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Ethyl 1-aminonaphtho[2,1-*b*]furan-2-carboxylate

K. S. Madan,[a] E. Shruthi,[b] A. M. Sagar,[b] M. M. M Abdoh,[c] V. P. Vaidya[b] and N. K. Lokanath[a]*

[a]Department of Studies in Physics, Manasagangotri, University of Mysore, Mysore 570 006, India, [b]Department of Chemistry, Kuvempu University, Shankaraghatta 577 451, India, and [c]Department of Physics, Faculty of Science, An Najah National University, Nabtus, West Bank, Palestinian Territories
Correspondence e-mail: lokanath@physics.uni-mysore.ac.in

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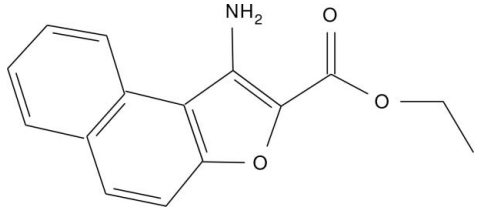
Key indicators: single-crystal X-ray study; T = 103 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.117; data-to-parameter ratio = 7.4.

In the title compound, $C_{15}H_{13}NO_3$, there is intramolecular N—H···O hydrogen bond between the amino group and the ester carbonyl O atom and the dihedral angle between the aromatic ring and the ester group is 2.05 (15)°. In the crystal, molecules are connected by N—H···O hydrogen bonds into chains parallel to [010]. In addition there are short C—H···O interactions and π–π stacking interactions with a distance of 3.555 (2) Å between the centroids of the furan and benzene rings.

Related literature

For bioactivity of naphthofuran compounds, see: Nagaraja *et al.* (2006); Mahadevan *et al.* (2005). For similar structures, see: Shruthi *et al.* (2012). For the synthesis of the title compound, see: Veena *et al.* (2011)



Experimental

Crystal data

$C_{15}H_{13}NO_3$
M_r = 255.26
Orthorhombic, $P2_12_12_1$
a = 6.2217 (5) Å
b = 8.3795 (6) Å
c = 23.4692 (17) Å
V = 1223.56 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 103 K
0.28 × 0.22 × 0.22 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
18171 measured reflections
1288 independent reflections
1093 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.117
S = 1.03
1288 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···O2	0.86	2.30	2.872 (4)	124
N4—H4B···O2^{i}	0.86	2.29	3.027 (4)	144
C9—H9···O2^{i}	0.93	2.50	3.308 (4)	146

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2494).

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supplementary materials

Acta Cryst. (2012). E**68**, o2114 [doi:10.1107/S1600536812025342]

Ethyl 1-aminonaphtho[2,1-*b*]furan-2-carboxylate

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Comment

Various derivatives of naphtho[2,1-b]furan synthesized in our laboratory have been shown to possess broad spectrum of pharmacological and biological activities (Nagaraja *et al.*, 2006).

The title compound was synthesized as an intermediate in the synthesis of aza heterocyclic derivative of naphtho[2,1-b]furan. The synthesis of the title compound was reported by Veena *et al.* (2011).

The ORTEP drawing of the title molecule is shown in Fig. 1. The naphthofuran system is basically planar and its geometry is similar to ethyl naphtho[2,1- b]furan-2-carboxylate (Shruthi *et al.*, 2012). The dihedral angle between aromatic ring and the ester group is 2.05 (15)°.

The molecules are connected by N-H···O interactions into chains along the *b* axis (Fig. 2). There are π-π stacking interactions between molecules related by unit translation along the *a* axis with the distance of 3.555 (2) Å between centroids of the furan and benzene rings.

Experimental

Ethyl 3-aminonaphtho[2,1-*b*]furan-2-carboxylate was synthesized as per the procedure reported in the literature (Veena *et al.*, 2011). The final product was obtained by recrystallization using aqueous ethanol as a solvent. Slow evaporation method yielded crystals.

