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3-Hydroxy-*N*′-phenylnaphthalene-2-carbohydrazide

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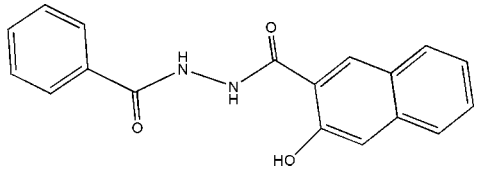
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.117; data-to-parameter ratio = 9.0.

The dihedral angle between the naphthalene and benzene planes in the title compound, $C_{18}H_{14}N_2O_3$, is 4.3 (2)°. The molecular conformation is characterized by an N—H···O hydrogen bond and the crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds.

Related literature

For related literature, see: Alexiou *et al.* (2002); Gaynor *et al.* (2002); Lah & Pecoraro (1989); Lehaire *et al.* (2002); Liu *et al.* (2001); Saalfrank *et al.* (2001).



Experimental

Crystal data

$C_{18}H_{14}N_2O_3$
M_r = 306.31
Monoclinic, $P2_1/c$
a = 4.8473 (10) Å
b = 33.907 (7) Å
c = 8.9148 (18) Å
β = 91.61 (3)°
V = 1464.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 273 (2) K
0.57 × 0.39 × 0.31 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.936, T_{max} = 0.976
7743 measured reflections
3401 independent reflections
2414 reflections with $I > 2\sigma(I)$
R_{int} = 0.069

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.117
S = 1.03
3401 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.12	2.864 (3)	145
N2—H2A···O3	0.86	1.88	2.582 (3)	138
O3—H3A···O2^{ii}	0.82	1.79	2.613 (3)	177

Symmetry codes: (i) $x-1, y, z$; (ii) $x+1, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2506).

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supplementary materials

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3-Hydroxy-*N*'-phenylnaphthalene-2-carbohydrazide

K.-W. Lei, H.-M. Feng and F.-Q. Li

Comment

Metallacrowns are a new class of metallamacrocycles, which have gained increasing attention over the past decade because of their unique properties (Alexiou *et al.*, 2002; Gaynor *et al.*, 2002; Lah & Pecoraro, 1989; Lehaire *et al.*, 2002; Liu *et al.*, 2001; Saalfrank *et al.*, 2001). These metallacrowns exhibit selective recognition of cations and anions (Saalfrank *et al.*, 2001; Lehaire *et al.*, 2002), can display intramolecular magnetic exchange interactions (Liu *et al.*, 2001), and can be used as building blocks for twodimensional or three-dimensional network structures (Gaynor *et al.*, 2002; Lah & Pecoraro, 1989; Lehaire *et al.*, 2002). The ability to control the generation of metallacrowns with different nuclear numbers, desired structures, and properties is still a substantial challenge. We now report structure of a designed pentadentate ligand.

The molecular structure of the title compound is illustrated in Fig.1. The bond lengths and bond angles are within normal ranges. The dihedral angel between the naphthalene and benzene planes is 4.3 (2)°. The molecular conformation is characterized by an N—H···O hydrogen bond and the crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds.

Experimental

Benzoic acid anhydride (13.56 g, 60.0 mmol) and 3-hydroxynaphthalene-2-carbohydrazide (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 off and rinsed with chloroform and diethyl ether (yield: 95.3%; m.p. 492–496 K). Calculatedd for $C_{18}H_{14}N_2O_3$: C 70.58, H 4.61, N 9.15%; found: C 70.24, H 4.75, N 9.02%.

Refinement

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

Figures

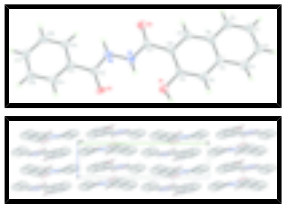

Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

3-Hydroxy-*N*'-phenylnaphthalene-2-carbohydrazide

Crystal data

$C_{18}H_{14}N_2O_3$	F_{000} = 640
M_r = 306.31	D_x = 1.389 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 219-223 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 4.8473 (10) Å	Cell parameters from 8325 reflections
b = 33.907 (7) Å	θ = 6.0–27.6º
c = 8.9148 (18) Å	μ = 0.10 mm^{-1}
β = 91.61 (3)º	T = 273 (2) K
V = 1464.6 (5) Å^3	Block, colourless
Z = 4	0.57 × 0.39 × 0.31 mm

