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N′-Isopropylidene-6-methoxy-2-naphthohydrazide

B. K. Sarojini,[a] B. Narayana,[b] K. Sunil,[b] H. S. Yathirajan[c] and Michael Bolte[d]*

[a]Department of Chemistry, P. A. College of Engineering, Nadupadavu, Mangalore 574 153, India, [b]Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India, [c]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and [d]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany
Correspondence e-mail: bolte@chemie.uni-frankfurt.de

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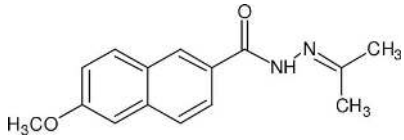
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.087; data-to-parameter ratio = 8.6.

The geometric parameters of the title compound, $C_{15}H_{16}N_2O_2$, are in the usual ranges. The molecule is almost planar; only the torsion angles about the N—N bond [164.11 (15)°] and the C—C bond between the naphthyl and the amide group [C—C—C—O = − 157.24 (15)°] differ significantly from 0 or 180°. The crystal packing is characterized by chains of molecules connected by N—H···O hydrogen bonds running along the c axis.

Related literature

For related structures, see: Narayana *et al.* (2007); Yathirajan, Sarojini *et al.* (2007); Yathirajan, Narayana *et al.* (2007); Sarojini *et al.* (2007). For related literature, see: Misra *et al.* (1981); Agarwal *et al.* (1983); Varma *et al.* (1986); Singh & Dash (1988); Liu *et al.* (2006); Narayana, Ashalatha *et al.* (2005); Narayana, Vijayaraj *et al.* (2005).



Experimental

Crystal data

$C_{15}H_{16}N_2O_2$
M_r = 256.30
Monoclinic, *Cc*
a = 24.037 (3) Å
b = 5.9957 (4) Å
c = 9.3014 (10) Å
β = 91.673 (9)°
V = 1339.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.38 × 0.24 × 0.24 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: none
8435 measured reflections
1539 independent reflections
1506 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.087
S = 1.07
1539 reflections
180 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88 (2)	1.98 (2)	2.8222 (17)	161.3 (19)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2341).

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supplementary materials

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N'-Isopropylidene-6-methoxy-2-naphthohydrazide

B. K. Sarojini, B. Narayana, K. Sunil, H. S. Yathirajan and M. Bolte

Comment

Hydrazides and the corresponding Schiff bases are useful precursors in the synthesis of several heterocyclic systems. Some substituted hydrazides are reported to exhibit carcinostatic activity against several types of tumors and also possess anti-microbial activity. Some substituted hydrazides and their Schiff bases are reported to exhibit carcinostatic activity against several types of tumors and also possess antimicrobial activity. It is also used as an intermediate in many pharmaceutically important compounds. A new Schiff base of the hydrazide, $C_{15}H_{16}N_2O_2$, was accidentally formed, when the synthesized hydrazide was recrystallized using a 1:1 mixture of acetone and dimethyl formamide. The crystal structure of the newly formed compound is reported.

Geometric parameters of the title compound (Fig. 1) are in the usual ranges. The crystal packing is characterized by chains of molecules connected by N—H···O hydrogen bonds running along the *c* axis (Fig. 2).

Experimental

Hydrazine hydrate (100%) (2 ml) was added to methyl 6-methoxy-2-naphthoate (4.3 g, 0.02 mol) in 20 ml e thanol and refluxed for 4 h on a water bath. The precipitate formed was collected and recrystallized from ethyl acetate. X-ray quality crystals were obtained from a mixture of acetone and DMF(1:1) and the compound obtained was a Schiff base of the hydrazide with acetone, $C_{15}H_{16}N_2O_2$ (m.p.: 445–447 K). Analysis for $C_{15}H_{16}N_2O_2$: Found (Calculated): C 70.20 (70.29), H 6.22 (6.29), N 10.87% (10.93%).

