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

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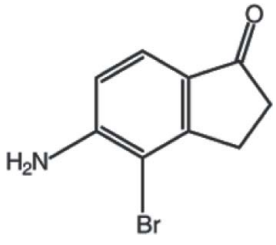
Key indicators: single-crystal X-ray study; $T$ = 297 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.049; $wR$ factor = 0.134; data-to-parameter ratio = 13.6.

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···$\pi$ interactions and a $\pi$–$\pi$ 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
$\beta$ = 113.128 (4)°
$V$ = 1700.37 (10) Å$^3$
$Z$ = 8
Cu $K\alpha$ radiation
$\mu$ = 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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# supporting information

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

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

**Ísmail Çelik, Mehmet Akkurt, Makbule Yılmaz, Ahmet Tutar, Ramazan Erenler and Santiago García-Granda**

### S1. Comment

In $^1$H-NMR spectrum, H7 appeared at $\delta$ 7.53 as a doublet with coupling constant 8.3 Hz and H6 appeared at $\delta$ 6.73 ($J$ = 8.3 Hz) as doublet. NH2 protons were observed at $\delta$ 4.82 with broad singlet. Two signal groups ($\delta$ 2.98, $\delta$ 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, $\pi$-$\pi$ 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···$\pi$ interactions further help in stabilizing the supramolecular structure (Table 1).

### S2. 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, $CDCl_3$); $\delta$ 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).

### S3. 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) = 1.2$U_{eq}$(C)]. The highest residual electron density peak and the deepest hole are located 1.03 Å and 0.89 Å from Br1, respectively.

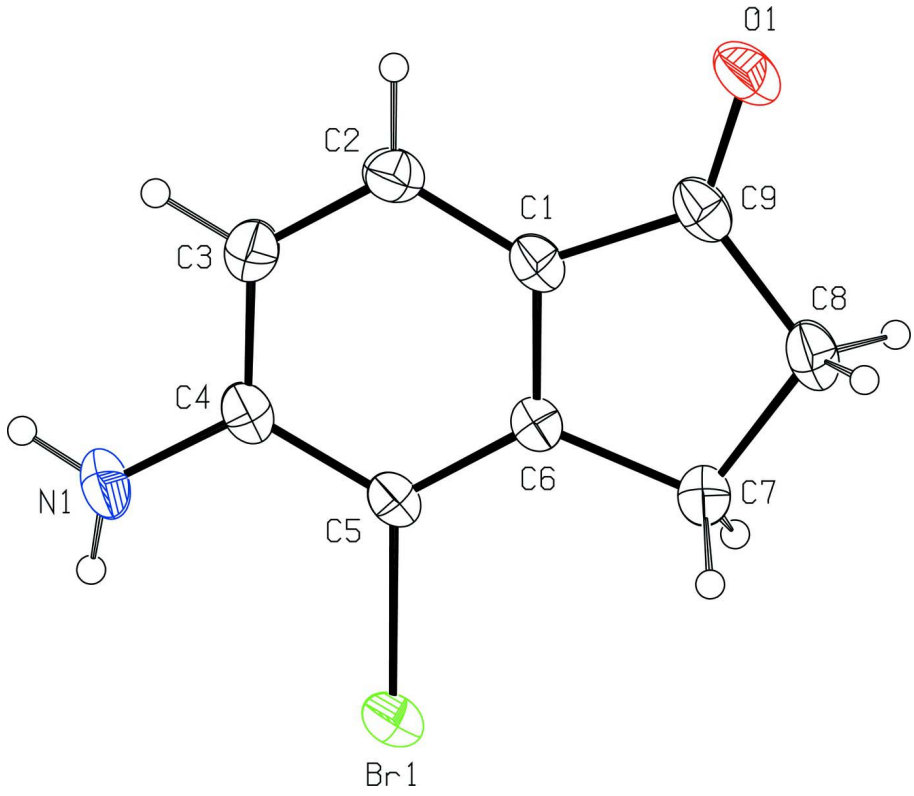


**Figure 1**

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

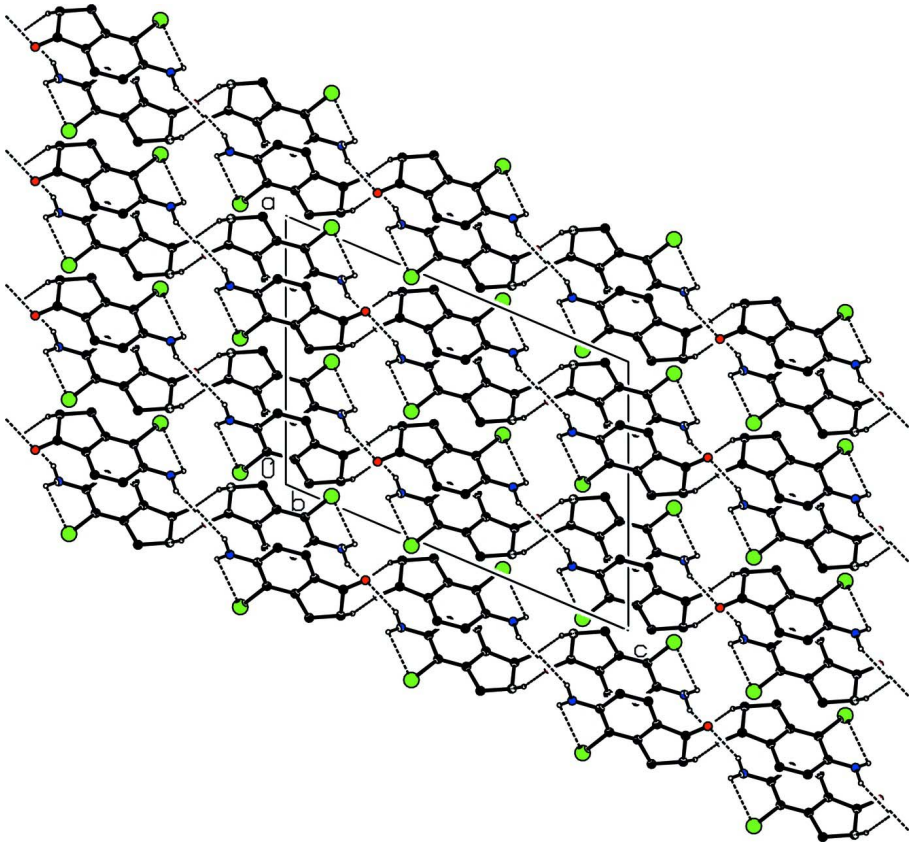


**Figure 2**

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
$\beta = 113.128\ (4)°$
$V = 1700.37\ (10)$ Å$^3$
$Z = 8$
$F(000) = 896$
$D_x = 1.766$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
Cell parameters from 2518 reflections
$\theta = 5.5–70.2°$
$\mu = 6.16$ 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$^{-1}$
$\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.026$

$\theta_{max}$ = 70.4°, $\theta_{min}$ = 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 (Å$^2$)*

| | *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*···*Cg*2$^{iii}$ | 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.