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5-Amino-7-(4-bromophenyl)indane-4,6-dicarbonitrile

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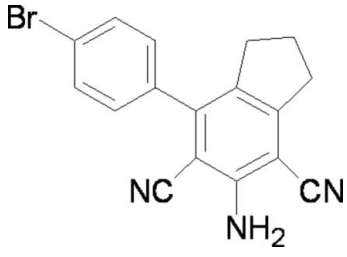
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.030; wR factor = 0.063; data-to-parameter ratio = 13.7.

In the title molecule, $C_{17}H_{12}BrN_3$, the mean planes of the bicyclic system and the attached aromatic ring form a dihedral angle of 63.12 (7)°. In the crystal structure, weak intermolecular N—H···N hydrogen bonds link adjacent molecules into ribbons extending along [010].

Related literature

Analogous compounds have been synthesized and reported by Hafidh *et al.* (2002) and Hafidh & Zantour (2003). For a related structure, see Mereiter *et al.* (2000).



Experimental

Crystal data

$C_{17}H_{12}BrN_3$
M_r = 338.21
Orthorhombic, $P2_12_12_1$
a = 7.5655 (14) Å
b = 11.811 (2) Å
c = 16.490 (3) Å
V = 1473.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.79 mm^{-1}
T = 298 K
0.35 × 0.28 × 0.20 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.442, T_{max} = 0.606
7199 measured reflections
2708 independent reflections
2171 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.063
S = 0.95
2708 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack (1983), 1253 Friedel pairs
Flack parameter: 0.008 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···N2^{i}	0.85 (3)	2.40 (3)	3.227 (4)	165 (3)
N1—H1A···N3^{ii}	0.82 (3)	2.46 (3)	3.247 (4)	161 (2)

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2633).

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supporting information

Acta Cryst. (2009). E**65**, o2875 [https://doi.org/10.1107/S1600536809043426]

5-Amino-7-(4-bromophenyl)indane-4,6-dicarbonitrile

Cunlan Zhang

S1. Comment

indanee derivatives have attracted some attention in a search of novel functional compounds (Hafidh *et al.* 2002; Hafidh & Zantour, 2003). As our contribution to this field, we present here the title compound, (I).

In (I) (Fig. 1), all bond lengths and angles are normal and correspond to those observed in related compound (Mereiter *et al.*, 2000). The mean plane of the bicycle system (C7/C8/C10/C11/C13-C17) and attached aromatic ring (C1-C6) form a dihedral angle of 63.12 (7)°.

In the crystal structure, weak intermolecular N—H···N hydrogen bonds (Table 1) link adjacent molecules into ribbons extended in direction [010].

S2. Experimental

The malononitrile (1.32 g,20 mmol) was added into the mixture of 4-bromobenzaldehyde (1.85 g,10 mmol) and cyclopentanone (0.84 g,10 mmol) in 1-butyl-3-methylimidazol-3-ium tetrafluoroborate (20 ml), and has stirred for three hours at 388 K. The solution was allowed to stand for 2 weeks, whereupon the crystals suitable for the *X*-ray study was obtained. Yield: 1.047 g, 31%. Anal. for $C_{17}H_{12}BrN_3$: Calc. C, 60.37; H, 3.58; N, 12.42; Found: C, 60.54; H, 3.71; N, 12.54%. The No. of CCDC: 750008.

S3. Refinement

C-bound H atoms were placed in geometrically idealized positions (C—H 0.93-0.97 Å) and treated as riding on their parent atoms , with U_{iso}(H)=1.2U_{eq}(C). Amino H atoms were located on a difference map and refined isotropically with the bond restraint of N—H = 0.84 (3) Å.

