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5-Amino-4-bromo-2,3-dihydro-1*H*-inden-1-one

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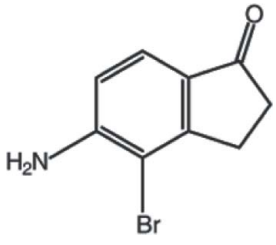
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.005 Å; R factor = 0.049; wR factor = 0.134; data-to-parameter ratio = 13.6.

In the title compound, C_9H_8BrNO, the non-H-atom framework is essentially planar, with a maximum deviation of 0.087 (3) Å. In the crystal, molecules are interconnected into a three-dimensional network by C—H···O and N—H···O hydrogen bonds. In addition, C—H···π interactions and a π–π stacking interaction, with a centroid–centroid distance of 3.5535 (19) Å, are also observed.

Related literature

For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

C_9H_8BrNO
M_r = 226.06
Monoclinic, $C2/c$
a = 12.6362 (4) Å
b = 8.3655 (2) Å
c = 17.4913 (5) Å
β = 113.128 (4)°
V = 1700.37 (10) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 6.16 mm^{-1}
T = 297 K
0.77 × 0.60 × 0.08 mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.031, T_{max} = 0.616
3085 measured reflections
1594 independent reflections
1544 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.134
S = 1.06
1594 reflections
117 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.89 e Å^{-3}

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

*Cg*2 is the centroid of the C1–C6 benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.83 (4)	2.14 (5)	2.915 (4)	155 (4)
C8—H8*B*···O1^{ii}	0.97	2.50	3.448 (4)	166
C7—H7*B*···*Cg*2^{iii}	0.97	2.85	3.659 (4)	141

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

Data collection: *CrysAlis PRO* (Agilent, 2011); 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: *ORTEP-3 for Windows* (Farrugia, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1997) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2519).

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supplementary materials

Acta Cryst. (2012). E**68**, o833 [doi:10.1107/S1600536812007489]

5-Amino-4-bromo-2,3-dihydro-1*H*-inden-1-one

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Comment

In ^{1}H-NMR spectrum, H7 appeared at δ 7.53 as a doublet with coupling constant 8.3 Hz and H6 appeared at δ 6.73 (J = 8.3 Hz) as doublet. NH2 protons were observed at δ 4.82 with broad singlet. Two signal groups (δ 2.98, δ 2.70) observed at aliphatic region fit with the aliphatic protons. In the present study, we describe the molecular and crystal structures of 5-amino-4-bromo-2,3-dihydro-1*H*-inden-1-one (I), using X-ray diffraction.

As shown in Fig. 1, the molecule of (I), except H atoms, is essentially planar with a maximum deviation of -0.087 (3) Å for O1 atom. Bond lengths and angles observed in (I) are normal (Allen *et al.*, 1987).

The crystal packing is stabilized by intermolecular C—H···O and N—H···O hydrogen bonds (Table 1, Fig. 2) forming a three-dimensional network. In addition, π-π stacking interactions [centroid-centroid distance = 3.5535 (19) Å] between the centroids of the C1–C6 benzene rings of the neighbouring molecules stacking interactions are also observed. C—H···π interactions further help in stabilizing the supramolecular structure (Table 1).

Experimental

To a stirred solution of 5- acetoaminoindanone (0.4 g, 1.95 mmol) in PEG (2.5 g) was added NBS (1.0 g, 5.6 mmol), SiO_2 (1.0 g) and $NaClO_4$ (0.2 g). The reaction mixture was stirred for 30 days at room temperature, diluted with water (15 ml), extracted with diethyl ether (3×25 ml), dried (Na_2SO_4). After removal of the solvent, the residue was chromatographed on silica gel eluted with chloroform/hexane (4/1) afforded the title compound which was crystallized from dichloromethane-hexane yielded the colourless plate crystal (0.11 g, 25%), ^{1}H-NMR (400 MHz, $CDCl_3$); δ 7.53 (d, J = 8.3 Hz, 1H, H7), 6.74 (d, J = 8.3 Hz, 1H, H6), 4.82 (brs, 2H, NH_2), 2.98 (m, 2H, H2), 2.70 (m, 2H, H3).

Refinement

The H atoms of the amino group was located in a difference Fourier map and were isotropically refined with the distance restraints (N—H = 0.86 (2) Å and H···H = 1.30 (2) Å). C-bound H-atoms were positioned geometrically and refined using a riding model [C—H = 0.93 and 0.97 Å, and U_{iso}(H) = 1.2U_{eq}(C)]. The highest residual electron density peak and the deepest hole are located 1.03 Å and 0.89 Å from Br1, respectively.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1997) and *PLATON* (Spek, 2009).

