organic compounds

1-(Hydroxyiminomethyl)-2-naphthol

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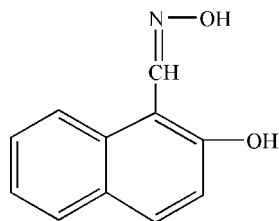Correspondence e-mail: lilianzhi1963@yahoo.com.cn

The title compound, $C_{11}H_9NO_2$, was prepared by a condensation reaction of 2-hydroxy-1-naphthaldehyde with hydroxylammonium chloride in refluxing ethanol. An intramolecular O—H···N hydrogen bond is observed. In the crystal structure, intermolecular O—H···O and C—H···O hydrogen-bond interactions result in a two-dimensional network.

Related literature

For general background, see: Desai *et al.* (2001); Hodnett *et al.* (1970).

Experimental

Crystal data

$C_{11}H_9NO_2$	b = 4.0462 (7) Å
M_r = 187.19	c = 16.527 (2) Å
Monoclinic, $P2_1/c$	β = 114.933 (2)°
a = 14.8382 (19) Å	V = 899.8 (2) Å^3

Z = 4	T = 298 (2) K
Mo $K\alpha$ radiation	$0.56 \times 0.45 \times 0.18$ mm
μ = 0.10 mm^{-1}	

Data collection

Bruker SMART 1K CCD diffractometer	3977 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1573 independent reflections
	1009 reflections with $I > 2\sigma(I)$
T_{min} = 0.953, T_{max} = 0.984	R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049	127 parameters
$wR(F^2)$ = 0.148	H-atom parameters constrained
S = 1.04	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
1573 reflections	$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N1	0.82	1.86	2.577 (2)	146
O1—H1···O2^i	0.82	1.97	2.771 (2)	164
C1—H1A···O1^{ii}	0.93	2.66	3.527 (3)	156
C8—H8···O1^{ii}	0.93	2.62	3.474 (3)	153

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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*.

References

Desai, S. B., Desai, P. B. & Desai, K. R. (2001). *Hetercycle Commun.* **7**, 83–90.
Hodnett, E. M. & Mooney, P. D. (1970). *J. Med. Chem.* **13**, 786–788.
Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supporting information

1-(Hydroxyiminomethyl)-2-naphthol

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S1. Comment

Schiff bases have been intensively investigated owing to their strong coordination capability and diverse biological activities, such as antibacterial, antitumor activities(Desai *et al.*, 2001; Hodnett *et al.*, 1970). We report here the synthesis and crystal structure of a new Schiff base compound derived from the condensation of 2-hydroxy-1-naphthaldehyde and hydroxylammonium chloride.

In the molecular structure(Scheme 1 and Fig.1), all the atoms are almost in one plane, with the C?N = 1.266 (3) Å. In the molecule, an intramolecular O2—H2···N1 hydrogen bond is observed(Table 1). The interactions of intermolecular hydrogen bond O1—H1···O2 form a one-dimensional chain-like structure(Fig. 2), which together with another two intermolecular H atoms C1—H1a···O1 and C8—H8···O1 (Table 1) result in the two-dimensional net-shaped structure.