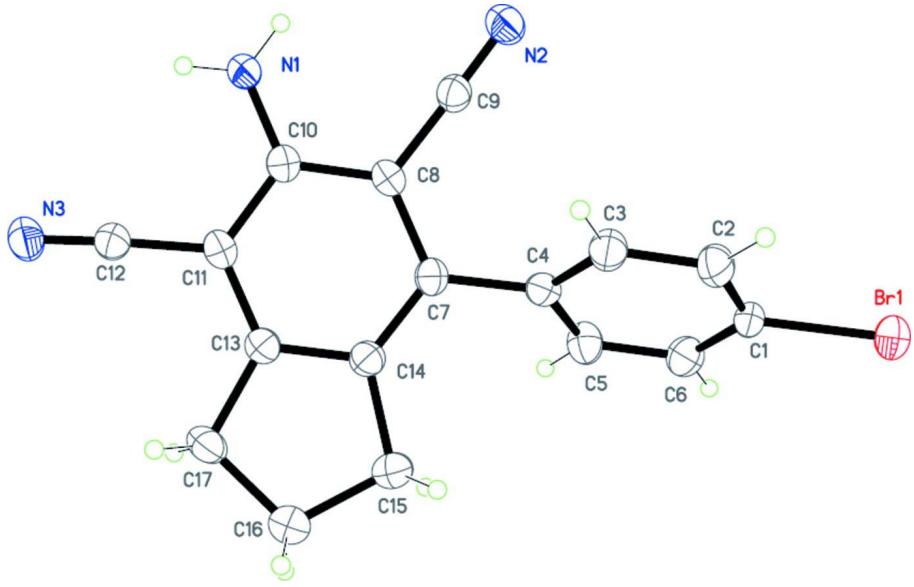

Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

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Crystal data

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Orthorhombic, $P2_12_12_1$
$a = 7.5655\ (14)$ Å
$b = 11.811\ (2)$ Å
$c = 16.490\ (3)$ Å
$V = 1473.5\ (5)$ Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.525$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2622 reflections
$\theta = 2.5$–$23.4°$
$\mu = 2.79$ mm^{-1}
$T = 298$ K
Block, colourless
0.35 × 0.28 × 0.20 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.442$, $T_{max} = 0.606$
7199 measured reflections
2708 independent reflections
2171 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.1°$
$h = -9\rightarrow8$
$k = -14\rightarrow9$
$l = -15\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.063$
$S = 0.95$
2708 reflections
198 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 atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0269P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Absolute structure: Flack (1983), 1253 Friedel pairs
Absolute structure parameter: 0.008 (10)

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 $F^2 > \sigma(F^2)$ 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}
Br1	0.61120 (5)	0.10058 (3)	0.17284 (2)	0.06606 (14)
C1	0.6025 (5)	0.2417 (2)	0.11713 (14)	0.0449 (7)
C2	0.7401 (4)	0.2717 (3)	0.06832 (18)	0.0530 (8)
H2	0.8383	0.2249	0.0633	0.064*
C3	0.7311 (4)	0.3727 (3)	0.02640 (18)	0.0484 (8)
H3	0.8243	0.3937	−0.0072	0.058*
C4	0.5870 (4)	0.4429 (2)	0.03340 (15)	0.0396 (7)
C5	0.4503 (4)	0.4104 (3)	0.08498 (17)	0.0501 (7)
H5	0.3533	0.4579	0.0918	0.060*
C6	0.4572 (4)	0.3083 (3)	0.12627 (18)	0.0521 (8)
H6	0.3643	0.2857	0.1595	0.062*
C7	0.5761 (4)	0.5533 (2)	−0.00930 (15)	0.0374 (6)
C8	0.5789 (4)	0.5572 (2)	−0.09608 (16)	0.0390 (7)
C9	0.5889 (4)	0.4534 (2)	−0.14014 (16)	0.0418 (7)
C10	0.5655 (4)	0.6602 (2)	−0.13850 (16)	0.0398 (7)
C11	0.5607 (4)	0.7613 (2)	−0.09254 (16)	0.0405 (7)
C12	0.5519 (4)	0.8699 (3)	−0.13046 (17)	0.0449 (8)
C13	0.5621 (4)	0.7567 (2)	−0.00771 (16)	0.0403 (7)
C14	0.5666 (3)	0.6538 (2)	0.03272 (15)	0.0399 (7)
C15	0.5628 (4)	0.6735 (3)	0.12311 (16)	0.0524 (8)
H15A	0.6629	0.6373	0.1492	0.063*
H15B	0.4546	0.6441	0.1466	0.063*
C16	0.5724 (5)	0.8015 (3)	0.13270 (18)	0.0554 (9)
H16A	0.4762	0.8278	0.1667	0.066*
H16B	0.6832	0.8228	0.1580	0.066*
C17	0.5589 (4)	0.8542 (3)	0.04933 (17)	0.0507 (8)
H17A	0.4498	0.8966	0.0438	0.061*
H17B	0.6578	0.9045	0.0393	0.061*
H1A	0.557 (3)	0.605 (3)	−0.2469 (16)	0.042 (8)*
H1B	0.538 (4)	0.724 (3)	−0.2445 (18)	0.052 (10)*
N1	0.5612 (4)	0.6630 (3)	−0.22009 (15)	0.0564 (8)