Data collection

Rigaku R-AXIS RAPID diffractometer	3401 independent reflections
Radiation source: fine-focus sealed tube	2414 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.069
Detector resolution: 0 pixels mm^{-1}	θ_{max} = 27.6º
T = 273(2) K	θ_{min} = 3.3º
ω scans	h = −6→6
Absorption correction: multi-scan (SADABS; Bruker, 1998)	k = −44→44
T_{min} = 0.936, T_{max} = 0.976	l = −11→11
7743 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
$R[F^2 > 2\sigma(F^2)]$ = 0.054	H-atom parameters constrained
$wR(F^2)$ = 0.117	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.5358P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
3401 reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
208 parameters	$\Delta\rho_{min}$ = −0.18 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}
C1	0.3553 (9)	0.97519 (9)	0.8154 (4)	0.0902 (12)
H1B	0.4486	0.9957	0.8639	0.108*
C2	0.0784 (6)	0.91469 (9)	0.6775 (4)	0.0685 (9)
H2B	−0.0180	0.8943	0.6301	0.082*
C3	0.0072 (7)	0.95336 (11)	0.6456 (4)	0.0884 (11)
H3B	−0.1360	0.9589	0.5773	0.106*
C4	0.1484 (9)	0.98325 (10)	0.7151 (5)	0.0905 (12)
H4A	0.1025	1.0093	0.6935	0.109*
C5	0.4286 (7)	0.93649 (8)	0.8464 (4)	0.0707 (9)
H5A	0.5734	0.9311	0.9140	0.085*
C6	0.2887 (5)	0.90608 (7)	0.7777 (3)	0.0464 (7)
C7	0.3757 (5)	0.86499 (7)	0.8103 (3)	0.0435 (6)
C8	0.1220 (5)	0.76940 (7)	0.7606 (3)	0.0403 (6)
C9	0.2169 (5)	0.72870 (7)	0.7964 (3)	0.0385 (6)
C10	0.5067 (5)	0.68088 (7)	0.9256 (3)	0.0469 (7)
H10A	0.6467	0.6752	0.9957	0.056*
C11	0.4322 (5)	0.71903 (7)	0.9015 (3)	0.0417 (6)
C12	0.0889 (5)	0.69841 (7)	0.7208 (3)	0.0454 (7)
H12A	−0.0537	0.7044	0.6527	0.054*
C13	0.1627 (5)	0.65876 (7)	0.7414 (3)	0.0431 (6)
C14	0.3760 (5)	0.64988 (7)	0.8464 (3)	0.0457 (7)
C15	0.4518 (6)	0.60999 (8)	0.8667 (4)	0.0624 (8)
H15A	0.5928	0.6036	0.9353	0.075*
C16	0.0314 (6)	0.62774 (8)	0.6613 (3)	0.0576 (8)
H16A	−0.1112	0.6333	0.5927	0.069*
C17	0.1121 (6)	0.58983 (8)	0.6841 (3)	0.0638 (8)
H17A	0.0261	0.5696	0.6299	0.077*
C18	0.3226 (6)	0.58093 (8)	0.7879 (4)	0.0673 (9)
H18A	0.3750	0.5548	0.8033	0.081*
O1	0.6144 (3)	0.85645 (5)	0.8467 (2)	0.0635 (6)
O2	−0.0689 (4)	0.77662 (5)	0.6712 (2)	0.0607 (6)
O3	0.5609 (3)	0.74934 (5)	0.9766 (2)	0.0548 (5)
H3A	0.6758	0.7404	1.0367	0.082*

