data reports

CrossMark

Crystal structure of 1-((1E)-{(E)-2-[(2-hydroxynaphthalen-1-yl)methylidene]-hydrazin-1-ylidene}methyl)naphthalen-2-ol

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The complete molecule of the title compound, $C_{22}H_{16}N_2O_2$, is generated by a crystallographic inversion centre at the mid-point of the central N—N bond. Two intramolecular O—H···N hydrogen bonds occur.

1. Related literature

For general background to Schiff base derivatives, see: Hoshino (1998); Kalaivani et al. (2013); Vijayan et al. (2014).
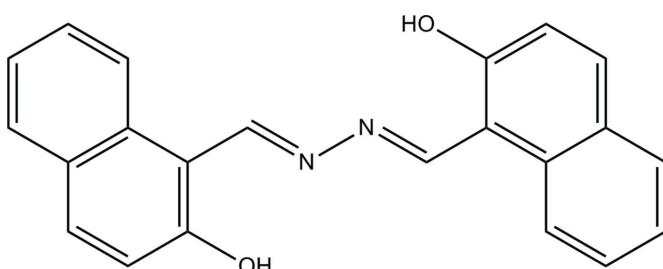
2. Experimental

2.1. Crystal data

$C_{22}H_{16}N_2O_2$
$M_r = 340.37$
Monoclinic, $P2_1/n$
$a = 8.5680 (7)$ Å
$b = 6.1020 (5)$ Å
$c = 15.9870 (6)$ Å
$\beta = 91.191 (5)°$

$V = 835.65 (10)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 293$ K
$0.22 \times 0.20 \times 0.18$ mm

2.2. Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2008)
$T_{min} = 0.981$, $T_{max} = 0.984$

1907 measured reflections
1907 independent reflections
1859 reflections with $I > 2\sigma(I)$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.007$
$wR(F^2) = 0.019$
$S = 1.03$
1907 reflections
122 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.02$ e Å^{-3}
$\Delta\rho_{min} = -0.08$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.98	1.67 (1)	2.5671 (3)	151 (1)

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; 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, 2012); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

Acknowledgements

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Crystal structure of 1-((1*E*)-{(*E*)-2-[(2-hydroxynaphthalen-1-yl)methylidene]hydrazin-1-ylidene}methyl)naphthalen-2-ol

Paranthaman Vijayan, Periasamy Viswanathamurthi, Michel Fleck, Sugumar Paramasivam and Ponnuswamy Mondikalipudur Nanjappagounder

S1. Comment

Schiff bases are important ligands, as moderate electron donors with a chelating structure and control the behaviour of metal ions in a diverse range of applications (Hoshino, 1998). Dithiocarbazate compounds are an important class of Schiff bases which can be easily obtained by condensation of dithiocarbazides with aldehydes or ketones. In particular hydrazone containing naphthalene ring compounds have drawn much attention because of their biological activities such as DNA/BSA binding affinities and anticancer activities *in vitro* (Kalaivani *et al.*, 2013; Vijayan *et al.*, 2014).

The *ORTEP* plot of the molecule is shown in Fig.1. The title compound (I), crystallized in the monoclinic spacegroup *P*2$_1$/*n* with half molecule in the asymmetric unit. Pair of molecules related by an crystallographic inversion centre generate another half of the molecule.

S2. Experimental

The title compound was obtained by the reaction of *S*-benzyldithiocarbazate and 2-Hydroxy-1-napthaldehyde in boiling ethanol. The unexpected formation of the hydrazone was probably due to the decomposition of *S*-benzyldithiocarbazate in solution resulting in the formation of hydrazine, which then reacted with 2-hydroxy-1-napthaldehyde to form the corresponding hydrazone. The dithiocarbazates are known to decompose on heating.

S3. Refinement

All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located geometrically (aromatic C—H 0.95 Å, secondary alkane C—H 0.99 Å, tertiary alkane C—H 1.0 Å) and refined using a riding model with the isotropic displacement parameters fixed at U_{iso}= 1.2 times U_{eq} of the parent carbon for all of the hydrogen atoms.
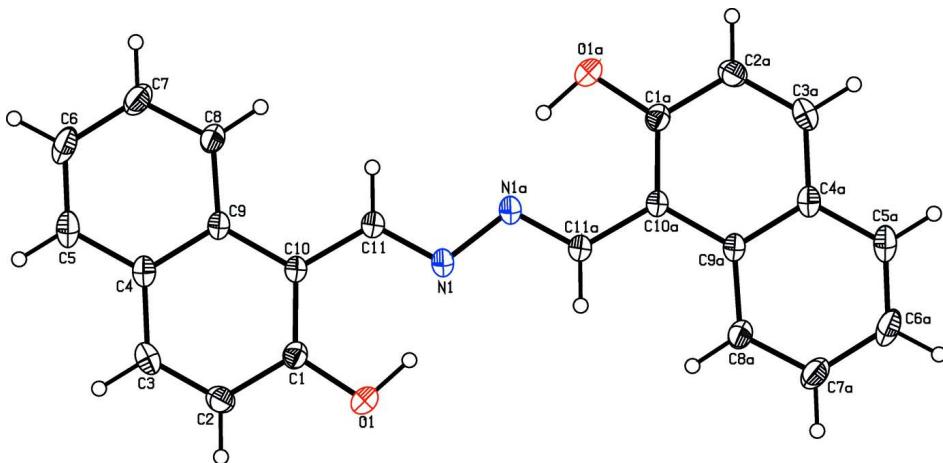
Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at 50% probability level.

