Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Hydroxy-*N*′-(phenylacetyl)-naphthalene-2-carbohydrazide

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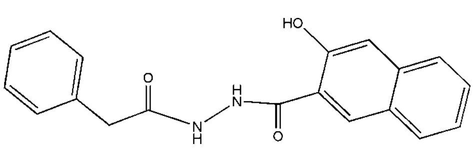
Received 23 September 2007; accepted 12 November 2007

Key indicators: single-crystal X-ray study; T = 78 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.057; wR factor = 0.184; data-to-parameter ratio = 11.7.

In the title compound $C_{19}H_{16}N_2O_3$, the molecules are packed *via* π–π stacking interactions [mean interplanar distances 3.445 (2) and 3.499 (2) Å] and hydrogen bonds, including one intramolecular N—H···O hydrogen bond..

Related literature

For related literature, see: Lah & Pecoraro (1989); Mezei *et al.* (2004); Moon *et al.* (2000); Pereira *et al.* (2004).



Experimental

Crystal data

$C_{19}H_{16}N_2O_3$
M_r = 320.34
Monoclinic, $P2_1/c$
a = 8.8398 (18) Å
b = 19.308 (4) Å
c = 9.7463 (19) Å
β = 103.80 (3)°
V = 1615.4 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 78 (2) K
0.44 × 0.33 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*XSCANS*; Siemens, 1996)
T_{min} = 0.962, T_{max} = 0.988
12085 measured reflections
2804 independent reflections
2184 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.184
S = 1.08
2804 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.86	1.84	2.559 (3)	140
O2—H2···O3^{i}	0.82	1.82	2.615 (2)	163
N2—H2*C*···O1^{ii}	0.86	1.95	2.789 (2)	165

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

This project was supported by the Talent Fund of Ningbo University (grant No. 2006668).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2037).

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 doi:10.1107/S1600536807058199

supplementary materials

Acta Cryst. (2007). E**63**, o4769 [**doi:10.1107/S1600536807058199**]

3-Hydroxy-*N*'-(phenylacetyl)naphthalene-2-carbohydrazide

K.-W. Lei and **H.-M. Feng**

Comment

Metallamacrocycles have become important in recent years because of their interesting molecular architecture, multinuclear structures and magnetic properties (Mezei *et al.*, 2004; Pereira *et al.*, 2004; Lah & Pecoraro, 1989). They have also been used as building blocks for the construction of two- or three-dimensional network structures (Moon *et al.*, 2000). A variety of metallamacrocycles and cages were found to form interesting host–guest systems with different metal ions of varying coordination and symmetry. We now report the structure of a designed pentadentate ligand, 3-hydroxy-*N*-phenylacetyl-2-naphthalenecarbohydrazide (I).

The molecular structure of (I), $C_{19}H_{16}N_2O_3$, is illustrated in Fig.1. Atom O1, O2, N1 and N2 are nearly coplanar with the naphthalene plane. The O3 atomic deviation is 0.838 (2) Å from naphthalene plane. The C15, C16, C17 atoms are disorder. C14 has a smaller adp than the other atoms in the disordered phenyl group because it is bonded directly to C19, which is not disordered, and thus has less freedom of movement. The larger than normal range of thermal motion is mostly due to the difference between the disordered group and the other atoms which are not disordered.

The mean interplanar distance of 3.445 (2)Å and 3.499 (2)Å alternately between naphthalene rings suggests that the ligands are engaged in π-π stacking interactions with a offset face-to-face style. The crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds (Table 1 and Fig.2).

Experimental

Phenylacetic anhydride (8.32 g, 66.8 mmol) and 3-Hydroxy-2-naphthalenecarbohydrazide (11.3 g, 56.0 mmol) were added to 120 ml of chloroform with an external ice-water bath. The reaction mixture was slowly warmed to room temperature and stirred for 8 h. After leaving overnight in a refrigerator, the resulting white precipitate was filtered and rinsed with chloroform and diethyl ether. Yield: 87.2%. Melting point: 220–225 °C. Calcd. for $C_{19}H_{16}N_2O_3$: C, 71.24; H, 5.03; N, 8.74; Found: C, 71.56; H, 5.12; N, 8.42%.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms (C—H = 0.93 Å; N—H = 0.86 Å; O—H = 0.82 Å) and U_{iso}(H) values were taken to be equal to 1.2 U_{eq}(C, N) and 1.5 U_{eq}(O).