N1	0.1807 (4)	0.83749 (6)	0.7983 (2)	0.0471 (6)
H1A	0.0145	0.8436	0.7710	0.056*
N2	0.2512 (4)	0.79894 (6)	0.8305 (2)	0.0474 (6)
H2A	0.3797	0.7940	0.8963	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.135 (3)	0.045 (2)	0.090 (3)	−0.012 (2)	0.001 (3)	−0.0035 (19)
C2	0.0663 (19)	0.0537 (18)	0.085 (2)	−0.0009 (15)	−0.0127 (18)	0.0181 (16)
C3	0.086 (2)	0.068 (2)	0.110 (3)	0.012 (2)	−0.015 (2)	0.032 (2)
C4	0.119 (3)	0.054 (2)	0.100 (3)	0.021 (2)	0.020 (3)	0.020 (2)
C5	0.083 (2)	0.0514 (19)	0.077 (2)	−0.0081 (17)	−0.0127 (18)	0.0005 (16)
C6	0.0409 (15)	0.0427 (15)	0.0557 (18)	0.0002 (12)	0.0008 (14)	0.0035 (13)
C7	0.0352 (15)	0.0459 (15)	0.0488 (16)	−0.0003 (13)	−0.0062 (12)	−0.0019 (12)
C8	0.0356 (13)	0.0444 (15)	0.0404 (15)	−0.0007 (12)	−0.0094 (13)	−0.0014 (12)
C9	0.0350 (13)	0.0407 (14)	0.0394 (15)	0.0005 (11)	−0.0060 (12)	0.0016 (12)
C10	0.0439 (14)	0.0448 (16)	0.0511 (16)	0.0045 (12)	−0.0141 (13)	0.0051 (13)
C11	0.0411 (14)	0.0410 (15)	0.0424 (15)	−0.0030 (12)	−0.0070 (12)	−0.0020 (12)
C12	0.0426 (15)	0.0472 (16)	0.0457 (16)	−0.0019 (12)	−0.0101 (13)	0.0019 (13)
C13	0.0447 (15)	0.0402 (15)	0.0443 (15)	−0.0039 (12)	0.0011 (13)	−0.0016 (12)
C14	0.0426 (15)	0.0427 (15)	0.0518 (16)	0.0031 (12)	−0.0001 (13)	0.0031 (13)
C15	0.0633 (19)	0.0454 (17)	0.078 (2)	0.0051 (14)	−0.0099 (16)	0.0045 (15)
C16	0.0643 (19)	0.0491 (18)	0.0589 (18)	−0.0085 (14)	−0.0060 (15)	−0.0029 (14)
C17	0.072 (2)	0.0476 (18)	0.072 (2)	−0.0113 (15)	0.0003 (18)	−0.0118 (15)
C18	0.074 (2)	0.0413 (16)	0.087 (2)	0.0031 (16)	0.0009 (19)	−0.0037 (16)
O1	0.0357 (10)	0.0544 (12)	0.0995 (17)	0.0012 (9)	−0.0154 (10)	0.0020 (11)
O2	0.0610 (12)	0.0479 (11)	0.0709 (14)	0.0019 (9)	−0.0377 (11)	−0.0008 (9)
O3	0.0550 (11)	0.0443 (10)	0.0634 (12)	0.0041 (9)	−0.0303 (10)	0.0009 (9)
N1	0.0340 (11)	0.0389 (12)	0.0673 (15)	0.0032 (10)	−0.0172 (10)	0.0033 (11)
N2	0.0442 (12)	0.0371 (12)	0.0593 (14)	0.0040 (10)	−0.0249 (11)	0.0021 (11)

Geometric parameters (Å, °)

C1—C4	1.352 (5)	C10—C14	1.407 (3)
C1—C5	1.385 (4)	C10—H10A	0.9300
C1—H1B	0.9300	C11—O3	1.368 (3)
C2—C6	1.368 (4)	C12—C13	1.402 (3)
C2—C3	1.383 (4)	C12—H12A	0.9300
C2—H2B	0.9300	C13—C14	1.408 (4)
C3—C4	1.362 (5)	C13—C16	1.413 (4)
C3—H3B	0.9300	C14—C15	1.412 (4)
C4—H4A	0.9300	C15—C18	1.353 (4)
C5—C6	1.369 (4)	C15—H15A	0.9300
C5—H5A	0.9300	C16—C17	1.357 (4)
C6—C7	1.482 (3)	C16—H16A	0.9300
C7—O1	1.227 (3)	C17—C18	1.392 (4)
C7—N1	1.330 (3)	C17—H17A	0.9300
C8—O2	1.229 (3)	C18—H18A	0.9300

