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(1-Bromonaphthalen-2-yl)acetonitrile

Andria D. Harris, Amy D. Baucom, Jessica L. Brown, Daniel S. Jones* and Craig A. Ogle*

Department of Chemistry, The University of North Carolina at Charlotte, 9201 University City Blvd, Charlotte, NC 28223, USA
Correspondence e-mail: djones@uncc.edu, cogle@uncc.edu

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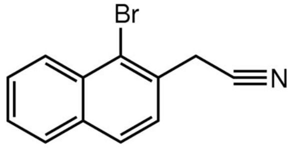
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.025; wR factor = 0.063; data-to-parameter ratio = 13.6.

The title compound, $C_{12}H_8BrN$, was prepared as a starting material for a Suzuki cross-coupling reaction with a pinacol ester. The torsion angle about the ring–methylene C—C bond is 30.7 (3)°, such that the N atom is displaced by 1.174 (4) Å from the plane of the naphthalene ring system.

Related literature

A search of the Cambridge Structural Database [Version 5.29 (Allen, 2002); *CONQUEST* (Bruno *et al.*, 2002)] yielded one comparable structure, (4-bromonaphthalen-2-yl)acetonitrile (Refcode BAGTEJ; Duthie *et al.*, 2001).



Experimental

Crystal data

$C_{12}H_8BrN$
M_r = 246.1
Monoclinic, $P2_1/n$
a = 11.3599 (13) Å
b = 7.2379 (8) Å
c = 11.8901 (15) Å
β = 102.538 (10)°
V = 954.31 (19) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 5.47 mm^{-1}
T = 295 (2) K
0.5 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
6502 measured reflections
1729 independent reflections
1558 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.031
3 standard reflections every 75 reflections intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.063
S = 1.01
1729 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.35 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.51 e Å^{-3}

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2183).

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supplementary materials

Acta Cryst. (2008). E**64**, o1284 [**doi:10.1107/S1600536808017418**]

(1-Bromonaphthalen-2-yl)acetonitrile

A. D. Harris, A. D. Baucom, J. L. Brown, D. S. Jones and C. A. Ogle

Comment

The title compound (Fig.1) was prepared as a starting material for a Suzuki cross coupling reaction with a pinacol ester. The C11—C12—N angle is 178.4 (3)°, and the plane of that grouping makes an angle of 42.5 (1)° with the plane of the naphthalene ring, while the N atom is displaced 1.174 (4) Å from the plane of the naphthalene ring. As shown in Figs. 2 and 3, the molecules form alternating layers when viewed edge-on and form columns when viewed along the *b* axis.

A search of the Cambridge Structural Database [Version 5.29; (Allen, 2002); *CONQUEST*, Version 1.10 (Bruno *et al.*, 2002)] yielded one comparable structure, (4-bromonapthalen-2-yl)acetonitrile (Refcode BAGTEJ; Duthie *et al.*, 2001). In that structure the acetonitrile C—C—N angle was 179.3°, and the plane of that grouping made an angle of 23.1° with the plane of the naphthalene ring. The N atom was displaced 0.287 Å from the plane of the naphthalene ring.

Experimental

Synthesis of 1-bromo-2-methylnaphthalene (II) (Fig. 4). A solution of 2-methylnaphthalene (I) in acetic acid was stirred while an equivalent amount of Br_2 in acetic acid was added dropwise at a rate that allowed the bromine color to dissipate between drops. Upon completion of addition the mixture was allowed to stir for 1 h at which time the entire mixture was poured into water. The organic phase was separated and washed repeatedly with water to remove the acetic acid. The product was dried with K_2CO_3 and used in the next step without further purification.

Synthesis of 1-bromo-2-(bromomethyl)naphthalene (III). *N*-Bromosuccinimide (1 eq) and benzoylperoxide (0.01 eq) were added to a solution of (II) dissolved in CCl_4. The reaction was then heated to reflux and the reaction progress was monitored with GC/MS. The reaction seemed to stall out at 3 h, and an additional portion of benzoylperoxide (0.01 eq) was added and allowed to reflux for an additional 3 h. The succinimide byproduct was removed by filtration from the cooled mixture. The CCl_4 was removed and the product (III) was recrystallized from isooctane.