Refinement

In the absence of significant anomalous dispersion effects Friedel pairs have been merged. All the hydrogen atoms of the compound are fixed geometrically (N-H = 0.86 and C-H= 0.93-0.97 Å) and allowed to ride on their parent atoms.

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2010); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* (Spek, 2009).

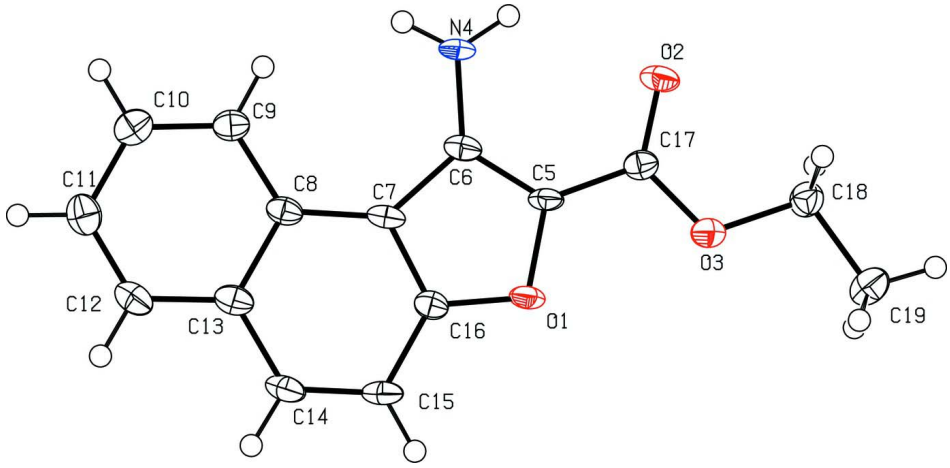

Figure 1

ORTEP diagram of the title compound with 50% probability ellipsoids.

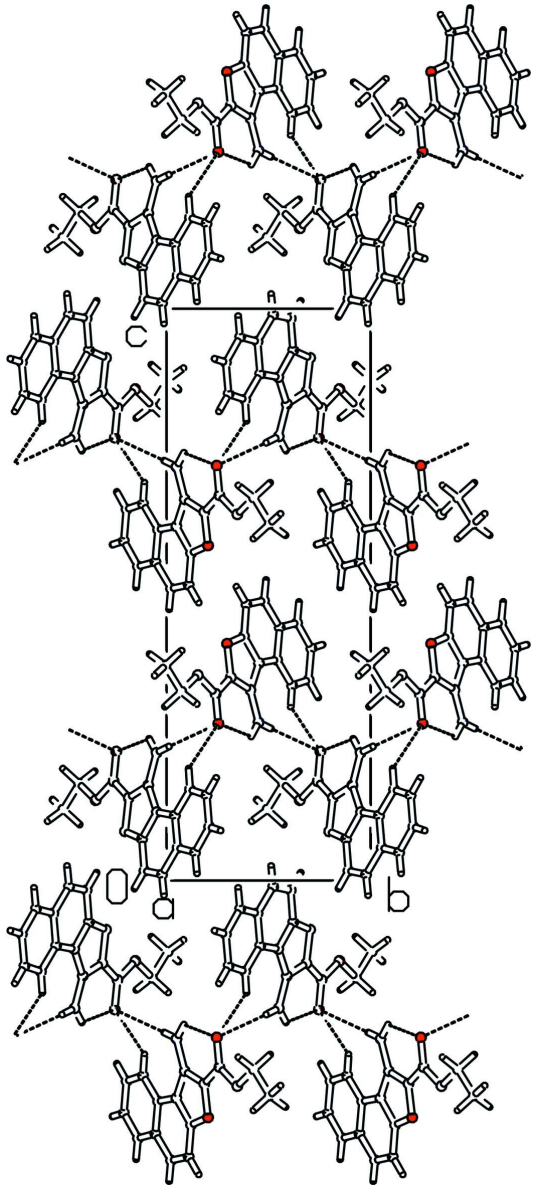

Figure 2

Packing diagram of the title compound viewed along the crystallographic *a* axis. N—H···O hydrogen bonds are indicated by dashed lines.