Figures

Fig. 1. The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

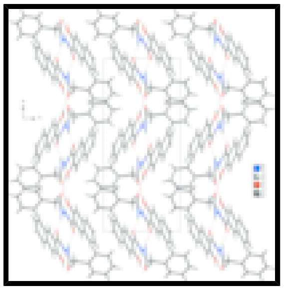

Fig. 2. A view of stacking of (I). C15', C16', C17' atoms have been omitted.

3-Hydroxy-*N*'-(phenylacetyl)naphthalene-2-carbohydrazide

Crystal data

$C_{19}H_{16}N_2O_3$	F_{000} = 672.0
M_r = 320.34	D_x = 1.317 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 220-225 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.8398 (18) Å	Cell parameters from 25 reflections
b = 19.308 (4) Å	θ = 5.0–12.5°
c = 9.7463 (19) Å	μ = 0.09 mm^{-1}
β = 103.80 (3)°	T = 78 (2) K
V = 1615.4 (6) Å^3	Block, pale yellow
Z = 4	0.44 × 0.33 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer	2804 independent reflections
Radiation source: fine-focus sealed tube	2184 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
Detector resolution: 0 pixels mm^{-1}	θ_{max} = 25.0°
T = 78(2) K	θ_{min} = 3.0°
ω scans	h = −9→10
Absorption correction: multi-scan (XSCANS; Siemens, 1996)	k = −22→22
T_{min} = 0.962, T_{max} = 0.988	l = −11→11
12085 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	H-atom parameters constrained
$wR(F^2)$ = 0.184	$w = 1/[\sigma^2(F_o^2) + (0.1053P)^2 + 0.6523P]$ where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.08$	$(\Delta/\sigma)_{max} = 0.001$
2804 reflections	$\Delta\rho_{max} = 0.48$ e Å^{-3}
239 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
N1	0.5095 (2)	0.39538 (10)	0.3349 (2)	0.0419 (5)	
H1	0.4895	0.3608	0.2779	0.050*	
N2	0.4632 (2)	0.39254 (9)	0.46034 (19)	0.0390 (5)	
H2C	0.4526	0.4296	0.5060	0.047*	
O1	0.6141 (2)	0.50060 (8)	0.37634 (17)	0.0481 (5)	
O2	0.5286 (2)	0.33028 (9)	0.1109 (2)	0.0609 (6)	
H2	0.5208	0.2995	0.0518	0.091*	
O3	0.46016 (19)	0.27754 (8)	0.44536 (17)	0.0446 (5)	
C1	0.5840 (2)	0.44975 (10)	0.2991 (2)	0.0345 (5)	
C2	0.7037 (2)	0.50080 (11)	0.1212 (2)	0.0366 (5)	
H2A	0.7208	0.5395	0.1797	0.044*	
C3	0.6299 (2)	0.44470 (10)	0.1616 (2)	0.0341 (5)	
C4	0.6040 (3)	0.38550 (11)	0.0713 (2)	0.0393 (5)	
C5	0.6550 (3)	0.38516 (12)	−0.0500 (2)	0.0423 (6)	
H5	0.6394	0.3458	−0.1067	0.051*	
C6	0.7309 (3)	0.44290 (12)	−0.0918 (2)	0.0399 (5)	
C7	0.7546 (3)	0.50251 (11)	−0.0047 (2)	0.0384 (5)	
C8	0.8314 (3)	0.56025 (13)	−0.0456 (3)	0.0492 (6)	
H8	0.8474	0.5996	0.0110	0.059*	
C9	0.8821 (3)	0.55901 (15)	−0.1669 (3)	0.0586 (7)	
H9	0.9330	0.5973	−0.1926	0.070*	
C10	0.8579 (3)	0.50031 (16)	−0.2527 (3)	0.0604 (8)	
H10	0.8925	0.5001	−0.3356	0.072*	
C11	0.7849 (3)	0.44361 (15)	−0.2178 (3)	0.0523 (7)	
H11	0.7702	0.4050	−0.2765	0.063*	
C12	0.4357 (2)	0.33061 (11)	0.5078 (2)	0.0343 (5)	
C13	0.3679 (3)	0.32965 (12)	0.6357 (2)	0.0395 (5)	