Refinement

H atoms were found in a difference map, but those bonded to C were refined using a riding model with C—H = 0.95Å and U_{iso}(H) = 1.2U_{eq}(C) [C—H = 0.98Å and U_{iso}(H) = 1.5U_{eq}(C) for the methyl groups, which were allowed to rotate but not to tip]. The amino H atom was freely refined. In the absence of anomalous scatterers, the Flack [(1983). Acta Cryst. A39, 876–881] parameter is meaningless and therefore, Friedel pairs had been merged prior to refinement.

Figures

Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

Fig. 2. Packing diagram of the title compound; hydrogen bonds are shown as dashed lines.


Fig. 3. the formation of the title compound.

N'-Isopropylidene-6-methoxy-2-naphthohydrazide

Crystal data

$C_{15}H_{16}N_2O_2$	F_{000} = 544
M_r = 256.30	D_x = 1.270 Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C -2yc	Cell parameters from 8050 reflections
a = 24.037 (3) Å	θ = 3.6–27.2º
b = 5.9957 (4) Å	μ = 0.09 mm^{-1}
c = 9.3014 (10) Å	*T* = 173 (2) K
β = 91.673 (9)º	Rod, colourless
V = 1339.9 (2) Å^3	0.38 × 0.24 × 0.24 mm
Z = 4	

Data collection

Stoe IPDS II two-circle diffractometer	1506 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.037
Monochromator: graphite	θ_{max} = 27.5º
T = 173(2) K	θ_{min} = 3.5º
ω scans	*h* = −27→30
Absorption correction: none	*k* = −7→7
8435 measured reflections	*l* = −12→12
1539 independent reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.1264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.087	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
1539 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
180 parameters	Extinction coefficient: 0.014 (3)
2 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Hydrogen site location: inferred from neighbouring sites

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}
N1	0.61518 (6)	0.6094 (2)	0.48733 (13)	0.0305 (3)
H1	0.6241 (8)	0.630 (3)	0.398 (2)	0.033 (5)*
N2	0.56452 (7)	0.6965 (3)	0.53277 (15)	0.0438 (4)
O1	0.62273 (6)	0.3857 (2)	0.68512 (13)	0.0409 (3)
O2	0.93004 (5)	0.1401 (2)	0.23424 (16)	0.0483 (4)
C1	0.69319 (6)	0.3547 (2)	0.51132 (14)	0.0274 (3)
C2	0.72496 (6)	0.4663 (2)	0.41240 (16)	0.0282 (3)
H2	0.7124	0.6055	0.3751	0.034*
C3	0.77597 (6)	0.3761 (2)	0.36589 (15)	0.0276 (3)
C4	0.80881 (7)	0.4902 (3)	0.26298 (18)	0.0338 (3)
H4	0.7960	0.6276	0.2234	0.041*
C5	0.85828 (7)	0.4036 (3)	0.22131 (19)	0.0373 (4)
H5	0.8792	0.4789	0.1510	0.045*
C6	0.87878 (7)	0.2009 (3)	0.28263 (18)	0.0363 (4)
C7	0.84830 (7)	0.0842 (3)	0.38110 (17)	0.0344 (3)
H7	0.8622	−0.0514	0.4208	0.041*
C8	0.79556 (6)	0.1691 (2)	0.42301 (15)	0.0284 (3)
C9	0.76168 (7)	0.0543 (2)	0.52314 (16)	0.0315 (3)
H9	0.7735	−0.0858	0.5607	0.038*
C10	0.71221 (7)	0.1449 (2)	0.56560 (15)	0.0308 (3)
H10	0.6903	0.0663	0.6323	0.037*
C11	0.64049 (6)	0.4497 (3)	0.56888 (14)	0.0291 (3)
C12	0.54904 (7)	0.8804 (3)	0.47282 (16)	0.0365 (4)
C13	0.49405 (11)	0.9730 (6)	0.5149 (3)	0.0729 (9)
H13A	0.4769	0.8719	0.5835	0.109*
H13B	0.4697	0.9880	0.4291	0.109*
H13C	0.4996	1.1196	0.5596	0.109*
C14	0.58033 (8)	1.0116 (3)	0.3649 (2)	0.0425 (4)
H14A	0.6202	1.0062	0.3895	0.064*
H14B	0.5676	1.1669	0.3655	0.064*
H14C	0.5736	0.9478	0.2690	0.064*