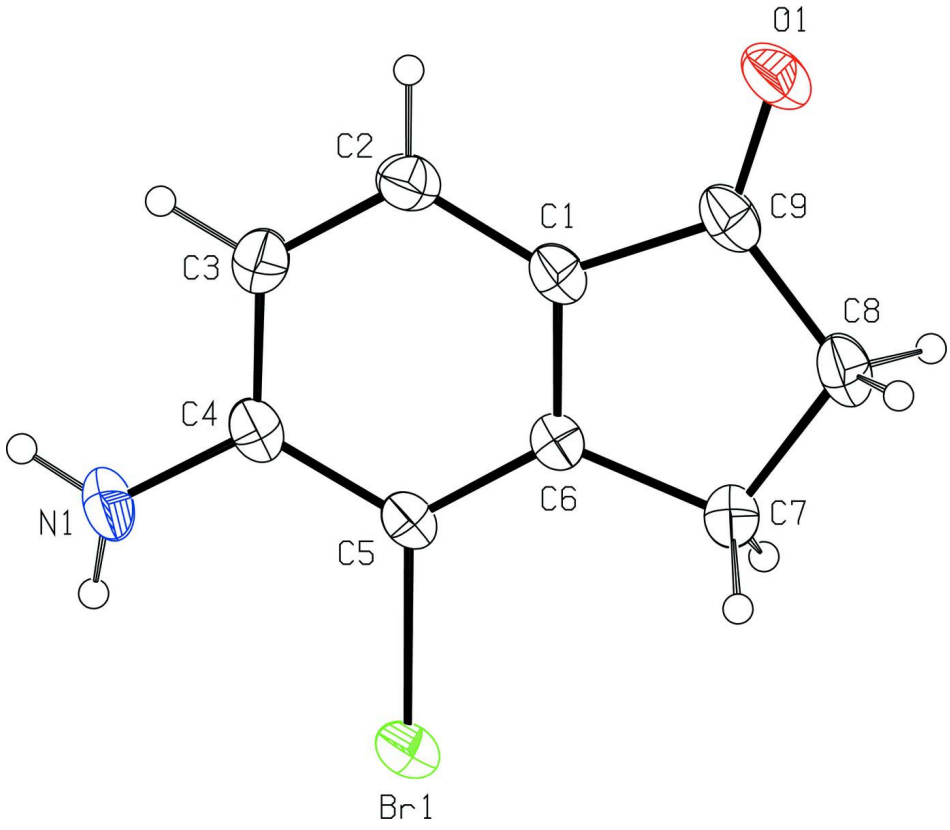

Figure 1

The molecular structure of (I) with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