Synthesis of the title compound (IV). KCN (1.1 eq) was dissolved in DMSO with stirring. III (1.0 eq) was added along with additional DMSO to the stirred reaction mixture. A slight exotherm was observed, and the homogeneous mixture was allowed to stir overnight. The reaction was judged to be complete by GC/MS analysis. The reaction mixture was poured into water with stirring. The product precipitated upon addition to water. After filtering, the product was dried on a watch glass, and crystals for the diffraction study were obtained by recrystallization from a 2:1 mixture of 1,2-dimethoxyethane and ethanol.

Refinement

H atoms were constrained using a riding model. The methylene C—H bond lengths were fixed at 0.97 Å, using an idealized tetrahedral geometry, with $U_{iso}(H) = 1.2 U_{eq.} (C)$. The aromatic C—H bond lengths were fixed at 0.93 Å, with $U_{iso}(H) = 1.2 U_{eq.} (C)$.

Figures

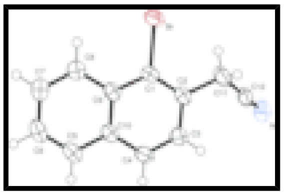
Fig. 1. View of the title compound (IV) showing 50% probability displacement ellipsoids.

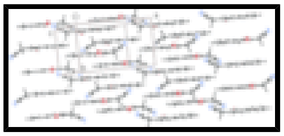
Fig. 2. Diagram showing how the molecules of (IV) pack in alternating layers when viewed edge-on.

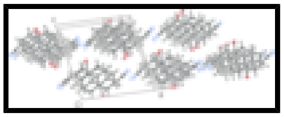
Fig. 3. Diagram showing how the molecules of (IV) form columns when viewed along the *b* axis.


Fig. 4. The formation of the title compound.

(1-Bromonaphthalen-2-yl)acetonitrile

Crystal data

$C_{12}H_8Br_1N_1$

M_r = 246.1

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

a = 11.3599 (13) Å

b = 7.2379 (8) Å

c = 11.8901 (15) Å

β = 102.538 (10)°

V = 954.31 (19) Å^3

Z = 4

F_{000} = 488

D_x = 1.713 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.54184 Å

Cell parameters from 22 reflections

θ = 8.6–16.7°

μ = 5.47 mm^{-1}

T = 295 (2) K

Prism, yellow

0.5 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer

Nonprofiled θ/2θ scans

Absorption correction: none

6502 measured reflections

1729 independent reflections

1558 reflections with $I > 2\sigma(I)$

R_{int} = 0.031

θ_{max} = 67.5°

θ_{min} = 4.9°

$h = -13 \rightarrow 13$

$k = -8 \rightarrow 8$

$l = -14 \rightarrow 14$

3 standard reflections

every 75 reflections

intensity decay: 2%

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 0.7384P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.025$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.063$	$\Delta\rho_{max} = 0.35$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -0.51$ e Å^{-3}
1729 reflections	Extinction correction: none
127 parameters	

