Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 5-Bromo-3-(indan-1-yloxy)pyridin-2-amine

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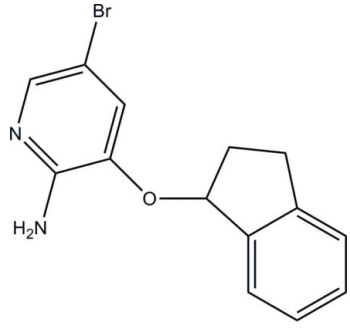
Received 29 January 2011; accepted 13 February 2011

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.023; $wR$ factor = 0.054; data-to-parameter ratio = 18.0.

The title compound, $C_{14}H_{13}BrN_2O$, was obtained by reaction of indan-1-yl methanesulfonate with 2-amino-5-bromopyridin-3-ol in the presence of caesium carbonate. The indane ring system is approximately planar [all but one of the C atoms are coplanar within 0.03 Å, the latter atom being displaced by 0.206 (2) Å from the mean plane through the remaining atoms] and forms a dihedral angle of 58.41 (4)° with the pyridine ring. In the crystal, centrosymmetrically related molecules are linked into dimers by N—H···N hydrogen bonds.

## Related literature

For related structures with an indane group linked to a pyridine derivative through a C—O—C bridge, see: Dinçer *et al.* (2004); Lifshits *et al.* (2008).




## Experimental

### Crystal data

$C_{14}H_{13}BrN_2O$
$M_r$ = 305.17
Monoclinic, $P2_1/n$
$a$ = 11.3944 (18) Å
$b$ = 9.4515 (15) Å
$c$ = 12.438 (2) Å
$\beta$ = 110.678 (2)°
$V$ = 1253.2 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 3.27 mm$^{-1}$
$T$ = 100 K
0.21 × 0.16 × 0.08 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.547, $T_{max}$ = 0.780
23669 measured reflections
2942 independent reflections
2595 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.054
$S$ = 1.03
2942 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2*NA*···N1$^{i}$ | 0.88 | 2.10 | 2.975 (2) | 178 |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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: RZ2555).

## References

Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
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# supporting information

*Acta Cryst.* (2011). E**67**, o650 [doi:10.1107/S1600536811005332]

## 5-Bromo-3-(indan-1-yloxy)pyridin-2-amine

**Sujin Cho-Schultz, John C. Kath, Curtis Moore, Arnold L. Rheingold and Alex Yanovsky**

### S1. Comment

The present study confirmed the expected structure of the title compound, the product of reaction between indan-1-yl methanesulfonate and 2-amino-5-bromopyridin-3-ol in the presence of caesium carbonate (Fig. 1).

All C atoms of the indane fragment with the exception of C7 are coplanar within 0.03 Å; the latter atom is displaced by 0.206 (2) Å from the mean plane based on all the remaining atoms of the bicyclic system. The O1 atom deviates from this plane by 1.008 (2) Å in the same direction as the C7 atom. The central C2—O1—C6 bridge is in fact coplanar with the pyridine ring so that the N1/C1/C2/C3/C3/C4/C5/O1/C6 fragment is planar within 0.01 Å and its plane forms the dihedral angle of 57.6 (3)° with the above mentioned indane plane. It is noteworthy, that general conformations of a few other structurally studied molecules featuring indane group linked to pyridine derivatives through the C—O—C bridge (Dinçer *et al.*, 2004; Lifshits *et al.*, 2008) bear close resemblance to that of the molecule of the title compound.

The N2—H2NA···N1$^{i}$ bonds [symmetry code (i): 2 - *x*, 1 - *y*, 2 - *z*] (Table 1) link molecules in the crystal of the title compounds into centrosymmetric dimers (Fig. 2). One more intermolecular contact N2—H2NB···Br1$^{ii}$ [symmetry code (ii): *x* - 1/2, 1.5 - *y*, *z* - 1/2] may also play certain role in the stability of the packing, although corresponding interaction seems to be too weak to be qualified as one more independent H-bond.

### S2. Experimental

To a solution of 2-amino-5-bromopyridin-3-ol (1.640 g, 8.48 mmol) in 42 ml of DMF was added 2,3-dihydro-1*H*-inden-1-yl methanesulfonate (0.9 g, 4.24 mmol) and caesium carbonate (1.380 g, 4.24 mmol) and heated to 60°C overnight. The reaction mixture was quenched with water and the aqueous layer was extracted with EtOAc (3 *x* 20 ml). The organic layers were combined, dried over $MgSO_4$, filtered and concentrated. The product was purified by flash chromatography (silica gel, 10–50% EtOAc/heptane) to give 525 mg (46%) 5-bromo-3-(2,3-dihydro-1*H*-inden-1-yl-oxy)pyridin-2-amine as a white solid.

