (3)	0.11853 (9)	0.0377 (3)
C11	0.17325 (8)	0.4299 (3)	0.10430 (9)	0.0383 (3)
H11	0.1777	0.5185	0.0537	0.046*
C12	-0.01922 (9)	0.7854 (3)	0.10281 (9)	0.0405 (3)
N1	0.11062 (7)	0.5029 (2)	0.13008 (7)	0.0419 (3)
N2	0.05492 (7)	0.7251 (2)	0.08591 (7)	0.0447 (3)
H2	0.0671	0.8266	0.0473	0.054*
N3	-0.04085 (8)	0.6020 (3)	0.15536 (8)	0.0551 (4)
H3A	-0.0873	0.6333	0.1678	0.066*
H3B	-0.0083	0.4530	0.1765	0.066*
O1	0.18694 (7)	0.2086 (2)	0.27791 (7)	0.0616 (3)

supplementary materials

H1	0.1521	0.3272	0.2436	0.092*
O2	-0.06341 (7)	1.00232 (19)	0.07058 (6)	0.0510 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0325 (6)	0.0356 (7)	0.0400 (7)	-0.0004 (5)	0.0127 (6)	0.0012 (6)
C2	0.0426 (7)	0.0456 (8)	0.0445 (7)	0.0005 (6)	0.0183 (6)	0.0036 (6)
C3	0.0547 (9)	0.0555 (9)	0.0458 (8)	0.0047 (7)	0.0151 (7)	0.0146 (7)
C4	0.0425 (8)	0.0467 (9)	0.0575 (9)	0.0073 (7)	0.0080 (7)	0.0094 (7)
C5	0.0317 (7)	0.0378 (8)	0.0543 (8)	-0.0019 (6)	0.0108 (6)	-0.0042 (6)
C6	0.0386 (8)	0.0501 (9)	0.0765 (11)	0.0066 (7)	0.0172 (7)	-0.0063 (8)
C7	0.0479 (8)	0.0625 (11)	0.0841 (12)	0.0004 (8)	0.0347 (8)	-0.0223 (9)
C8	0.0560 (9)	0.0636 (10)	0.0593 (9)	-0.0035 (8)	0.0313 (8)	-0.0097 (8)
C9	0.0436 (8)	0.0519 (9)	0.0478 (8)	0.0035 (6)	0.0191 (7)	-0.0028 (6)
C10	0.0313 (6)	0.0356 (7)	0.0438 (7)	-0.0040 (5)	0.0131 (6)	-0.0052 (6)
C11	0.0371 (7)	0.0381 (8)	0.0389 (7)	-0.0001 (6)	0.0150 (6)	0.0004 (6)
C12	0.0416 (7)	0.0368 (8)	0.0434 (7)	0.0014 (6)	0.0177 (6)	-0.0071 (6)
N1	0.0385 (6)	0.0396 (6)	0.0480 (6)	0.0057 (5)	0.0182 (5)	0.0030 (5)
N2	0.0428 (6)	0.0401 (7)	0.0560 (7)	0.0097 (5)	0.0250 (6)	0.0108 (5)
N3	0.0579 (8)	0.0484 (8)	0.0733 (8)	0.0123 (6)	0.0414 (7)	0.0112 (6)
O1	0.0642 (7)	0.0759 (8)	0.0575 (6)	0.0184 (6)	0.0378 (6)	0.0190 (5)
O2	0.0525 (6)	0.0432 (6)	0.0616 (6)	0.0139 (5)	0.0280 (5)	0.0039 (5)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.3916 (17)	C8—C9	1.3667 (18)
C1—C10	1.4390 (16)	C8—H8A	0.9300
C1—C11	1.4540 (17)	C9—C10	1.4126 (18)
C2—O1	1.3525 (15)	C9—H9A	0.9300
C2—C3	1.4013 (19)	C11—N1	1.2798 (15)
C3—C4	1.352 (2)	C11—H11	0.9300
C3—H3C	0.9300	C12—O2	1.2336 (14)
C4—C5	1.414 (2)	C12—N3	1.3417 (16)
C4—H4A	0.9300	C12—N2	1.3554 (15)
C5—C6	1.4120 (19)	N1—N2	1.3719 (14)
C5—C10	1.4172 (18)	N2—H2	0.8600
C6—C7	1.360 (2)	N3—H3A	0.8600
C6—H6A	0.9300	N3—H3B	0.8600
C7—C8	1.392 (2)	O1—H1	0.8200
C7—H7A	0.9300		
C2—C1—C10	118.31 (12)	C9—C8—H8A	119.7
C2—C1—C11	120.31 (11)	C7—C8—H8A	119.7
C10—C1—C11	121.34 (11)	C8—C9—C10	121.44 (14)
O1—C2—C1	122.52 (12)	C8—C9—H9A	119.3
O1—C2—C3	115.95 (12)	C10—C9—H9A	119.3
C1—C2—C3	121.53 (12)	C9—C10—C5	117.51 (11)
C4—C3—C2	120.36 (13)	C9—C10—C1	123.13 (12)