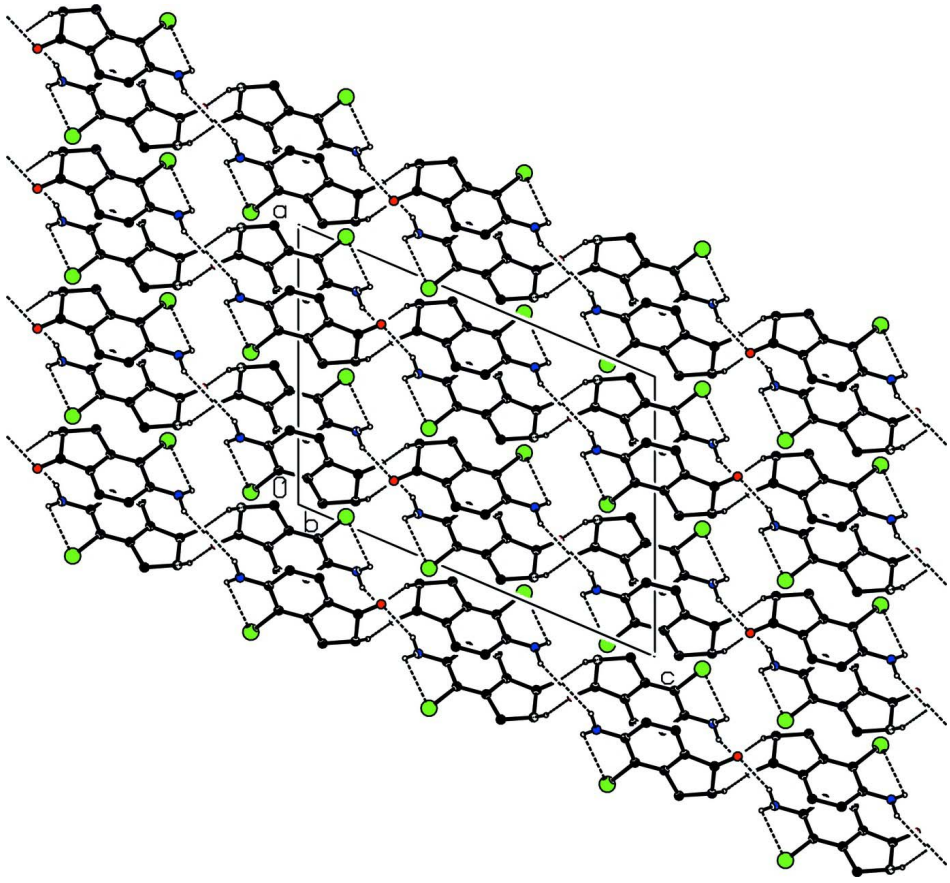

Figure 2

The packing and hydrogen bonding of (I), viewing down the *b* axis. H atoms not involved in hydrogen bonding have been omitted.

5-Amino-4-bromo-2,3-dihydro-1*H*-inden-1-one

Crystal data

C_9H_8BrNO
M_r = 226.06
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 12.6362 (4) Å
b = 8.3655 (2) Å
c = 17.4913 (5) Å
β = 113.128 (4)°
V = 1700.37 (10) Å^3
Z = 8

F(000) = 896
D_x = 1.766 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.5418 Å
Cell parameters from 2518 reflections
θ = 5.5–70.2°
μ = 6.16 mm^{-1}
T = 297 K
Plate, colourless
0.77 × 0.60 × 0.08 mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.2673 pixels mm^{-1}
ω scans

Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.031, T_{max} = 0.616
3085 measured reflections
1594 independent reflections
1544 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

θ_{max} = 70.4°, θ_{min} = 5.5°
h = −13→15
k = −6→10
l = −19→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.134
S = 1.06
1594 reflections
117 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.1063P)^2 + 1.0273P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.89 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.52857 (3)	0.21900 (4)	0.63347 (2)	0.0470 (2)
O1	0.2303 (2)	0.4628 (3)	0.26770 (13)	0.0544 (8)
N1	0.3501 (3)	0.4337 (4)	0.66390 (17)	0.0535 (9)
C1	0.3037 (3)	0.4330 (4)	0.41603 (17)	0.0381 (8)
C2	0.2318 (3)	0.5262 (4)	0.44160 (18)	0.0422 (8)
C3	0.2489 (3)	0.5253 (4)	0.5240 (2)	0.0431 (9)
C4	0.3357 (3)	0.4322 (4)	0.58293 (17)	0.0399 (8)
C5	0.4086 (2)	0.3422 (3)	0.55556 (16)	0.0368 (8)
C6	0.3917 (2)	0.3426 (3)	0.47291 (16)	0.0345 (8)
C7	0.4597 (3)	0.2542 (4)	0.4321 (2)	0.0430 (9)
C8	0.4016 (3)	0.3016 (4)	0.3399 (2)	0.0493 (10)
C9	0.3006 (3)	0.4086 (4)	0.33249 (17)	0.0425 (8)
H1N	0.301 (3)	0.472 (5)	0.679 (3)	0.062 (12)*
H2	0.17350	0.58760	0.40350	0.0510*
H2N	0.387 (4)	0.369 (6)	0.701 (3)	0.10 (2)*
H3	0.20180	0.58790	0.54150	0.0520*
H7A	0.45550	0.13960	0.43890	0.0520*
H7B	0.53980	0.28670	0.45540	0.0520*
H8A	0.45530	0.35870	0.32260	0.0590*
H8B	0.37490	0.20740	0.30530	0.0590*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0466 (3)	0.0552 (4)	0.0315 (3)	0.0060 (1)	0.0070 (2)	0.0028 (1)
O1	0.0541 (13)	0.0741 (16)	0.0303 (10)	−0.0051 (12)	0.0114 (10)	0.0072 (10)
N1	0.0619 (17)	0.0712 (18)	0.0322 (13)	0.0012 (15)	0.0237 (13)	−0.0070 (12)
C1	0.0412 (14)	0.0433 (13)	0.0290 (13)	−0.0056 (12)	0.0128 (11)	0.0001 (10)
C2	0.0393 (14)	0.0471 (15)	0.0368 (15)	0.0014 (12)	0.0114 (12)	0.0050 (12)
C3	0.0443 (15)	0.0451 (14)	0.0443 (16)	0.0013 (13)	0.0221 (13)	−0.0033 (12)
C4	0.0444 (14)	0.0451 (14)	0.0305 (14)	−0.0062 (12)	0.0152 (12)	−0.0046 (11)
C5	0.0386 (13)	0.0414 (14)	0.0274 (12)	−0.0026 (12)	0.0098 (10)	−0.0017 (10)
C6	0.0371 (13)	0.0362 (13)	0.0298 (13)	−0.0009 (10)	0.0126 (11)	−0.0008 (10)
C7	0.0438 (17)	0.0496 (13)	0.0376 (17)	0.0024 (14)	0.0182 (14)	−0.0045 (13)
C8	0.0578 (19)	0.0602 (18)	0.0344 (16)	−0.0064 (15)	0.0230 (15)	−0.0064 (13)
C9	0.0474 (15)	0.0501 (15)	0.0299 (14)	−0.0140 (13)	0.0151 (12)	−0.0006 (11)