S2. Experimental

2-hydroxy-1-naphthaldehyde (1 mmol, 172.18 mg) in absolute ethanol (5 ml) was added dropwise to a absolute ethanol solution (10 ml) of hydroxylammonium chloride (1 mmol, 69.49 mg). The mixture was heated under reflux with stirring for 2 h and then filtered. The resulting solution was held at room temperature for 14 days, whereupon the colourless needle crystals of the title complex suitable for X-ray diffraction analysis were obtained.
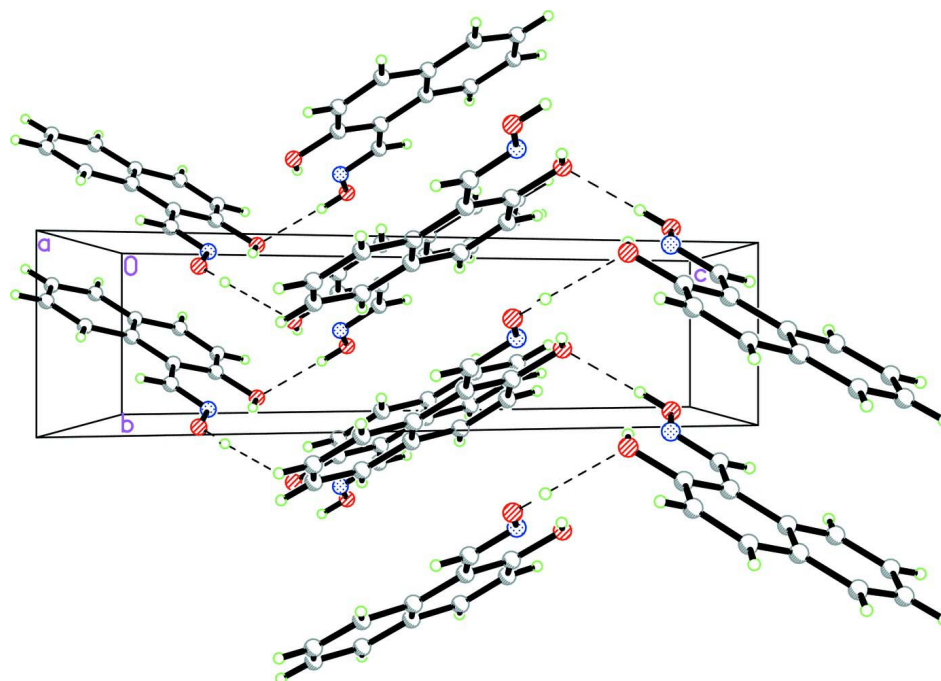
S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å (aromatic), 0.82 Å (hydroxyl) and $U_{iso}(H) = 1.2U_{eq}(C)$.
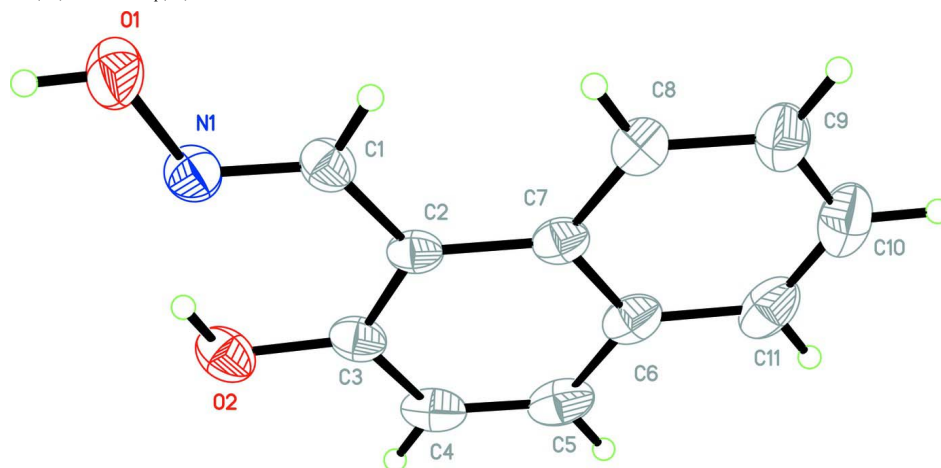
Figure 1

The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Figure 2

The crystal packing of the title complex, viewed approximately along the *a* axis.

1-(Hydroxyiminomethyl)-2-naphthol

Crystal data

$C_{11}H_9NO_2$	$F(000) = 392$
$M_r = 187.19$	$D_x = 1.382$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1237 reflections
$a = 14.8382$ (19) Å	$\theta = 2.5–27.0°$
$b = 4.0462$ (7) Å	$\mu = 0.10$ mm^{-1}
$c = 16.527$ (2) Å	$T = 298$ K
$\beta = 114.933$ (2)°	Block, yellow
$V = 899.8$ (2) Å^3	$0.56 \times 0.45 \times 0.18$ mm
$Z = 4$	