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.22261 (2)	0.12936 (4)	0.490726 (19)	0.04829 (11)
N	−0.2239 (2)	−0.0251 (4)	0.2063 (2)	0.0621 (6)
C2	0.0751 (2)	0.1301 (3)	0.2652 (2)	0.0364 (5)
C9	0.29263 (19)	0.1292 (3)	0.2734 (2)	0.0339 (4)
C1	0.1920 (2)	0.1289 (3)	0.32691 (18)	0.0334 (4)
C10	0.2689 (2)	0.1334 (3)	0.1517 (2)	0.0353 (5)
C4	0.1481 (2)	0.1373 (3)	0.0887 (2)	0.0416 (5)
H4	0.132	0.1414	0.0087	0.05*
C8	0.4149 (2)	0.1250 (3)	0.3346 (2)	0.0421 (5)
H8	0.4327	0.1222	0.4147	0.051*
C12	−0.1394 (2)	0.0450 (3)	0.2562 (2)	0.0434 (5)
C11	−0.0312 (2)	0.1321 (4)	0.3232 (2)	0.0485 (6)
H11A	−0.0082	0.0694	0.3968	0.058*
H11B	−0.0498	0.2593	0.3384	0.058*
C3	0.0552 (2)	0.1353 (3)	0.1439 (2)	0.0416 (5)
H3	−0.0235	0.1373	0.1007	0.05*
C5	0.3660 (2)	0.1332 (3)	0.0944 (2)	0.0435 (5)
H5	0.3506	0.1351	0.0143	0.052*
C7	0.5062 (2)	0.1252 (3)	0.2767 (2)	0.0488 (6)
H7	0.5857	0.1219	0.3181	0.059*
C6	0.4826 (2)	0.1302 (3)	0.1566 (2)	0.0484 (6)
H6	0.5462	0.1314	0.1187	0.058*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.05003 (18)	0.06614 (18)	0.03112 (16)	−0.00544 (11)	0.01412 (12)	−0.00341 (10)
N	0.0373 (12)	0.0871 (17)	0.0637 (15)	−0.0030 (12)	0.0149 (11)	−0.0019 (13)
C2	0.0366 (11)	0.0376 (10)	0.0387 (12)	−0.0030 (8)	0.0163 (10)	−0.0017 (9)
C9	0.0362 (11)	0.0297 (9)	0.0391 (11)	−0.0019 (8)	0.0152 (10)	−0.0016 (8)
C1	0.0398 (11)	0.0328 (10)	0.0302 (10)	−0.0025 (8)	0.0133 (9)	−0.0015 (8)
C10	0.0393 (11)	0.0323 (10)	0.0383 (12)	−0.0023 (8)	0.0170 (10)	0.0000 (8)
C4	0.0449 (13)	0.0510 (13)	0.0311 (11)	−0.0025 (10)	0.0132 (10)	0.0018 (9)
C8	0.0389 (12)	0.0459 (12)	0.0421 (13)	−0.0006 (9)	0.0099 (10)	0.0001 (10)
C12	0.0353 (12)	0.0531 (13)	0.0465 (13)	0.0060 (11)	0.0195 (11)	0.0062 (11)
C11	0.0406 (13)	0.0632 (15)	0.0476 (14)	−0.0070 (11)	0.0222 (12)	−0.0079 (11)
C3	0.0346 (11)	0.0530 (13)	0.0380 (12)	−0.0023 (10)	0.0092 (10)	0.0011 (10)
C5	0.0496 (14)	0.0430 (12)	0.0451 (13)	−0.0010 (10)	0.0260 (12)	0.0002 (10)
C7	0.0336 (12)	0.0531 (13)	0.0609 (16)	0.0003 (10)	0.0130 (12)	−0.0011 (11)
C6	0.0413 (13)	0.0485 (13)	0.0636 (17)	−0.0002 (10)	0.0292 (13)	0.0008 (11)

Geometric parameters (Å, °)

Br—C1	1.903 (2)	C8—C7	1.364 (3)
N—C12	1.132 (3)	C8—H8	0.93
C2—C1	1.371 (3)	C12—C11	1.455 (4)
C2—C3	1.410 (3)	C11—H11A	0.97
C2—C11	1.515 (3)	C11—H11B	0.97
C9—C10	1.413 (3)	C3—H3	0.93
C9—C1	1.424 (3)	C5—C6	1.370 (4)
C9—C8	1.422 (3)	C5—H5	0.93
C10—C5	1.417 (3)	C7—C6	1.396 (4)
C10—C4	1.413 (3)	C7—H7	0.93
C4—C3	1.358 (3)	C6—H6	0.93
C4—H4	0.93		
C1—C2—C3	117.95 (19)	C12—C11—C2	114.1 (2)
C1—C2—C11	122.1 (2)	C12—C11—H11A	108.7
C3—C2—C11	119.9 (2)	C2—C11—H11A	108.7
C10—C9—C1	117.7 (2)	C12—C11—H11B	108.7
C10—C9—C8	118.25 (19)	C2—C11—H11B	108.7
C1—C9—C8	124.1 (2)	H11A—C11—H11B	107.6
C2—C1—C9	122.6 (2)	C4—C3—C2	121.7 (2)
C2—C1—Br	119.22 (15)	C4—C3—H3	119.1
C9—C1—Br	118.16 (17)	C2—C3—H3	119.1
C5—C10—C9	119.7 (2)	C6—C5—C10	120.2 (2)
C5—C10—C4	120.9 (2)	C6—C5—H5	119.9
C9—C10—C4	119.36 (19)	C10—C5—H5	119.9
C3—C4—C10	120.7 (2)	C8—C7—C6	121.2 (2)
C3—C4—H4	119.6	C8—C7—H7	119.4
C10—C4—H4	119.6	C6—C7—H7	119.4

C7—C8—C9	120.5 (2)	C5—C6—C7	120.1 (2)
C7—C8—H8	119.8	C5—C6—H6	120
C9—C8—H8	119.8	C7—C6—H6	120
N—C12—C11	178.4 (3)		