1-((1*E*)-{(*E*)-2-[(2-Hydroxynaphthalen-1-yl)methylidene]hydrazin-1-ylidene}methyl)naphthalen-2-ol

Crystal data
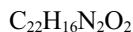
$C_{22}H_{16}N_2O_2$	$F(000) = 356$
$M_r = 340.37$	$D_x = 1.353$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1859 reflections
$a = 8.5680$ (7) Å	$\theta = 2.6–27.5°$
$b = 6.1020$ (5) Å	$\mu = 0.09$ mm^{-1}
$c = 15.9870$ (6) Å	$T = 293$ K
$\beta = 91.191$ (5)°	Block, yellow
$V = 835.65$ (10) Å^3	$0.22 \times 0.20 \times 0.18$ mm
$Z = 2$	

Data collection

Bruker SMART APEXII CCD diffractometer	1907 measured reflections
Radiation source: fine-focus sealed tube	1907 independent reflections
Graphite monochromator	1859 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.000$
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)	$\theta_{max} = 27.5°$, $\theta_{min} = 2.6°$
	$h = -11 \rightarrow 11$
	$k = 0 \rightarrow 7$
$T_{min} = 0.981$, $T_{max} = 0.984$	$l = 0 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.007$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.019$	
$S = 1.03$	H atoms treated by a mixture of independent and constrained refinement
1907 reflections	
122 parameters	$w = 1/[\sigma^2(F_o^2) + (0.008P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
1 restraint	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.02$ e Å^{-3}
	$\Delta\rho_{min} = -0.08$ 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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.37033 (2)	0.02490 (3)	0.395255 (11)	0.03120 (4)
C2	0.32482 (2)	−0.16485 (3)	0.350605 (13)	0.03653 (5)
H2	0.2610	−0.2683	0.3755	0.044*
C3	0.37391 (2)	−0.19647 (3)	0.271551 (13)	0.03672 (5)
H3	0.3450	−0.3240	0.2434	0.044*
C4	0.46786 (2)	−0.04107 (3)	0.230675 (12)	0.03129 (5)
C5	0.51123 (3)	−0.06977 (4)	0.146509 (13)	0.04195 (5)
H5	0.4815	−0.1967	0.1182	0.050*
C6	0.59566 (3)	0.08510 (5)	0.106276 (13)	0.04842 (6)
H6	0.6213	0.0654	0.0505	0.058*
C7	0.64395 (3)	0.27462 (5)	0.149184 (13)	0.04576 (6)
H7	0.7021	0.3802	0.1217	0.055*
C8	0.60616 (2)	0.30547 (4)	0.231137 (12)	0.03661 (5)
H8	0.6408	0.4310	0.2588	0.044*
C9	0.51573 (2)	0.15099 (3)	0.274652 (11)	0.02726 (4)
C10	0.46705 (2)	0.18089 (3)	0.359484 (10)	0.02658 (4)
C11	0.51614 (2)	0.37178 (3)	0.406697 (11)	0.02991 (4)
H11	0.5827	0.4727	0.3824	0.036*
N1	0.46961 (2)	0.40510 (3)	0.481933 (10)	0.03414 (4)
O1	0.31470 (2)	0.04548 (3)	0.472921 (9)	0.04471 (5)
H1	0.3596 (5)	0.1824 (7)	0.4943 (2)	0.0995 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.03470 (9)	0.03336 (10)	0.02553 (9)	0.00099 (8)	0.00052 (7)	−0.00058 (7)
C2	0.03940 (11)	0.03200 (10)	0.03805 (11)	−0.00574 (8)	−0.00259 (8)	0.00167 (8)
C3	0.03929 (11)	0.03106 (10)	0.03943 (11)	0.00132 (8)	−0.00822 (8)	−0.00859 (8)
C4	0.02942 (9)	0.03540 (10)	0.02889 (9)	0.00595 (8)	−0.00317 (7)	−0.00776 (8)
C5	0.04097 (11)	0.05126 (13)	0.03346 (11)	0.00604 (10)	−0.00287 (8)	−0.01694 (9)
C6	0.04610 (12)	0.07303 (17)	0.02636 (10)	0.00235 (12)	0.00672 (9)	−0.01219 (10)
C7	0.04615 (12)	0.06073 (15)	0.03086 (10)	−0.00308 (11)	0.01184 (9)	−0.00236 (10)
C8	0.03867 (10)	0.04315 (12)	0.02823 (10)	−0.00353 (9)	0.00582 (8)	−0.00574 (8)
C9	0.02591 (8)	0.03176 (9)	0.02405 (8)	0.00337 (7)	−0.00108 (6)	−0.00519 (7)
C10	0.02861 (9)	0.02830 (9)	0.02281 (8)	0.00107 (7)	−0.00030 (6)	−0.00319 (7)
C11	0.03341 (9)	0.03091 (10)	0.02545 (9)	−0.00130 (7)	0.00150 (7)	−0.00289 (7)