The colorless crystals were grown by slow cooling of the solution of the title compound in boiling dichloroetane.

### S3. Refinement

All H atoms were placed in geometrically calculated positions (N—H 0.88 Å, C—H 0.95 Å, 0.99 Å and 1.00 Å for aromatic, methylene and methine groups respectively) and included in the refinement in the riding motion approximation. The $U_{iso}$(H) were set to 1.2$U_{eq}$ of the carrying atom.

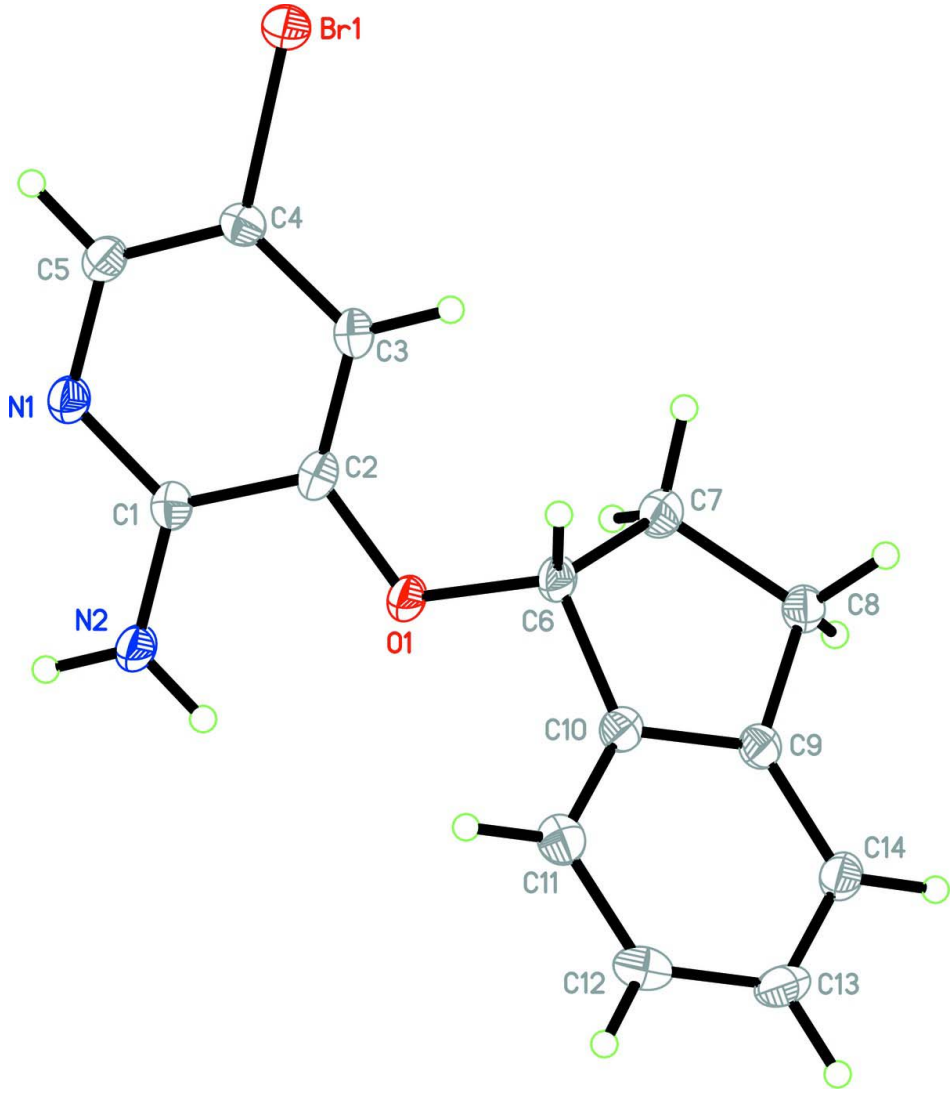


**Figure 1**

Molecular structure of the title compound, showing 50% probability displacement ellipsoids. H atoms are drawn as circles of arbitrary small radius.