H13A	0.3892	0.3734	0.6853	0.047*	
H13B	0.4169	0.2932	0.6994	0.047*	
C14	0.1945 (3)	0.31770 (13)	0.5932 (3)	0.0492 (6)	
C15	0.1310 (13)	0.2652 (7)	0.656 (2)	0.085 (4)	0.49 (2)
H15	0.1953	0.2365	0.7218	0.102*	0.49 (2)
C16	−0.0300 (11)	0.2551 (6)	0.623 (2)	0.092 (4)	0.49 (2)
H16	−0.0728	0.2186	0.6627	0.111*	0.49 (2)
C17	−0.122 (3)	0.2977 (16)	0.533 (2)	0.085 (4)	0.49 (2)
H17	−0.2270	0.2869	0.4994	0.102*	0.49 (2)
C15'	0.1330 (12)	0.2527 (6)	0.5770 (19)	0.079 (3)	0.51 (2)
H15'	0.1983	0.2145	0.6006	0.095*	0.51 (2)
C16'	−0.0247 (10)	0.2427 (5)	0.526 (2)	0.090 (4)	0.51 (2)
H16'	−0.0661	0.1985	0.5272	0.108*	0.51 (2)
C17'	−0.121 (3)	0.2964 (16)	0.474 (2)	0.085 (4)	0.51 (2)
H17'	−0.2255	0.2888	0.4299	0.102*	0.51 (2)
C18	−0.0619 (4)	0.3598 (2)	0.4874 (5)	0.0882 (11)	
H18	−0.1258	0.3914	0.4287	0.106*	
C19	0.0962 (4)	0.37045 (18)	0.5351 (4)	0.0761 (10)	
H19	0.1370	0.4143	0.5276	0.091*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0598 (12)	0.0308 (10)	0.0409 (10)	−0.0084 (8)	0.0235 (9)	−0.0064 (8)
N2	0.0550 (11)	0.0267 (10)	0.0406 (10)	−0.0036 (8)	0.0218 (9)	−0.0034 (8)
O1	0.0725 (12)	0.0321 (9)	0.0472 (9)	−0.0114 (7)	0.0291 (9)	−0.0117 (7)
O2	0.0968 (15)	0.0368 (10)	0.0604 (12)	−0.0257 (9)	0.0409 (11)	−0.0186 (8)
O3	0.0620 (11)	0.0267 (8)	0.0492 (10)	0.0052 (7)	0.0215 (8)	0.0017 (7)
C1	0.0374 (11)	0.0256 (11)	0.0403 (12)	0.0022 (8)	0.0090 (9)	−0.0007 (9)
C2	0.0396 (11)	0.0297 (11)	0.0404 (12)	0.0011 (8)	0.0089 (9)	−0.0014 (9)
C3	0.0359 (11)	0.0288 (11)	0.0381 (12)	0.0013 (8)	0.0098 (9)	−0.0020 (9)
C4	0.0460 (12)	0.0300 (11)	0.0429 (12)	−0.0031 (9)	0.0126 (10)	−0.0041 (9)
C5	0.0499 (13)	0.0371 (12)	0.0398 (12)	−0.0004 (10)	0.0106 (10)	−0.0089 (10)
C6	0.0363 (11)	0.0468 (13)	0.0357 (12)	0.0051 (10)	0.0071 (9)	0.0016 (10)
C7	0.0370 (11)	0.0378 (12)	0.0401 (12)	0.0039 (9)	0.0087 (9)	0.0061 (10)
C8	0.0521 (14)	0.0424 (14)	0.0555 (15)	−0.0019 (11)	0.0173 (12)	0.0063 (11)
C9	0.0632 (16)	0.0555 (17)	0.0626 (17)	−0.0011 (13)	0.0263 (14)	0.0164 (14)
C10	0.0658 (17)	0.074 (2)	0.0479 (15)	0.0045 (14)	0.0257 (13)	0.0119 (14)
C11	0.0562 (15)	0.0627 (17)	0.0400 (13)	0.0023 (12)	0.0153 (12)	−0.0014 (12)
C12	0.0345 (11)	0.0285 (11)	0.0386 (11)	0.0011 (8)	0.0060 (9)	0.0027 (9)
C13	0.0458 (13)	0.0322 (12)	0.0413 (12)	−0.0006 (9)	0.0120 (10)	0.0046 (9)
C14	0.0442 (13)	0.0414 (13)	0.0662 (17)	0.0012 (10)	0.0214 (12)	0.0024 (12)
C15	0.059 (5)	0.077 (7)	0.116 (10)	−0.006 (4)	0.014 (6)	0.028 (7)
C16	0.068 (5)	0.080 (6)	0.127 (12)	−0.015 (4)	0.020 (6)	0.022 (7)
C17	0.052 (2)	0.083 (3)	0.117 (13)	−0.001 (2)	0.011 (9)	−0.003 (11)
C15'	0.052 (4)	0.059 (4)	0.123 (10)	−0.002 (3)	0.015 (6)	0.002 (6)
C16'	0.061 (4)	0.074 (5)	0.131 (11)	−0.013 (4)	0.010 (6)	0.002 (6)
C17'	0.052 (2)	0.083 (3)	0.117 (13)	−0.001 (2)	0.011 (9)	−0.003 (11)

