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# 2-Chloro-*N*′-(2-hydroxy-3,5-diiodobenzylidene)benzohydrazide

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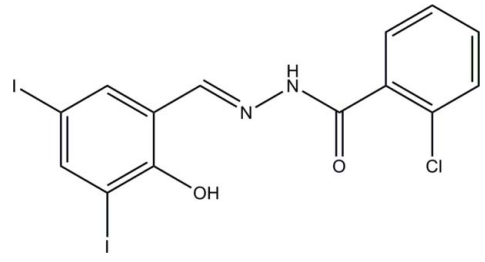
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.013 Å; $R$ factor = 0.055; $wR$ factor = 0.112; data-to-parameter ratio = 17.4.

In the title compound, $C_{14}H_9ClI_2N_2O_2$, the dihedral angle between the benzene rings is 65.9 (2)° and an intramolecular O—H···N hydrogen bond generates an $S$(6) ring. The molecule has an $E$ conformation about the C=N bond. In the crystal, molecules are linked into $C$(4) chains propagating in [001] by N—H···O hydrogen bonds.

## Related literature

For background to hydrazone compounds and their biological properties, see: Kucukguzel *et al.* (2006); Khattab (2005); Karthikeyan *et al.* (2006); Okabe *et al.* (1993). For reference bond-length values, see: Allen *et al.* (1987). For related structures, see: Shan *et al.* (2008); Fun *et al.* (2008); Yang (2008); Ma *et al.* (2008); Diao *et al.* (2008*a*,*b*); Ejsmont *et al.* (2008).




## Experimental

### Crystal data

$C_{14}H_9ClI_2N_2O_2$
$M_r$ = 526.48
Monoclinic, $P2_1/c$
$a$ = 14.311 (3) Å
$b$ = 11.469 (2) Å
$c$ = 9.736 (2) Å
$\beta$ = 90.032 (2)°
$V$ = 1598.0 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 4.11 mm$^{-1}$
$T$ = 298 K
0.18 × 0.17 × 0.17 mm

### Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.525, $T_{max}$ = 0.542
7381 measured reflections
3383 independent reflections
1747 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.069

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.112
$S$ = 0.95
3383 reflections
194 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.93 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.80 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···N1 | 0.82 | 1.83 | 2.556 (8) | 146 |
| N2—H2···O2$^{i}$ | 0.91 (4) | 1.88 (2) | 2.768 (8) | 168 (8) |

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5809).

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Diao, Y.-P., Huang, S.-S., Zhang, J.-K. & Kang, T.-G. (2008*a*). *Acta Cryst.* E**64**, o470.
Diao, Y.-P., Zhen, Y.-H., Han, X. & Deng, S. (2008*b*). *Acta Cryst.* E**64**, o101.
Ejsmont, K., Zareef, M., Arfan, M., Bashir, S. A. & Zaleski, J. (2008). *Acta Cryst.* E**64**, o1128.
Fun, H.-K., Sujith, K. V., Patil, P. S., Kalluraya, B. & Chantrapromma, S. (2008). *Acta Cryst.* E**64**, o1961–o1962.
Karthikeyan, M. S., Prasad, D. J., Poojary, B., Bhat, K. S., Holla, B. S. & Kumari, N. S. (2006). *Bioorg. Med. Chem.* **14**, 7482–7489.
Khattab, S. N. (2005). *Molecules* **10**, 1218–1228.
Kucukguzel, G., Kocatepe, A., De Clercq, E., Sahi, F. & Gulluce, M. (2006). *Eur. J. Med. Chem.* **41**, 353–359.
Ma, H.-B., Huang, S.-S. & Diao, Y.-P. (2008). *Acta Cryst.* E**64**, o210.
Okabe, N., Nakamura, T. & Fukuda, H. (1993). *Acta Cryst.* C**49**, 1678–1680.
Shan, S., Tian, Y.-L., Wang, S.-H., Wang, W.-L. & Xu, Y.-L. (2008). *Acta Cryst.* E**64**, o1363.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yang, D.-S. (2008). *Acta Cryst.* E**64**, o1759.