Data collection

Bruker SMART 1K CCD diffractometer	3977 measured reflections
Radiation source: fine-focus sealed tube	1573 independent reflections
Graphite monochromator	1009 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.053$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
$T_{min} = 0.953$, $T_{max} = 0.984$	$h = -17 \rightarrow 15$
	$k = -4 \rightarrow 4$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.049$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.149$	
$S = 1.04$	H-atom parameters constrained
1573 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 0.1603P]$ where $P = (F_o^2 + 2F_c^2)/3$
127 parameters	
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.19$ e Å^{-3}
	$\Delta\rho_{min} = -0.20$ 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}
N1	0.02725 (13)	0.4774 (6)	0.36227 (12)	0.0492 (6)
O1	−0.05788 (11)	0.5784 (5)	0.37113 (10)	0.0675 (6)
H1	−0.0944	0.6796	0.3262	0.101*
O2	0.14880 (11)	0.4181 (5)	0.28864 (9)	0.0576 (6)
H2	0.0978	0.4758	0.2925	0.086*
C1	0.08718 (16)	0.3186 (6)	0.42939 (14)	0.0450 (6)
H1A	0.0709	0.2805	0.4771	0.054*
C2	0.18092 (14)	0.1946 (6)	0.43332 (13)	0.0398 (6)
C3	0.20781 (15)	0.2491 (6)	0.36359 (14)	0.0447 (6)
C4	0.29816 (17)	0.1329 (7)	0.36695 (16)	0.0543 (7)
H4	0.3149	0.1746	0.3197	0.065*
C5	0.36087 (17)	−0.0385 (7)	0.43808 (18)	0.0574 (7)
H5	0.4197	−0.1189	0.4383	0.069*
C6	0.33969 (16)	−0.1004 (6)	0.51308 (16)	0.0498 (7)
C7	0.24800 (15)	0.0168 (6)	0.50985 (14)	0.0426 (6)
C8	0.22784 (17)	−0.0505 (7)	0.58514 (15)	0.0525 (7)
H8	0.1680	0.0189	0.5849	0.063*
C9	0.29445 (19)	−0.2143 (7)	0.65726 (17)	0.0627 (7)
H9	0.2795	−0.2534	0.7057	0.075*
C10	0.38420 (19)	−0.3245 (7)	0.66013 (19)	0.0707 (8)
H10	0.4294	−0.4333	0.7104	0.085*
C11	0.40583 (18)	−0.2721 (7)	0.58864 (19)	0.0634 (8)
H11	0.4653	−0.3514	0.5900	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0425 (10)	0.0617 (16)	0.0470 (11)	0.0002 (10)	0.0225 (9)	−0.0005 (10)
O1	0.0471 (9)	0.1046 (17)	0.0565 (10)	0.0234 (10)	0.0274 (8)	0.0178 (11)
O2	0.0556 (10)	0.0760 (14)	0.0459 (9)	−0.0074 (9)	0.0260 (8)	−0.0040 (9)
C1	0.0442 (13)	0.0525 (17)	0.0411 (11)	−0.0030 (12)	0.0207 (10)	−0.0005 (12)
C2	0.0390 (12)	0.0386 (15)	0.0440 (11)	−0.0084 (11)	0.0196 (10)	−0.0125 (11)
C3	0.0429 (12)	0.0469 (16)	0.0451 (12)	−0.0123 (12)	0.0195 (10)	−0.0131 (12)
C4	0.0516 (14)	0.060 (2)	0.0611 (15)	−0.0153 (13)	0.0333 (13)	−0.0238 (15)
C5	0.0419 (13)	0.0546 (19)	0.0820 (17)	−0.0088 (13)	0.0322 (13)	−0.0240 (16)
C6	0.0382 (12)	0.0392 (16)	0.0676 (15)	−0.0088 (11)	0.0180 (12)	−0.0152 (13)
C7	0.0398 (12)	0.0355 (15)	0.0515 (13)	−0.0087 (10)	0.0184 (10)	−0.0121 (12)
C8	0.0519 (14)	0.0489 (18)	0.0569 (14)	0.0029 (12)	0.0232 (12)	0.0007 (13)
C9	0.0660 (17)	0.054 (2)	0.0632 (15)	0.0036 (15)	0.0220 (14)	0.0115 (15)
C10	0.0581 (17)	0.054 (2)	0.0781 (18)	0.0009 (14)	0.0070 (15)	0.0103 (16)
C11	0.0430 (14)	0.0439 (18)	0.0911 (19)	0.0002 (13)	0.0165 (14)	−0.0073 (16)

Geometric parameters (Å, º)

N1—C1	1.266 (3)	C5—C6	1.423 (3)
N1—O1	1.393 (2)	C5—H5	0.9300
O1—H1	0.8200	C6—C11	1.404 (3)
O2—C3	1.362 (3)	C6—C7	1.420 (3)
O2—H2	0.8200	C7—C8	1.423 (3)
C1—C2	1.454 (3)	C8—C9	1.358 (3)
C1—H1A	0.9300	C8—H8	0.9300
C2—C3	1.387 (3)	C9—C10	1.386 (3)
C2—C7	1.431 (3)	C9—H9	0.9300
C3—C4	1.399 (3)	C10—C11	1.365 (4)
C4—C5	1.344 (3)	C10—H10	0.9300
C4—H4	0.9300	C11—H11	0.9300
C1—N1—O1	112.96 (16)	C11—C6—C7	119.9 (2)
N1—O1—H1	109.5	C11—C6—C5	122.2 (2)
C3—O2—H2	109.5	C7—C6—C5	117.9 (2)
N1—C1—C2	121.31 (19)	C6—C7—C8	117.0 (2)
N1—C1—H1A	119.3	C6—C7—C2	119.96 (19)
C2—C1—H1A	119.3	C8—C7—C2	123.0 (2)
C3—C2—C7	118.56 (19)	C9—C8—C7	121.2 (2)
C3—C2—C1	120.9 (2)	C9—C8—H8	119.4
C7—C2—C1	120.59 (18)	C7—C8—H8	119.4
O2—C3—C2	122.40 (19)	C8—C9—C10	121.4 (2)
O2—C3—C4	116.27 (19)	C8—C9—H9	119.3
C2—C3—C4	121.3 (2)	C10—C9—H9	119.3
C5—C4—C3	120.4 (2)	C11—C10—C9	119.6 (3)
C5—C4—H4	119.8	C11—C10—H10	120.2
C3—C4—H4	119.8	C9—C10—H10	120.2

C4—C5—C6	121.8 (2)	C10—C11—C6	121.0 (2)
C4—C5—H5	119.1	C10—C11—H11	119.5
C6—C5—H5	119.1	C6—C11—H11	119.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1	0.82	1.86	2.577 (2)	146
O1—H1···O2[i]	0.82	1.97	2.771 (2)	164
C1—H1*A*···O1[ii]	0.93	2.66	3.527 (3)	156
C8—H8···O1[ii]	0.93	2.62	3.474 (3)	153

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