C18	0.059 (2)	0.091 (3)	0.110 (3)	0.0146 (19)	0.0119 (19)	0.017 (2)
C19	0.0599 (18)	0.071 (2)	0.093 (2)	0.0043 (15)	0.0095 (17)	0.0238 (18)

Geometric parameters (Å, °)

N1—C1	1.329 (3)	C10—H10	0.9300
N1—N2	1.380 (3)	C11—H11	0.9300
N1—H1	0.8600	C12—C13	1.506 (3)
N2—C12	1.325 (3)	C13—C14	1.507 (3)
N2—H2C	0.8600	C13—H13A	0.9700
O1—C1	1.227 (2)	C13—H13B	0.9700
O2—C4	1.361 (3)	C14—C15'	1.363 (11)
O2—H2	0.8200	C14—C19	1.371 (4)
O3—C12	1.237 (3)	C14—C15	1.373 (12)
C1—C3	1.493 (3)	C15—C16	1.396 (15)
C2—C3	1.370 (3)	C15—H15	0.9300
C2—C7	1.405 (3)	C16—C17	1.33 (3)
C2—H2A	0.9300	C16—H16	0.9300
C3—C4	1.428 (3)	C17—C18	1.42 (3)
C4—C5	1.362 (3)	C17—H17	0.9300
C5—C6	1.410 (3)	C15'—C16'	1.377 (14)
C5—H5	0.9300	C15'—H15'	0.9300
C6—C7	1.416 (3)	C16'—C17'	1.36 (3)
C6—C11	1.420 (3)	C16'—H16'	0.9300
C7—C8	1.411 (3)	C17'—C18	1.33 (3)
C8—C9	1.361 (4)	C17'—H17'	0.9300
C8—H8	0.9300	C18—C19	1.379 (5)
C9—C10	1.394 (4)	C18—H18	0.9300
C9—H9	0.9300	C19—H19	0.9300
C10—C11	1.354 (4)		
C1—N1—N2	122.28 (18)	O3—C12—N2	120.5 (2)
C1—N1—H1	118.9	O3—C12—C13	123.22 (19)
N2—N1—H1	118.9	N2—C12—C13	116.25 (19)
C12—N2—N1	117.59 (18)	C12—C13—C14	110.71 (19)
C12—N2—H2C	121.2	C12—C13—H13A	109.5
N1—N2—H2C	121.2	C14—C13—H13A	109.5
C4—O2—H2	109.5	C12—C13—H13B	109.5
O1—C1—N1	121.3 (2)	C14—C13—H13B	109.5
O1—C1—C3	122.47 (19)	H13A—C13—H13B	108.1
N1—C1—C3	116.21 (18)	C15'—C14—C19	116.2 (5)
C3—C2—C7	122.9 (2)	C19—C14—C15	116.6 (6)
C3—C2—H2A	118.5	C15'—C14—C13	121.6 (5)
C7—C2—H2A	118.5	C19—C14—C13	120.2 (2)
C2—C3—C4	118.3 (2)	C15—C14—C13	119.2 (5)
C2—C3—C1	117.16 (19)	C14—C15—C16	120.0 (10)
C4—C3—C1	124.58 (19)	C14—C15—H15	120.0
O2—C4—C5	121.9 (2)	C16—C15—H15	120.0
O2—C4—C3	118.0 (2)	C17—C16—C15	120.0 (15)
C5—C4—C3	120.1 (2)	C17—C16—H16	120.0