N2	0.5985 (4)	0.3714 (2)	−0.17670 (16)	0.0587 (7)
N3	0.5450 (4)	0.9583 (2)	−0.15788 (15)	0.0635 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1015 (3)	0.04187 (17)	0.05480 (18)	0.0033 (2)	0.0026 (2)	0.01060 (16)
C1	0.067 (2)	0.0317 (15)	0.0355 (14)	0.0015 (18)	−0.0058 (16)	0.0039 (11)
C2	0.060 (2)	0.046 (2)	0.0525 (19)	0.0155 (17)	0.0067 (16)	0.0055 (16)
C3	0.0497 (19)	0.046 (2)	0.0489 (18)	0.0075 (15)	0.0097 (14)	0.0064 (15)
C4	0.0448 (18)	0.0371 (15)	0.0368 (14)	−0.0012 (15)	0.0014 (14)	−0.0036 (12)
C5	0.0471 (18)	0.0439 (18)	0.0593 (18)	0.0037 (15)	0.0043 (14)	0.0052 (16)
C6	0.058 (2)	0.049 (2)	0.0496 (18)	−0.0066 (17)	0.0089 (15)	0.0064 (15)
C7	0.0343 (16)	0.0352 (15)	0.0428 (15)	−0.0003 (13)	−0.0004 (12)	0.0031 (12)
C8	0.0408 (17)	0.0320 (14)	0.0443 (15)	0.0018 (13)	−0.0001 (13)	−0.0031 (12)
C9	0.0455 (18)	0.0389 (17)	0.0412 (15)	0.0004 (17)	−0.0042 (14)	0.0032 (14)
C10	0.0401 (18)	0.0361 (16)	0.0431 (15)	−0.0013 (14)	−0.0004 (13)	0.0014 (13)
C11	0.0409 (18)	0.0325 (15)	0.0481 (16)	−0.0012 (13)	−0.0022 (13)	−0.0009 (13)
C12	0.055 (2)	0.0398 (18)	0.0402 (16)	−0.0030 (14)	−0.0039 (14)	0.0005 (14)
C13	0.0423 (18)	0.0380 (16)	0.0405 (15)	0.0023 (14)	0.0009 (13)	−0.0014 (13)
C14	0.0408 (17)	0.0400 (16)	0.0389 (15)	0.0048 (13)	0.0018 (13)	0.0001 (13)
C15	0.066 (2)	0.052 (2)	0.0387 (15)	0.0052 (17)	0.0024 (15)	−0.0013 (14)
C16	0.069 (2)	0.0491 (19)	0.0478 (16)	0.0015 (18)	0.0004 (16)	−0.0055 (15)
C17	0.062 (2)	0.0390 (16)	0.0510 (17)	0.0047 (15)	−0.0014 (15)	−0.0112 (14)
N1	0.099 (3)	0.0337 (16)	0.0359 (16)	0.0023 (17)	−0.0036 (15)	−0.0014 (13)
N2	0.0819 (18)	0.0439 (15)	0.0501 (14)	0.0088 (15)	−0.0110 (17)	−0.0058 (13)
N3	0.096 (2)	0.0393 (16)	0.0557 (16)	−0.0058 (15)	−0.0125 (15)	0.0036 (13)

Geometric parameters (Å, º)

Br1—C1	1.905 (3)	C10—C11	1.415 (4)
C1—C6	1.360 (4)	C11—C13	1.400 (4)
C1—C2	1.363 (4)	C11—C12	1.428 (4)
C2—C3	1.381 (4)	C12—N3	1.139 (4)
C2—H2	0.9300	C13—C14	1.387 (4)
C3—C4	1.375 (4)	C13—C17	1.487 (4)
C3—H3	0.9300	C14—C15	1.509 (4)
C4—C5	1.393 (4)	C15—C16	1.521 (4)
C4—C7	1.484 (4)	C15—H15A	0.9700
C5—C6	1.386 (4)	C15—H15B	0.9700
C5—H5	0.9300	C16—C17	1.513 (4)
C6—H6	0.9300	C16—H16A	0.9700
C7—C14	1.376 (4)	C16—H16B	0.9700
C7—C8	1.432 (4)	C17—H17A	0.9700
C8—C10	1.408 (4)	C17—H17B	0.9700
C8—C9	1.426 (4)	N1—H1A	0.82 (3)
C9—N2	1.144 (3)	N1—H1B	0.85 (3)
C10—N1	1.346 (4)		