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Crystal data

$C_{15}H_{13}NO_3$
$M_r = 255.26$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.2217\ (5)$ Å
$b = 8.3795\ (6)$ Å
$c = 23.4692\ (17)$ Å
$V = 1223.56\ (16)$ Å^3
$Z = 4$
$F(000) = 536$
$D_x = 1.386$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1288 reflections
$\theta = 1.7$–$25.0°$
$\mu = 0.10$ mm^{-1}
$T = 103$ K
Block, colorless
0.28 × 0.22 × 0.22 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.0839 pixels mm^{-1}
ω scans
18171 measured reflections
1288 independent reflections
1093 reflections with $I > 2\sigma(I)$
$R_{int} = 0.064$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.7°$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -27 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.117$
$S = 1.03$
1288 reflections
173 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.4783P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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}
O1	0.9439 (3)	0.7967 (3)	0.08591 (8)	0.0215 (7)
O2	0.7201 (4)	0.7539 (3)	0.22562 (9)	0.0245 (7)
O3	0.6156 (4)	0.6531 (3)	0.14062 (9)	0.0243 (7)
N4	1.0980 (4)	0.9507 (3)	0.22348 (11)	0.0233 (8)
C5	0.9186 (5)	0.8149 (4)	0.14469 (12)	0.0196 (9)
C6	1.0771 (5)	0.9106 (4)	0.16666 (13)	0.0200 (10)
C7	1.2157 (5)	0.9524 (4)	0.11969 (12)	0.0194 (9)
C8	1.4090 (5)	1.0413 (4)	0.11392 (13)	0.0199 (9)
C9	1.5215 (5)	1.1151 (4)	0.15900 (13)	0.0220 (9)
C10	1.7085 (5)	1.1961 (4)	0.14924 (14)	0.0258 (10)
C11	1.7922 (6)	1.2091 (4)	0.09382 (14)	0.0276 (10)
C12	1.6865 (6)	1.1397 (4)	0.04934 (14)	0.0263 (11)
C13	1.4948 (6)	1.0545 (4)	0.05737 (13)	0.0226 (9)
C14	1.3886 (6)	0.9777 (4)	0.01060 (13)	0.0252 (10)
C15	1.2054 (6)	0.8914 (4)	0.01695 (12)	0.0241 (10)
C16	1.1224 (5)	0.8797 (4)	0.07259 (13)	0.0201 (9)
C17	0.7458 (5)	0.7406 (4)	0.17366 (13)	0.0197 (10)
C18	0.4392 (5)	0.5720 (4)	0.16960 (14)	0.0249 (10)