C4—C3—H3C	119.8	C5—C10—C1	119.36 (11)
C2—C3—H3C	119.8	N1—C11—C1	120.08 (11)
C3—C4—C5	121.27 (13)	N1—C11—H11	120.0
C3—C4—H4A	119.4	C1—C11—H11	120.0
C5—C4—H4A	119.4	O2—C12—N3	122.80 (12)
C6—C5—C4	121.35 (14)	O2—C12—N2	119.96 (12)
C6—C5—C10	119.48 (13)	N3—C12—N2	117.24 (12)
C4—C5—C10	119.17 (12)	C11—N1—N2	118.76 (10)
C7—C6—C5	121.13 (15)	C12—N2—N1	120.39 (10)
C7—C6—H6A	119.4	C12—N2—H2	119.8
C5—C6—H6A	119.4	N1—N2—H2	119.8
C6—C7—C8	119.79 (13)	C12—N3—H3A	120.0
C6—C7—H7A	120.1	C12—N3—H3B	120.0
C8—C7—H7A	120.1	H3A—N3—H3B	120.0
C9—C8—C7	120.64 (14)	C2—O1—H1	109.5
C10—C1—C2—O1	-179.71 (12)	C8—C9—C10—C1	179.65 (12)
C11—C1—C2—O1	2.7 (2)	C6—C5—C10—C9	0.63 (18)
C10—C1—C2—C3	1.2 (2)	C4—C5—C10—C9	-179.00 (12)
C11—C1—C2—C3	-176.41 (13)	C6—C5—C10—C1	179.93 (12)
O1—C2—C3—C4	-179.60 (13)	C4—C5—C10—C1	0.30 (18)
C1—C2—C3—C4	-0.4 (2)	C2—C1—C10—C9	178.15 (12)
C2—C3—C4—C5	-0.4 (2)	C11—C1—C10—C9	-4.27 (19)
C3—C4—C5—C6	-179.15 (13)	C2—C1—C10—C5	-1.10 (18)
C3—C4—C5—C10	0.5 (2)	C11—C1—C10—C5	176.48 (11)
C4—C5—C6—C7	179.75 (13)	C2—C1—C11—N1	-12.17 (19)
C10—C5—C6—C7	0.1 (2)	C10—C1—C11—N1	170.30 (11)
C5—C6—C7—C8	-0.5 (2)	C1—C11—N1—N2	175.68 (11)
C6—C7—C8—C9	0.0 (2)	O2—C12—N2—N1	172.35 (11)
C7—C8—C9—C10	0.8 (2)	N3—C12—N2—N1	-7.35 (18)
C8—C9—C10—C5	-1.1 (2)	C11—N1—N2—C12	171.46 (11)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1...N1	0.82	1.83	2.5563 (15)	146
N2—H2...O2 ⁱ	0.86	2.01	2.8385 (15)	163
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Fig. 1

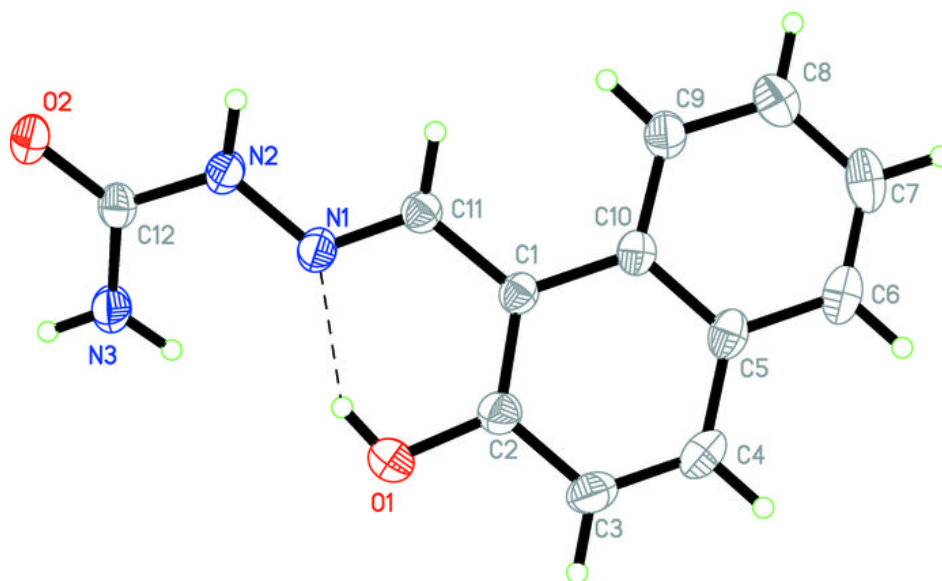


Fig. 2