C6—C1—C2	122.2 (3)	C10—C11—C12	121.6 (2)
C6—C1—Br1	118.7 (2)	N3—C12—C11	177.4 (3)
C2—C1—Br1	119.1 (3)	C14—C13—C11	121.0 (3)
C1—C2—C3	118.8 (3)	C14—C13—C17	112.0 (2)
C1—C2—H2	120.6	C11—C13—C17	127.0 (3)
C3—C2—H2	120.6	C7—C14—C13	121.0 (2)
C4—C3—C2	121.2 (3)	C7—C14—C15	129.2 (3)
C4—C3—H3	119.4	C13—C14—C15	109.8 (2)
C2—C3—H3	119.4	C14—C15—C16	104.8 (2)
C3—C4—C5	118.3 (3)	C14—C15—H15A	110.8
C3—C4—C7	122.3 (2)	C16—C15—H15A	110.8
C5—C4—C7	119.4 (3)	C14—C15—H15B	110.8
C6—C5—C4	120.8 (3)	C16—C15—H15B	110.8
C6—C5—H5	119.6	H15A—C15—H15B	108.9
C4—C5—H5	119.6	C17—C16—C15	108.1 (2)
C1—C6—C5	118.6 (3)	C17—C16—H16A	110.1
C1—C6—H6	120.7	C15—C16—H16A	110.1
C5—C6—H6	120.7	C17—C16—H16B	110.1
C14—C7—C8	118.5 (2)	C15—C16—H16B	110.1
C14—C7—C4	121.4 (2)	H16A—C16—H16B	108.4
C8—C7—C4	120.1 (2)	C13—C17—C16	104.8 (2)
C10—C8—C9	119.6 (2)	C13—C17—H17A	110.8
C10—C8—C7	121.5 (2)	C16—C17—H17A	110.8
C9—C8—C7	118.9 (2)	C13—C17—H17B	110.8
N2—C9—C8	178.7 (3)	C16—C17—H17B	110.8
N1—C10—C8	121.3 (3)	H17A—C17—H17B	108.9
N1—C10—C11	121.0 (3)	C10—N1—H1A	121 (2)
C8—C10—C11	117.7 (2)	C10—N1—H1B	120 (2)
C13—C11—C10	120.1 (3)	H1A—N1—H1B	117 (3)
C13—C11—C12	118.3 (3)		

C6—C1—C2—C3	−0.4 (5)	N1—C10—C11—C13	178.8 (3)
Br1—C1—C2—C3	178.3 (2)	C8—C10—C11—C13	−2.5 (4)
C1—C2—C3—C4	0.2 (5)	N1—C10—C11—C12	−0.2 (5)
C2—C3—C4—C5	0.9 (4)	C8—C10—C11—C12	178.4 (3)
C2—C3—C4—C7	178.4 (3)	C13—C11—C12—N3	2 (8)
C3—C4—C5—C6	−2.0 (4)	C10—C11—C12—N3	−179 (100)
C7—C4—C5—C6	−179.5 (3)	C10—C11—C13—C14	−0.5 (4)
C2—C1—C6—C5	−0.6 (5)	C12—C11—C13—C14	178.6 (3)
Br1—C1—C6—C5	−179.3 (2)	C10—C11—C13—C17	179.5 (3)
C4—C5—C6—C1	1.8 (4)	C12—C11—C13—C17	−1.4 (5)
C3—C4—C7—C14	−116.8 (3)	C8—C7—C14—C13	−0.8 (4)
C5—C4—C7—C14	60.6 (4)	C4—C7—C14—C13	178.0 (3)
C3—C4—C7—C8	62.0 (4)	C8—C7—C14—C15	−179.7 (3)
C5—C4—C7—C8	−120.6 (3)	C4—C7—C14—C15	−0.9 (5)
C14—C7—C8—C10	−2.4 (4)	C11—C13—C14—C7	2.3 (4)
C4—C7—C8—C10	178.8 (3)	C17—C13—C14—C7	−177.8 (3)

C14—C7—C8—C9	179.9 (3)	C11—C13—C14—C15	−178.7 (3)
C4—C7—C8—C9	1.1 (4)	C17—C13—C14—C15	1.3 (3)
C10—C8—C9—N2	28 (15)	C7—C14—C15—C16	173.9 (3)
C7—C8—C9—N2	−154 (15)	C13—C14—C15—C16	−5.0 (3)
C9—C8—C10—N1	0.3 (5)	C14—C15—C16—C17	6.8 (3)
C7—C8—C10—N1	−177.4 (3)	C14—C13—C17—C16	3.1 (3)
C9—C8—C10—C11	−178.3 (3)	C11—C13—C17—C16	−177.0 (3)
C7—C8—C10—C11	4.0 (4)	C15—C16—C17—C13	−6.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···N2^{i}	0.85 (3)	2.40 (3)	3.227 (4)	165 (3)
N1—H1*A*···N3^{ii}	0.82 (3)	2.46 (3)	3.247 (4)	161 (2)

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