C4—C5—C6	121.7 (2)	C15—C16—H16	120.0
C4—C5—H5	119.1	C16—C17—C18	121 (2)
C6—C5—H5	119.1	C16—C17—H17	119.7
C5—C6—C7	118.8 (2)	C18—C17—H17	119.7
C5—C6—C11	122.6 (2)	C14—C15'—C16'	120.8 (9)
C7—C6—C11	118.6 (2)	C14—C15'—H15'	119.6
C2—C7—C8	122.8 (2)	C16'—C15'—H15'	119.6
C2—C7—C6	118.2 (2)	C17'—C16'—C15'	121.3 (14)
C8—C7—C6	119.0 (2)	C17'—C16'—H16'	119.3
C9—C8—C7	120.8 (2)	C15'—C16'—H16'	119.3
C9—C8—H8	119.6	C18—C17'—C16'	118 (2)
C7—C8—H8	119.6	C18—C17'—H17'	121.1
C8—C9—C10	120.0 (3)	C16'—C17'—H17'	121.1
C8—C9—H9	120.0	C17'—C18—C19	121.1 (12)
C10—C9—H9	120.0	C19—C18—C17	116.4 (11)
C11—C10—C9	121.4 (3)	C17'—C18—H18	112.2
C11—C10—H10	119.3	C19—C18—H18	121.8
C9—C10—H10	119.3	C17—C18—H18	121.8
C10—C11—C6	120.2 (3)	C14—C19—C18	121.3 (3)
C10—C11—H11	119.9	C14—C19—H19	119.3
C6—C11—H11	119.9	C18—C19—H19	119.3

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	1.84	2.559 (3)	140
O2—H2···O3^{i}	0.82	1.82	2.615 (2)	163
N2—H2C···O1^{ii}	0.86	1.95	2.789 (2)	165

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

Fig. 1

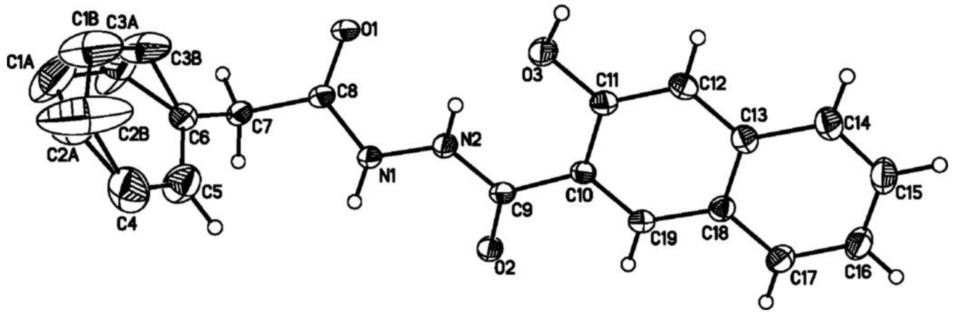
C1B
C3A
C1A
C3B
O1
O3
C11
C12
C13
C14
C15
C8
C7
C6
C2B
C2A
N2
N1
C9
C10
C19
C18
C17
C16
C5
C4
O2

Fig. 2