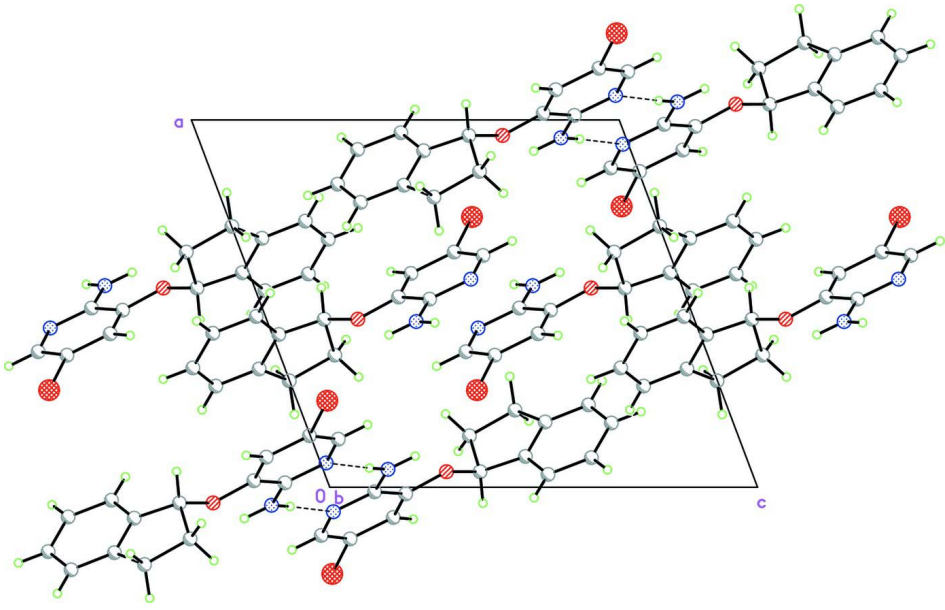


**Figure 2**

Packing diagram of the title compound viewed down the *b* axis. H-bonds are shown as dashed lines.

### 5-Bromo-3-(indan-1-yloxy)pyridin-2-amine

*Crystal data*

$C_{14}H_{13}BrN_2O$
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Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.3944\ (18)$ Å
$b = 9.4515\ (15)$ Å
$c = 12.438\ (2)$ Å
$\beta = 110.678\ (2)°$
$V = 1253.2\ (3)$ Å$^3$
$Z = 4$
$F(000) = 616$
$D_x = 1.617$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9797 reflections
$\theta = 2.8–27.7°$
$\mu = 3.27$ mm$^{-1}$
$T = 100$ K
Block, colorless
0.21 × 0.16 × 0.08 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.547$, $T_{max} = 0.780$
23669 measured reflections
2942 independent reflections
2595 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.1°$
$h = -15\rightarrow14$
$k = -12\rightarrow12$
$l = -16\rightarrow15$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.054$
$S = 1.03$
2942 reflections
163 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
$w = 1/[\sigma^2(F_o^2) + (0.0191P)^2 + 0.7815P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.38$ e Å$^{-3}$
$\Delta\rho_{min} = -0.30$ e Å$^{-3}$