# supporting information

*Acta Cryst.* (2011). E**67**, o810 [doi:10.1107/S1600536811007653]

## 2-Chloro-*N*′-(2-hydroxy-3,5-diiodobenzylidene)benzohydrazide

**Fei Wang, Da-Yong Liu, Hai-Bo Wang, Xian-Sheng Meng and Ting-Guo Kang**

### S1. Comment

Hydrazones have been attracted much attention for their excellent biological properties, especially for their potential pharmacological and antitumor properties (Kucukguzel *et al.*, 2006; Khattab *et al.*, 2005; Karthikeyan *et al.*, 2006; Okabe *et al.*, 1993). Recently, a large number of hydrazone derivatives have been prepared and structurally characterized (Shan *et al.*, 2008; Fun *et al.*, 2008; Yang, 2008; Ma *et al.*, 2008; Diao *et al.*, 2008*a*,*b*; Ejsmont *et al.*, 2008). In this paper, the title new hydrazone compound is reported.

The molecular structure of the title compound is shown in Fig. 1. The bond distances and angles are normal (Allen *et al.*, 1987). The dihedral angle between the two benzene rings is 65.9 (2)°. The molecule of the compound displays an *E* geometry about the C═N bond. The molecules are linked into chains along the *c* axis by intermolecular N—H···O hydrogen bonds (Fig. 2 and Table 1).

### S2. Experimental

2-Hydroxy-3,5-diiodobenzaldehyde (1.0 mmol, 373.9 mg) was dissolved in methanol (50 ml), then 2-chloro-benzohydrazide (1.0 mmol, 170.6 mg) was added slowly into the solution, and the mixture was kept at reflux with continuous stirring for 2 h. After the solution had cooled to room temperature colourless powder crystals appeared. The powder crystals were filtered and washed with methanol for three times. Recrystallization from absolute methanol yielded colourless block-shaped single crystals of the title compound.

### S3. Refinement

H2 was located in a difference Fourier map and refined isotropically, with N—H distance restrained to 0.90 (1) Å. Other H atoms were placed in calculated positions with O—H = 0.82 Å, C—H = 0.93 Å, and refined in riding mode with $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(O)$.

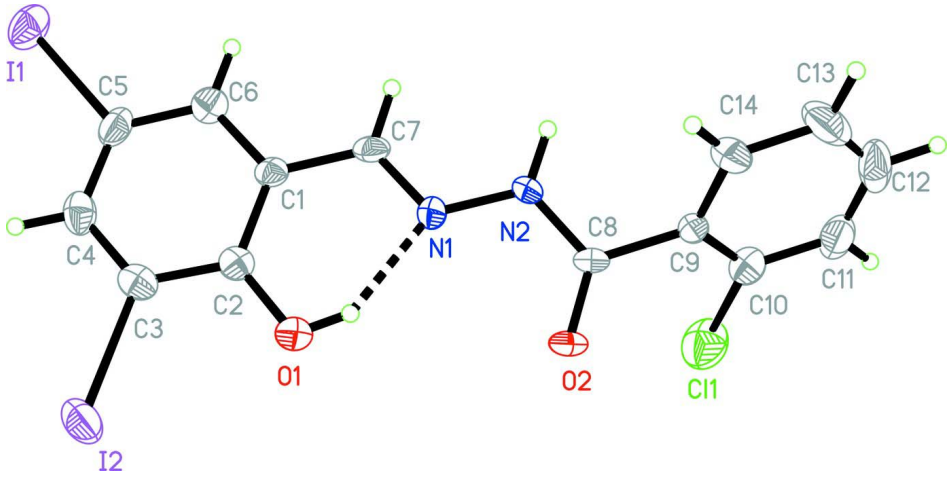


**Figure 1**

The molecular structure of the title compound with 30% probability displacement ellipsoids for non-H atoms. Intramolecular O—H···N hydrogen bond is drawn as a dashed line.