C19	0.3046 (6)	0.4943 (4)	0.12417 (15)	0.0302 (11)
H4A	1.00740	0.91480	0.24800	0.0280*
H4B	1.20160	1.01150	0.23430	0.0280*
H9	1.46750	1.10840	0.19590	0.0260*
H10	1.78100	1.24320	0.17960	0.0310*
H11	1.91940	1.26470	0.08750	0.0330*
H12	1.74280	1.14920	0.01280	0.0320*
H14	1.44760	0.98740	−0.02560	0.0300*
H15	1.13830	0.84250	−0.01390	0.0290*
H18A	0.35390	0.64800	0.19110	0.0300*
H18B	0.49460	0.49230	0.19570	0.0300*
H19A	0.25390	0.57410	0.09810	0.0450*
H19B	0.18410	0.44170	0.14150	0.0450*
H19C	0.38990	0.41750	0.10390	0.0450*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0281 (12)	0.0267 (12)	0.0098 (10)	−0.0011 (11)	0.0003 (9)	−0.0002 (9)
O2	0.0339 (13)	0.0273 (13)	0.0122 (11)	0.0006 (12)	0.0006 (9)	0.0017 (9)
O3	0.0282 (12)	0.0274 (12)	0.0173 (11)	−0.0038 (11)	−0.0004 (9)	−0.0010 (9)
N4	0.0301 (15)	0.0319 (15)	0.0080 (12)	−0.0032 (15)	−0.0007 (11)	−0.0010 (11)
C5	0.0276 (17)	0.0217 (16)	0.0094 (13)	−0.0001 (16)	−0.0013 (13)	−0.0003 (12)
C6	0.0288 (18)	0.0185 (17)	0.0126 (15)	0.0043 (15)	0.0007 (13)	0.0009 (12)
C7	0.0282 (17)	0.0197 (16)	0.0104 (14)	0.0061 (15)	0.0012 (14)	−0.0007 (12)
C8	0.0270 (17)	0.0173 (16)	0.0153 (15)	0.0042 (16)	0.0013 (14)	0.0020 (12)
C9	0.0255 (17)	0.0211 (16)	0.0193 (16)	0.0032 (16)	−0.0019 (14)	−0.0002 (13)
C10	0.0281 (18)	0.0224 (18)	0.0268 (17)	0.0030 (17)	−0.0058 (14)	0.0010 (15)
C11	0.0261 (18)	0.0242 (18)	0.0324 (18)	0.0009 (17)	0.0018 (15)	0.0057 (15)
C12	0.0325 (19)	0.0256 (19)	0.0207 (17)	0.0016 (16)	0.0074 (15)	0.0033 (14)
C13	0.0289 (17)	0.0212 (17)	0.0177 (15)	0.0065 (16)	0.0020 (14)	0.0009 (13)
C14	0.038 (2)	0.0237 (18)	0.0138 (15)	0.0006 (18)	0.0060 (14)	−0.0007 (13)
C15	0.0369 (19)	0.0236 (18)	0.0119 (15)	0.0025 (17)	−0.0006 (14)	−0.0037 (13)
C16	0.0236 (16)	0.0209 (16)	0.0157 (15)	0.0016 (15)	0.0002 (13)	0.0022 (13)
C17	0.0237 (17)	0.0177 (17)	0.0177 (16)	0.0021 (15)	−0.0026 (13)	0.0007 (13)
C18	0.0269 (18)	0.0228 (18)	0.0249 (17)	−0.0034 (16)	−0.0020 (14)	0.0014 (14)
C19	0.033 (2)	0.0276 (19)	0.0300 (19)	−0.0028 (17)	−0.0091 (16)	−0.0003 (15)

Geometric parameters (Å, º)

O1—C5	1.397 (3)	C11—C12	1.364 (5)
O1—C16	1.347 (4)	C12—C13	1.403 (5)
O2—C17	1.235 (4)	C13—C14	1.434 (5)
O3—C17	1.340 (4)	C14—C15	1.358 (5)
O3—C18	1.459 (4)	C15—C16	1.408 (4)
N4—C6	1.381 (4)	C18—C19	1.504 (5)
N4—H4A	0.8600	C9—H9	0.9300
N4—H4B	0.8600	C10—H10	0.9300
C5—C17	1.416 (4)	C11—H11	0.9300
C5—C6	1.372 (4)	C12—H12	0.9300

C6—C7	1.443 (4)	C14—H14	0.9300
C7—C16	1.389 (4)	C15—H15	0.9300
C7—C8	1.421 (4)	C18—H18A	0.9700
C8—C9	1.411 (4)	C18—H18B	0.9700
C8—C13	1.435 (4)	C19—H19A	0.9600
C9—C10	1.366 (4)	C19—H19B	0.9600
C10—C11	1.405 (5)	C19—H19C	0.9600