*Special details*

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 | 1.235622 (16) | 1.080477 (17) | 1.070416 (15) | 0.02063 (6) |
| O1 | 0.95752 (11) | 0.77404 (12) | 0.71352 (10) | 0.0192 (3) |
| N1 | 1.06647 (13) | 0.68706 (14) | 1.01301 (12) | 0.0179 (3) |
| N2 | 0.95112 (15) | 0.56292 (15) | 0.84811 (13) | 0.0227 (3) |
| H2NA | 0.9477 | 0.4894 | 0.8903 | 0.027* |
| H2NB | 0.9150 | 0.5591 | 0.7729 | 0.027* |
| C1 | 1.01219 (16) | 0.68247 (17) | 0.89913 (14) | 0.0163 (3) |
| C2 | 1.01793 (15) | 0.79884 (17) | 0.82822 (14) | 0.0165 (3) |
| C3 | 1.08206 (16) | 0.91835 (17) | 0.87805 (15) | 0.0171 (3) |
| H3 | 1.0868 | 0.9979 | 0.8331 | 0.020* |
| C4 | 1.14048 (16) | 0.91900 (16) | 0.99797 (15) | 0.0161 (3) |
| C5 | 1.13089 (16) | 0.80499 (17) | 1.06228 (15) | 0.0180 (3) |
| H5 | 1.1704 | 0.8086 | 1.1435 | 0.022* |
| C6 | 0.95743 (16) | 0.88611 (17) | 0.63483 (14) | 0.0168 (3) |
| H6 | 1.0431 | 0.9283 | 0.6559 | 0.020* |
| C7 | 0.85977 (17) | 1.00246 (18) | 0.62819 (15) | 0.0209 (4) |
| H7A | 0.8003 | 0.9696 | 0.6647 | 0.025* |
| H7B | 0.9019 | 1.0892 | 0.6681 | 0.025* |
| C8 | 0.78992 (16) | 1.03247 (17) | 0.49914 (15) | 0.0184 (3) |
| H8A | 0.6983 | 1.0385 | 0.4815 | 0.022* |
| H8B | 0.8193 | 1.1220 | 0.4759 | 0.022* |
| C9 | 0.82218 (16) | 0.90756 (16) | 0.43914 (15) | 0.0164 (3) |
| C10 | 0.91601 (15) | 0.82524 (17) | 0.51553 (14) | 0.0163 (3) |
| C11 | 0.96492 (16) | 0.70772 (17) | 0.47841 (15) | 0.0200 (4) |
| H11 | 1.0284 | 0.6517 | 0.5316 | 0.024* |
| C12 | 0.91930 (17) | 0.67388 (18) | 0.36230 (16) | 0.0225 (4) |
| H12 | 0.9523 | 0.5947 | 0.3353 | 0.027* |
| C13 | 0.82497 (17) | 0.75614 (19) | 0.28514 (15) | 0.0224 (4) |
| H13 | 0.7942 | 0.7322 | 0.2059 | 0.027* |
| C14 | 0.77560 (17) | 0.87233 (18) | 0.32274 (15) | 0.0204 (4) |
| H14 | 0.7109 | 0.9272 | 0.2699 | 0.025* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.02147 (10) | 0.01833 (9) | 0.01970 (10) | −0.00397 (7) | 0.00432 (7) | −0.00082 (6) |
| O1 | 0.0247 (7) | 0.0182 (6) | 0.0125 (6) | −0.0039 (5) | 0.0038 (5) | 0.0033 (4) |
| N1 | 0.0195 (7) | 0.0179 (7) | 0.0156 (7) | −0.0011 (6) | 0.0055 (6) | 0.0024 (5) |
| N2 | 0.0298 (9) | 0.0198 (7) | 0.0140 (7) | −0.0074 (6) | 0.0021 (6) | 0.0035 (6) |
| C1 | 0.0150 (8) | 0.0174 (7) | 0.0168 (8) | 0.0007 (6) | 0.0058 (7) | 0.0029 (6) |
| C2 | 0.0149 (8) | 0.0209 (8) | 0.0137 (8) | 0.0019 (6) | 0.0052 (6) | 0.0030 (6) |
| C3 | 0.0178 (8) | 0.0168 (8) | 0.0172 (9) | 0.0013 (6) | 0.0070 (7) | 0.0039 (6) |
| C4 | 0.0141 (8) | 0.0155 (7) | 0.0184 (8) | −0.0002 (6) | 0.0053 (7) | −0.0013 (6) |
| C5 | 0.0187 (9) | 0.0201 (8) | 0.0145 (8) | 0.0009 (7) | 0.0049 (7) | 0.0003 (6) |
| C6 | 0.0195 (9) | 0.0169 (7) | 0.0140 (8) | −0.0022 (6) | 0.0057 (7) | 0.0033 (6) |
| C7 | 0.0257 (10) | 0.0202 (8) | 0.0161 (9) | 0.0025 (7) | 0.0067 (7) | 0.0009 (7) |
| C8 | 0.0184 (9) | 0.0171 (8) | 0.0180 (9) | 0.0002 (6) | 0.0045 (7) | 0.0020 (6) |
| C9 | 0.0170 (8) | 0.0158 (7) | 0.0166 (8) | −0.0037 (6) | 0.0062 (7) | 0.0018 (6) |
| C10 | 0.0159 (8) | 0.0177 (8) | 0.0158 (8) | −0.0032 (6) | 0.0061 (7) | 0.0012 (6) |
| C11 | 0.0179 (9) | 0.0193 (8) | 0.0225 (9) | −0.0002 (7) | 0.0067 (7) | 0.0013 (7) |
| C12 | 0.0233 (9) | 0.0200 (8) | 0.0262 (10) | −0.0048 (7) | 0.0113 (8) | −0.0068 (7) |
| C13 | 0.0256 (10) | 0.0258 (9) | 0.0154 (9) | −0.0086 (7) | 0.0066 (7) | −0.0050 (7) |
| C14 | 0.0213 (9) | 0.0199 (8) | 0.0169 (9) | −0.0040 (7) | 0.0028 (7) | 0.0029 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C4 | 1.9044 (16) | C7—C8 | 1.545 (2) |
| O1—C2 | 1.368 (2) | C7—H7A | 0.9900 |
| O1—C6 | 1.4419 (19) | C7—H7B | 0.9900 |
| N1—C1 | 1.331 (2) | C8—C9 | 1.510 (2) |
| N1—C5 | 1.356 (2) | C8—H8A | 0.9900 |
| N2—C1 | 1.361 (2) | C8—H8B | 0.9900 |
| N2—H2NA | 0.8800 | C9—C10 | 1.391 (2) |
| N2—H2NB | 0.8800 | C9—C14 | 1.395 (2) |
| C1—C2 | 1.426 (2) | C10—C11 | 1.393 (2) |
| C2—C3 | 1.369 (2) | C11—C12 | 1.388 (3) |
| C3—C4 | 1.402 (2) | C11—H11 | 0.9500 |
| C3—H3 | 0.9500 | C12—C13 | 1.397 (3) |
| C4—C5 | 1.369 (2) | C12—H12 | 0.9500 |
| C5—H5 | 0.9500 | C13—C14 | 1.388 (3) |
| C6—C10 | 1.504 (2) | C13—H13 | 0.9500 |
| C6—C7 | 1.546 (2) | C14—H14 | 0.9500 |
| C6—H6 | 1.0000 | | |
| | | | |
| C2—O1—C6 | 117.49 (13) | C6—C7—H7A | 110.4 |
| C1—N1—C5 | 118.79 (14) | C8—C7—H7B | 110.4 |
| C1—N2—H2NA | 120.0 | C6—C7—H7B | 110.4 |
| C1—N2—H2NB | 120.0 | H7A—C7—H7B | 108.6 |
| H2NA—N2—H2NB | 120.0 | C9—C8—C7 | 104.11 (13) |
| N1—C1—N2 | 119.49 (15) | C9—C8—H8A | 110.9 |