C15	0.95677 (9)	−0.0466 (4)	0.3035 (3)	0.0538 (5)
H15A	0.9593	−0.0207	0.4075	0.081*
H15B	0.9942	−0.0649	0.2664	0.081*
H15C	0.9350	−0.1820	0.2838	0.081*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0336 (7)	0.0371 (7)	0.0210 (6)	0.0077 (5)	0.0070 (5)	0.0023 (5)
N2	0.0427 (8)	0.0602 (10)	0.0294 (6)	0.0208 (7)	0.0142 (6)	0.0113 (6)
O1	0.0504 (7)	0.0507 (7)	0.0218 (5)	0.0072 (5)	0.0062 (4)	0.0086 (5)
O2	0.0358 (7)	0.0496 (8)	0.0601 (8)	0.0133 (6)	0.0107 (6)	0.0061 (6)
C1	0.0316 (7)	0.0270 (7)	0.0235 (6)	−0.0004 (5)	−0.0019 (5)	−0.0013 (5)
C2	0.0310 (7)	0.0249 (7)	0.0287 (7)	0.0011 (5)	−0.0019 (5)	0.0027 (5)
C3	0.0276 (7)	0.0251 (7)	0.0301 (7)	−0.0007 (5)	−0.0019 (5)	0.0011 (5)
C4	0.0310 (7)	0.0307 (7)	0.0395 (8)	0.0011 (6)	0.0007 (6)	0.0088 (6)
C5	0.0306 (8)	0.0377 (8)	0.0438 (9)	−0.0010 (6)	0.0038 (6)	0.0070 (6)
C6	0.0317 (8)	0.0357 (8)	0.0414 (8)	0.0051 (6)	0.0013 (6)	−0.0001 (7)
C7	0.0358 (8)	0.0283 (7)	0.0392 (8)	0.0064 (6)	0.0006 (6)	−0.0008 (6)
C8	0.0317 (8)	0.0238 (6)	0.0294 (7)	0.0005 (5)	−0.0028 (5)	−0.0013 (5)
C9	0.0393 (8)	0.0225 (6)	0.0326 (8)	0.0016 (5)	−0.0030 (6)	0.0031 (5)
C10	0.0390 (8)	0.0271 (7)	0.0261 (6)	−0.0018 (5)	0.0005 (5)	0.0029 (5)
C11	0.0356 (7)	0.0321 (7)	0.0196 (6)	−0.0007 (6)	0.0007 (5)	−0.0010 (5)
C12	0.0372 (8)	0.0485 (9)	0.0241 (7)	0.0111 (7)	0.0017 (5)	−0.0021 (6)
C13	0.0629 (15)	0.099 (2)	0.0581 (13)	0.0480 (14)	0.0258 (11)	0.0284 (13)
C14	0.0404 (8)	0.0315 (8)	0.0555 (10)	0.0012 (6)	−0.0009 (8)	0.0041 (7)
C15	0.0437 (11)	0.0486 (11)	0.0692 (13)	0.0189 (8)	0.0049 (9)	0.0008 (10)

Geometric parameters (Å, °)

N1—C11	1.355 (2)	C6—C7	1.380 (2)
N1—N2	1.4017 (18)	C7—C8	1.431 (2)
N1—H1	0.88 (2)	C7—H7	0.9500
N2—C12	1.286 (2)	C8—C9	1.431 (2)
O1—C11	1.2352 (19)	C9—C10	1.376 (2)
O2—C6	1.3733 (19)	C9—H9	0.9500
O2—C15	1.434 (2)	C10—H10	0.9500
C1—C2	1.385 (2)	C12—C14	1.496 (3)
C1—C10	1.426 (2)	C12—C13	1.496 (2)
C1—C11	1.502 (2)	C13—H13A	0.9800
C2—C3	1.419 (2)	C13—H13B	0.9800
C2—H2	0.9500	C13—H13C	0.9800
C3—C8	1.4245 (18)	C14—H14A	0.9800
C3—C4	1.432 (2)	C14—H14B	0.9800
C4—C5	1.364 (2)	C14—H14C	0.9800
C4—H4	0.9500	C15—H15A	0.9800
C5—C6	1.424 (2)	C15—H15B	0.9800
C5—H5	0.9500	C15—H15C	0.9800