N1	0.04353 (9)	0.03257 (9)	0.02637 (8)	−0.00328 (7)	0.00204 (7)	−0.00689 (7)
O1	0.05713 (10)	0.04698 (10)	0.03052 (8)	−0.01041 (8)	0.01283 (7)	−0.00151 (7)

Geometric parameters (Å, º)

C1—O1	1.3451 (2)	C6—H6	0.9300
C1—C10	1.3927 (3)	C7—C8	1.3691 (3)
C1—C2	1.4109 (3)	C7—H7	0.9300
C2—C3	1.3541 (3)	C8—C9	1.4125 (3)
C2—H2	0.9300	C8—H8	0.9300
C3—C4	1.4129 (3)	C9—C10	1.4388 (2)
C3—H3	0.9300	C10—C11	1.4458 (3)
C4—C5	1.4142 (3)	C11—N1	1.2911 (2)
C4—C9	1.4226 (3)	C11—H11	0.9300
C5—C6	1.3600 (4)	N1—N1^{i}	1.3906 (3)
C5—H5	0.9300	O1—H1	0.978 (4)
C6—C7	1.4026 (4)		

O1—C1—C10	122.721 (18)	C8—C7—C6	120.54 (2)
O1—C1—C2	116.367 (18)	C8—C7—H7	119.7
C10—C1—C2	120.911 (18)	C6—C7—H7	119.7
C3—C2—C1	120.079 (19)	C7—C8—C9	121.51 (2)
C3—C2—H2	120.0	C7—C8—H8	119.2
C1—C2—H2	120.0	C9—C8—H8	119.2
C2—C3—C4	121.809 (19)	C8—C9—C4	117.502 (17)
C2—C3—H3	119.1	C8—C9—C10	123.528 (18)
C4—C3—H3	119.1	C4—C9—C10	118.951 (18)
C3—C4—C5	121.360 (19)	C1—C10—C9	119.145 (17)
C3—C4—C9	119.045 (17)	C1—C10—C11	120.359 (17)
C5—C4—C9	119.570 (19)	C9—C10—C11	120.491 (17)
C6—C5—C4	121.12 (2)	N1—C11—C10	121.408 (18)
C6—C5—H5	119.4	N1—C11—H11	119.3
C4—C5—H5	119.4	C10—C11—H11	119.3
C5—C6—C7	119.739 (19)	C11—N1—N1^{i}	113.45 (2)
C5—C6—H6	120.1	C1—O1—H1	104.9 (2)
C7—C6—H6	120.1		

O1—C1—C2—C3	179.043 (18)	C3—C4—C9—C10	0.06 (3)
C10—C1—C2—C3	−0.64 (3)	C5—C4—C9—C10	178.266 (17)
C1—C2—C3—C4	−1.51 (3)	O1—C1—C10—C9	−177.234 (17)
C2—C3—C4—C5	−176.393 (19)	C2—C1—C10—C9	2.43 (3)
C2—C3—C4—C9	1.78 (3)	O1—C1—C10—C11	1.96 (3)
C3—C4—C5—C6	176.95 (2)	C2—C1—C10—C11	−178.377 (17)
C9—C4—C5—C6	−1.21 (3)	C8—C9—C10—C1	176.247 (18)
C4—C5—C6—C7	1.46 (4)	C4—C9—C10—C1	−2.11 (3)
C5—C6—C7—C8	−0.31 (4)	C8—C9—C10—C11	−2.94 (3)
C6—C7—C8—C9	−1.12 (4)	C4—C9—C10—C11	178.703 (16)
C7—C8—C9—C4	1.33 (3)	C1—C10—C11—N1	−1.45 (3)

C7—C8—C9—C10	−177.044 (19)	C9—C10—C11—N1	177.730 (17)
C3—C4—C9—C8	−178.391 (18)	C10—C11—N1—N1[i]	179.073 (19)
C5—C4—C9—C8	−0.19 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.98	1.67 (1)	2.5671 (3)	151 (1)