| | | | |
|---|---|---|---|
| N1—C1—C2 | 121.84 (15) | C7—C8—H8A | 110.9 |
| N2—C1—C2 | 118.66 (15) | C9—C8—H8B | 110.9 |
| O1—C2—C3 | 127.25 (15) | C7—C8—H8B | 110.9 |
| O1—C2—C1 | 113.39 (14) | H8A—C8—H8B | 109.0 |
| C3—C2—C1 | 119.35 (15) | C10—C9—C14 | 119.63 (16) |
| C2—C3—C4 | 117.53 (15) | C10—C9—C8 | 111.25 (15) |
| C2—C3—H3 | 121.2 | C14—C9—C8 | 129.04 (16) |
| C4—C3—H3 | 121.2 | C9—C10—C11 | 121.40 (16) |
| C5—C4—C3 | 120.80 (15) | C9—C10—C6 | 110.97 (14) |
| C5—C4—Br1 | 120.25 (13) | C11—C10—C6 | 127.55 (15) |
| C3—C4—Br1 | 118.94 (12) | C12—C11—C10 | 118.81 (16) |
| N1—C5—C4 | 121.67 (16) | C12—C11—H11 | 120.6 |
| N1—C5—H5 | 119.2 | C10—C11—H11 | 120.6 |
| C4—C5—H5 | 119.2 | C11—C12—C13 | 120.06 (16) |
| O1—C6—C10 | 108.27 (13) | C11—C12—H12 | 120.0 |
| O1—C6—C7 | 112.76 (14) | C13—C12—H12 | 120.0 |
| C10—C6—C7 | 104.49 (14) | C14—C13—C12 | 120.92 (16) |
| O1—C6—H6 | 110.4 | C14—C13—H13 | 119.5 |
| C10—C6—H6 | 110.4 | C12—C13—H13 | 119.5 |
| C7—C6—H6 | 110.4 | C13—C14—C9 | 119.18 (16) |
| C8—C7—C6 | 106.45 (14) | C13—C14—H14 | 120.4 |
| C8—C7—H7A | 110.4 | C9—C14—H14 | 120.4 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*NA*···N1$^{i}$ | 0.88 | 2.10 | 2.975 (2) | 178 |

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