C11—N1—N2	118.32 (12)	C10—C9—H9	119.7
C11—N1—H1	121.1 (13)	C8—C9—H9	119.7
N2—N1—H1	118.2 (13)	C9—C10—C1	121.18 (13)
C12—N2—N1	115.58 (13)	C9—C10—H10	119.4
C6—O2—C15	117.03 (15)	C1—C10—H10	119.4
C2—C1—C10	118.95 (14)	O1—C11—N1	123.28 (15)
C2—C1—C11	122.73 (13)	O1—C11—C1	120.72 (14)
C10—C1—C11	118.26 (12)	N1—C11—C1	115.98 (12)
C1—C2—C3	121.09 (13)	N2—C12—C14	126.69 (15)
C1—C2—H2	119.5	N2—C12—C13	116.84 (16)
C3—C2—H2	119.5	C14—C12—C13	116.47 (16)
C2—C3—C8	119.83 (12)	C12—C13—H13A	109.5
C2—C3—C4	121.29 (13)	C12—C13—H13B	109.5
C8—C3—C4	118.87 (13)	H13A—C13—H13B	109.5
C5—C4—C3	120.66 (14)	C12—C13—H13C	109.5
C5—C4—H4	119.7	H13A—C13—H13C	109.5
C3—C4—H4	119.7	H13B—C13—H13C	109.5
C4—C5—C6	120.35 (15)	C12—C14—H14A	109.5
C4—C5—H5	119.8	C12—C14—H14B	109.5
C6—C5—H5	119.8	H14A—C14—H14B	109.5
O2—C6—C7	125.58 (15)	C12—C14—H14C	109.5
O2—C6—C5	113.48 (14)	H14A—C14—H14C	109.5
C7—C6—C5	120.94 (14)	H14B—C14—H14C	109.5
C6—C7—C8	119.49 (14)	O2—C15—H15A	109.5
C6—C7—H7	120.3	O2—C15—H15B	109.5
C8—C7—H7	120.3	H15A—C15—H15B	109.5
C3—C8—C7	119.61 (13)	O2—C15—H15C	109.5
C3—C8—C9	118.25 (13)	H15A—C15—H15C	109.5
C7—C8—C9	122.13 (13)	H15B—C15—H15C	109.5
C10—C9—C8	120.66 (13)		

C11—N1—N2—C12	164.11 (15)	C4—C3—C8—C9	−178.54 (15)
C10—C1—C2—C3	−0.6 (2)	C6—C7—C8—C3	−1.9 (2)
C11—C1—C2—C3	176.83 (12)	C6—C7—C8—C9	179.15 (14)
C1—C2—C3—C8	−1.0 (2)	C3—C8—C9—C10	−1.6 (2)
C1—C2—C3—C4	179.63 (15)	C7—C8—C9—C10	177.36 (13)
C2—C3—C4—C5	178.74 (14)	C8—C9—C10—C1	0.1 (2)
C8—C3—C4—C5	−0.7 (2)	C2—C1—C10—C9	1.0 (2)
C3—C4—C5—C6	−1.7 (3)	C11—C1—C10—C9	−176.49 (13)
C15—O2—C6—C7	−7.3 (3)	N2—N1—C11—O1	−4.0 (2)
C15—O2—C6—C5	172.53 (17)	N2—N1—C11—C1	177.15 (14)
C4—C5—C6—O2	−177.47 (15)	C2—C1—C11—O1	−157.24 (15)
C4—C5—C6—C7	2.3 (3)	C10—C1—C11—O1	20.2 (2)
O2—C6—C7—C8	179.27 (14)	C2—C1—C11—N1	21.6 (2)
C5—C6—C7—C8	−0.5 (2)	C10—C1—C11—N1	−160.97 (13)
C2—C3—C8—C7	−176.96 (13)	N1—N2—C12—C14	−2.2 (3)
C4—C3—C8—C7	2.4 (2)	N1—N2—C12—C13	177.68 (19)
C2—C3—C8—C9	2.07 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88 (2)	1.98 (2)	2.8222 (17)	161.3 (19)