Fig. 1

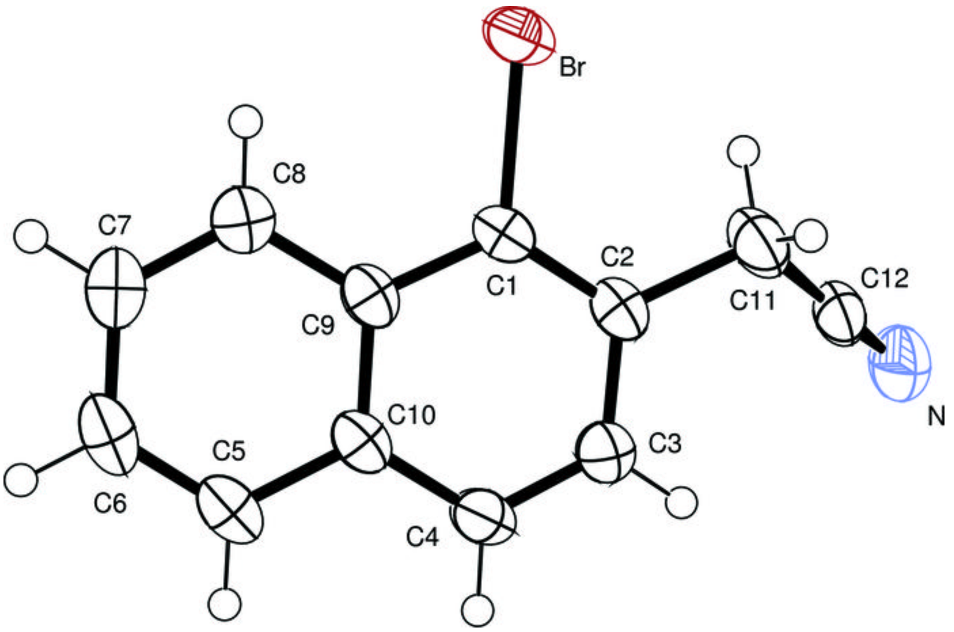

Fig. 2

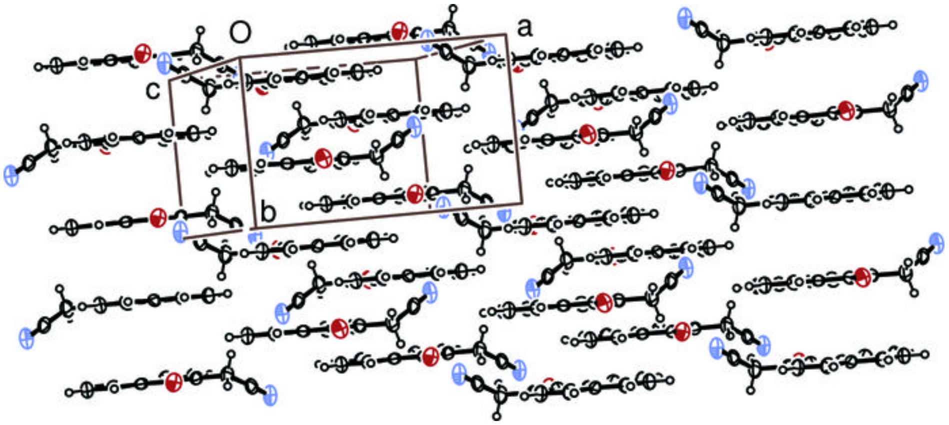

Fig. 3

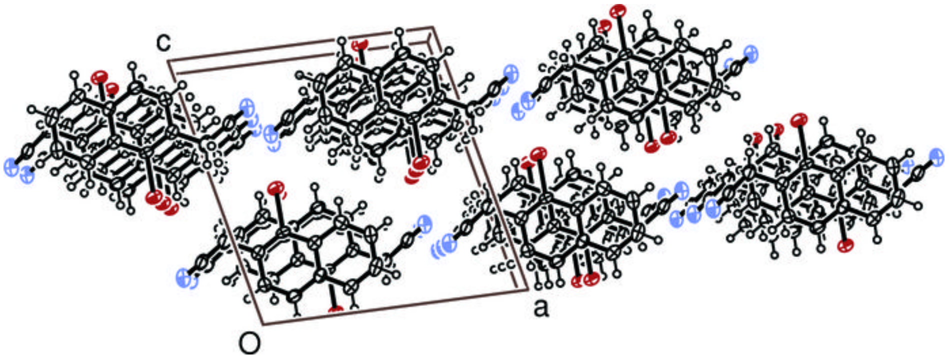

Fig. 4