C5—O1—C16	105.4 (2)	O1—C16—C15	123.6 (3)
C17—O3—C18	116.1 (2)	O2—C17—O3	122.9 (3)
H4A—N4—H4B	120.00	O2—C17—C5	122.2 (3)
C6—N4—H4B	120.00	O3—C17—C5	114.9 (3)
C6—N4—H4A	120.00	O3—C18—C19	106.9 (3)
C6—C5—C17	128.5 (3)	C8—C9—H9	119.00
O1—C5—C17	120.8 (3)	C10—C9—H9	120.00
O1—C5—C6	110.7 (3)	C9—C10—H10	120.00
N4—C6—C5	125.0 (3)	C11—C10—H10	120.00
N4—C6—C7	128.5 (3)	C10—C11—H11	120.00
C5—C6—C7	106.5 (3)	C12—C11—H11	120.00
C8—C7—C16	120.5 (3)	C11—C12—H12	119.00
C6—C7—C8	134.9 (3)	C13—C12—H12	119.00
C6—C7—C16	104.6 (3)	C13—C14—H14	119.00
C7—C8—C9	125.3 (3)	C15—C14—H14	119.00
C7—C8—C13	116.3 (3)	C14—C15—H15	122.00
C9—C8—C13	118.4 (3)	C16—C15—H15	122.00
C8—C9—C10	121.0 (3)	O3—C18—H18A	110.00
C9—C10—C11	120.6 (3)	O3—C18—H18B	110.00
C10—C11—C12	119.8 (3)	C19—C18—H18A	110.00
C11—C12—C13	121.6 (3)	C19—C18—H18B	110.00
C8—C13—C14	120.1 (3)	H18A—C18—H18B	109.00
C8—C13—C12	118.7 (3)	C18—C19—H19A	109.00
C12—C13—C14	121.2 (3)	C18—C19—H19B	109.00
C13—C14—C15	122.8 (3)	C18—C19—H19C	109.00
C14—C15—C16	116.5 (3)	H19A—C19—H19B	109.00
C7—C16—C15	123.7 (3)	H19A—C19—H19C	109.00
O1—C16—C7	112.7 (3)	H19B—C19—H19C	109.00

C16—O1—C5—C6	1.3 (4)	C16—C7—C8—C13	1.9 (5)
C16—O1—C5—C17	−179.2 (3)	C6—C7—C16—O1	−0.8 (4)
C5—O1—C16—C7	−0.3 (4)	C6—C7—C16—C15	179.3 (3)
C5—O1—C16—C15	179.7 (3)	C8—C7—C16—O1	178.2 (3)
C18—O3—C17—O2	−0.3 (4)	C8—C7—C16—C15	−1.8 (5)
C18—O3—C17—C5	178.6 (3)	C7—C8—C9—C10	178.9 (3)
C17—O3—C18—C19	175.1 (3)	C13—C8—C9—C10	−0.5 (5)
O1—C5—C6—N4	−179.5 (3)	C7—C8—C13—C12	−179.4 (3)
O1—C5—C6—C7	−1.8 (4)	C7—C8—C13—C14	−1.2 (5)
C17—C5—C6—N4	1.1 (6)	C9—C8—C13—C12	0.1 (5)
C17—C5—C6—C7	178.8 (3)	C9—C8—C13—C14	178.3 (3)
O1—C5—C17—O2	179.6 (3)	C8—C9—C10—C11	0.6 (5)

O1—C5—C17—O3	0.7 (4)	C9—C10—C11—C12	−0.3 (5)
C6—C5—C17—O2	−1.0 (6)	C10—C11—C12—C13	−0.1 (5)
C6—C5—C17—O3	−179.9 (3)	C11—C12—C13—C8	0.2 (5)
N4—C6—C7—C8	0.4 (6)	C11—C12—C13—C14	−178.0 (3)
N4—C6—C7—C16	179.1 (3)	C8—C13—C14—C15	0.4 (5)
C5—C6—C7—C8	−177.2 (4)	C12—C13—C14—C15	178.6 (3)
C5—C6—C7—C16	1.5 (4)	C13—C14—C15—C16	−0.2 (5)
C6—C7—C8—C9	1.0 (6)	C14—C15—C16—O1	−179.1 (3)
C6—C7—C8—C13	−179.6 (4)	C14—C15—C16—C7	0.9 (5)
C16—C7—C8—C9	−177.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···O2	0.86	2.30	2.872 (4)	124
N4—H4*B*···O2^{i}	0.86	2.29	3.027 (4)	144
C9—H9···O2^{i}	0.93	2.50	3.308 (4)	146

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