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

Fig. 1

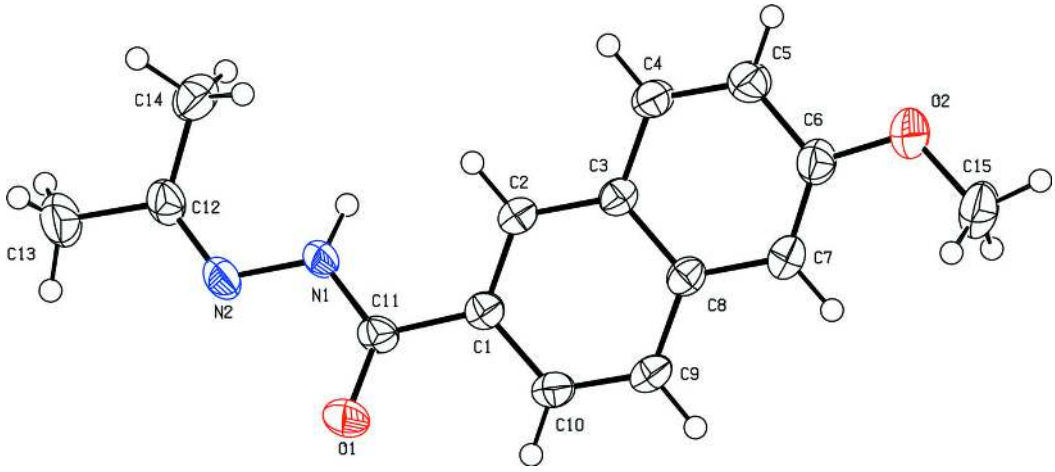

Fig. 2

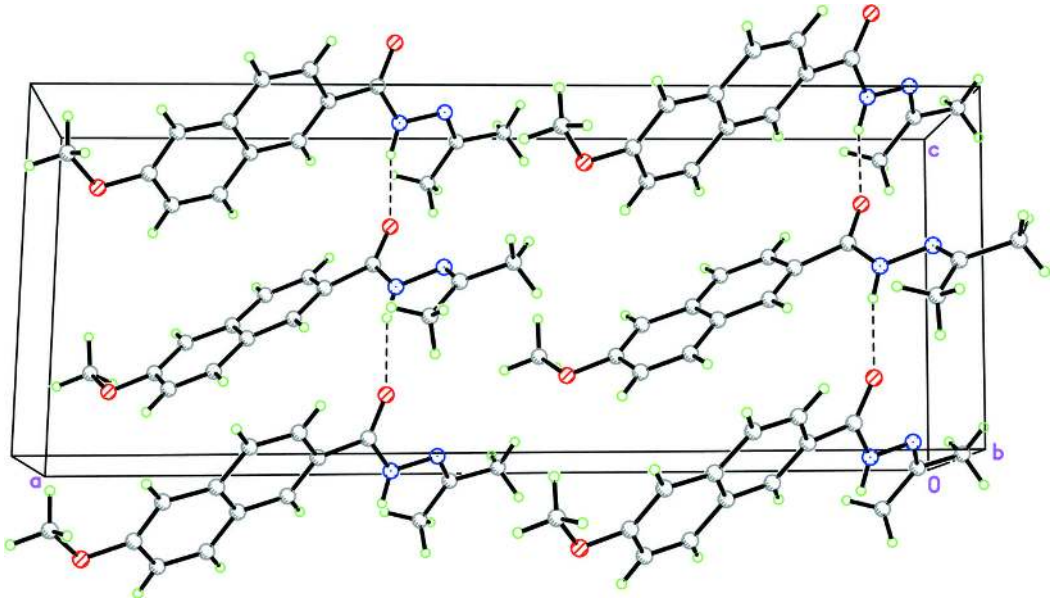

Fig. 3