Geometric parameters (Å, º)

Br1—C5	1.896 (3)	C5—C6	1.376 (4)
O1—C9	1.220 (4)	C6—C7	1.509 (5)
N1—C4	1.355 (4)	C7—C8	1.539 (5)
N1—H1N	0.83 (4)	C8—C9	1.522 (5)
N1—H2N	0.83 (5)	C2—H2	0.9300
C1—C9	1.461 (4)	C3—H3	0.9300
C1—C2	1.397 (5)	C7—H7A	0.9700
C1—C6	1.389 (4)	C7—H7B	0.9700
C2—C3	1.371 (4)	C8—H8A	0.9700
C3—C4	1.409 (5)	C8—H8B	0.9700
C4—C5	1.411 (5)		
H1N—N1—H2N	105 (5)	C7—C8—C9	106.3 (3)
C4—N1—H1N	122 (3)	O1—C9—C1	126.9 (3)
C4—N1—H2N	128 (3)	O1—C9—C8	125.3 (3)
C2—C1—C6	120.9 (3)	C1—C9—C8	107.8 (3)
C2—C1—C9	129.3 (3)	C1—C2—H2	121.00
C6—C1—C9	109.9 (3)	C3—C2—H2	121.00
C1—C2—C3	118.7 (3)	C2—C3—H3	119.00
C2—C3—C4	121.9 (3)	C4—C3—H3	119.00
C3—C4—C5	118.1 (3)	C6—C7—H7A	111.00
N1—C4—C5	121.5 (3)	C6—C7—H7B	111.00
N1—C4—C3	120.4 (3)	C8—C7—H7A	111.00
Br1—C5—C4	119.4 (2)	C8—C7—H7B	111.00
Br1—C5—C6	120.5 (2)	H7A—C7—H7B	109.00
C4—C5—C6	120.2 (2)	C7—C8—H8A	110.00
C1—C6—C5	120.3 (3)	C7—C8—H8B	111.00
C1—C6—C7	111.9 (3)	C9—C8—H8A	110.00
C5—C6—C7	127.9 (3)	C9—C8—H8B	110.00
C6—C7—C8	104.1 (3)	H8A—C8—H8B	109.00
C6—C1—C2—C3	−0.8 (5)	N1—C4—C5—Br1	0.6 (4)

C9—C1—C2—C3	177.4 (4)	N1—C4—C5—C6	179.8 (3)
C2—C1—C6—C5	0.7 (5)	C3—C4—C5—Br1	178.7 (2)
C2—C1—C6—C7	−178.9 (3)	C3—C4—C5—C6	−2.1 (5)
C9—C1—C6—C5	−177.8 (3)	Br1—C5—C6—C1	180.0 (2)
C9—C1—C6—C7	2.7 (4)	Br1—C5—C6—C7	−0.5 (4)
C2—C1—C9—O1	−2.6 (6)	C4—C5—C6—C1	0.8 (4)
C2—C1—C9—C8	177.6 (4)	C4—C5—C6—C7	−179.7 (3)
C6—C1—C9—O1	175.7 (3)	C1—C6—C7—C8	−0.1 (4)
C6—C1—C9—C8	−4.1 (4)	C5—C6—C7—C8	−179.7 (3)
C1—C2—C3—C4	−0.7 (5)	C6—C7—C8—C9	−2.4 (3)
C2—C3—C4—N1	−179.8 (4)	C7—C8—C9—O1	−175.9 (3)
C2—C3—C4—C5	2.1 (5)	C7—C8—C9—C1	4.0 (4)

Hydrogen-bond geometry (Å, º)

Cg2 is a centroid of the C1–C6 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.83 (4)	2.14 (5)	2.915 (4)	155 (4)
N1—H2*N*···Br1	0.83 (5)	2.80 (5)	3.088 (4)	103 (4)
C8—H8*B*···O1^{ii}	0.97	2.50	3.448 (4)	166
C7—H7*B*···*Cg2*^{iii}	0.97	2.85	3.659 (4)	141

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