C8—N2	1.327 (3)	O3—H3A	0.8200
C8—C9	1.487 (3)	N1—N2	1.379 (3)
C9—C12	1.367 (3)	N1—H1A	0.8600
C9—C11	1.421 (3)	N2—H2A	0.8600
C10—C11	1.358 (3)		
C4—C1—C5	120.3 (3)	C10—C11—C9	120.7 (2)
C4—C1—H1B	119.8	O3—C11—C9	117.8 (2)
C5—C1—H1B	119.8	C9—C12—C13	123.0 (2)
C6—C2—C3	120.9 (3)	C9—C12—H12A	118.5
C6—C2—H2B	119.6	C13—C12—H12A	118.5
C3—C2—H2B	119.6	C12—C13—C14	118.2 (2)
C4—C3—C2	119.5 (3)	C12—C13—C16	122.6 (2)
C4—C3—H3B	120.2	C14—C13—C16	119.3 (2)
C2—C3—H3B	120.2	C10—C14—C13	119.1 (2)
C1—C4—C3	120.3 (3)	C10—C14—C15	122.7 (2)
C1—C4—H4A	119.9	C13—C14—C15	118.3 (2)
C3—C4—H4A	119.9	C18—C15—C14	121.1 (3)
C6—C5—C1	120.2 (3)	C18—C15—H15A	119.5
C6—C5—H5A	119.9	C14—C15—H15A	119.5
C1—C5—H5A	119.9	C17—C16—C13	120.4 (3)
C2—C6—C5	118.8 (3)	C17—C16—H16A	119.8
C2—C6—C7	122.1 (2)	C13—C16—H16A	119.8
C5—C6—C7	119.0 (2)	C16—C17—C18	120.5 (3)
O1—C7—N1	121.2 (2)	C16—C17—H17A	119.7
O1—C7—C6	122.3 (2)	C18—C17—H17A	119.7
N1—C7—C6	116.4 (2)	C15—C18—C17	120.5 (3)
O2—C8—N2	119.4 (2)	C15—C18—H18A	119.8
O2—C8—C9	123.1 (2)	C17—C18—H18A	119.8
N2—C8—C9	117.4 (2)	C11—O3—H3A	109.5
C12—C9—C11	117.8 (2)	C7—N1—N2	118.4 (2)
C12—C9—C8	117.3 (2)	C7—N1—H1A	120.8
C11—C9—C8	124.9 (2)	N2—N1—H1A	120.8
C11—C10—C14	121.2 (2)	C8—N2—N1	120.4 (2)
C11—C10—H10A	119.4	C8—N2—H2A	119.8
C14—C10—H10A	119.4	N1—N2—H2A	119.8
C10—C11—O3	121.5 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1^{i}	0.86	2.12	2.864 (3)	145
N2—H2A···O3	0.86	1.88	2.582 (3)	138
O3—H3A···O2^{ii}	0.82	1.79	2.613 (3)	177

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*+1, −*y*+3/2, *z*+1/2.

Fig. 1

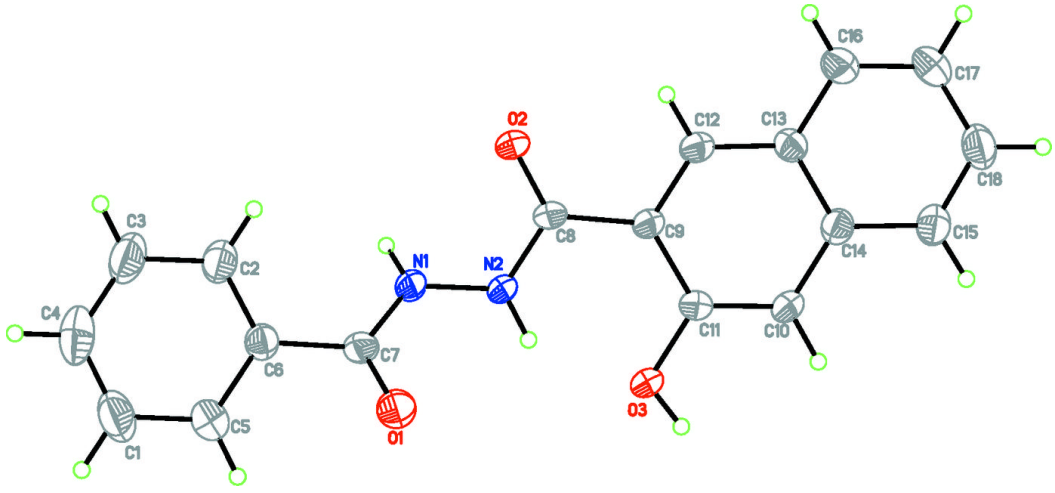
C16
C17
O2
C12
C13
C18
C3
C8
C9
C15
C2
N1
N2
C14
C4
C11
C10
C7
C6
O3
O1
C5
C1

Fig. 2