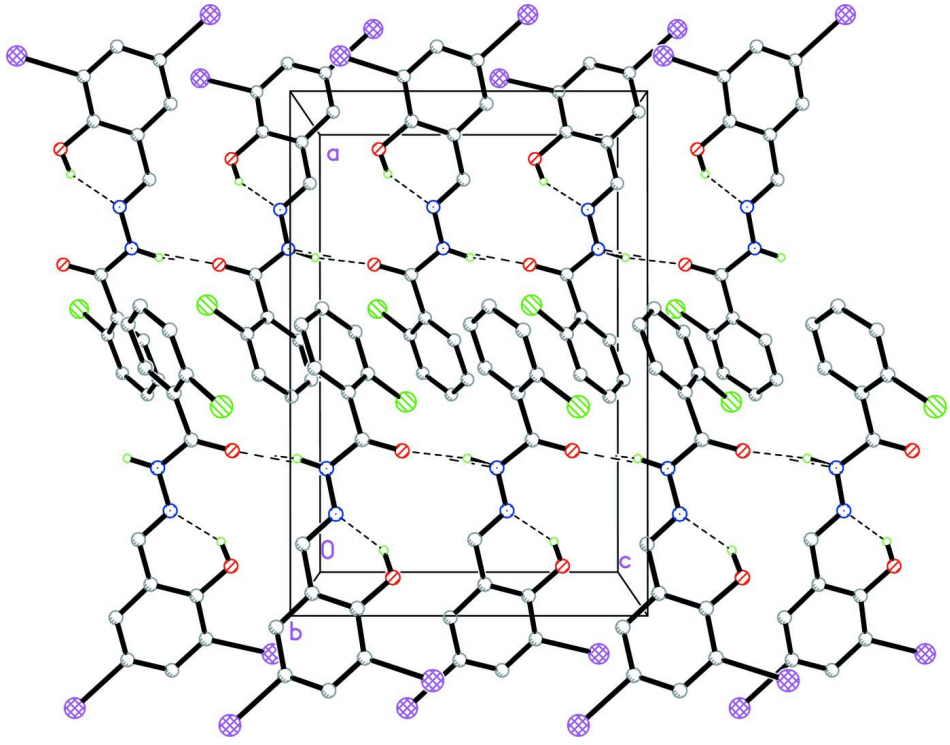


**Figure 2**

Molecular packing as viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

### 2-Chloro-*N*′-(2-hydroxy-3,5-diiodobenzylidene)benzohydrazide

*Crystal data*

$C_{14}H_9ClI_2N_2O_2$
$M_r$ = 526.48
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

$a$ = 14.311 (3) Å
$b$ = 11.469 (2) Å
$c$ = 9.736 (2) Å
$\beta$ = 90.032 (2)°

$V$ = 1598.0 (5) Å$^3$
$Z$ = 4
$F$(000) = 984
$D_x$ = 2.188 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 989 reflections

$\theta$ = 2.5–24.5°
$\mu$ = 4.11 mm$^{-1}$
$T$ = 298 K
Block, colourless
0.18 × 0.17 × 0.17 mm

*Data collection*

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.525, $T_{max}$ = 0.542

7381 measured reflections
3383 independent reflections
1747 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.069
$\theta_{max}$ = 27.0°, $\theta_{min}$ = 2.7°
$h$ = −18→13
$k$ = −14→9
$l$ = −12→12

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.112
$S$ = 0.95
3383 reflections
194 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.93 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.80 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| I1 | −0.22657 (4) | 0.87447 (6) | −0.18612 (7) | 0.0664 (2) |
| I2 | −0.12679 (4) | 0.97420 (6) | 0.39763 (7) | 0.0689 (2) |
| Cl1 | 0.39839 (19) | 0.5079 (3) | 0.3068 (3) | 0.0908 (9) |
| N1 | 0.1820 (4) | 0.8044 (6) | 0.1129 (6) | 0.0385 (16) |
| N2 | 0.2702 (4) | 0.7650 (6) | 0.0825 (6) | 0.0402 (17) |
| O1 | 0.0634 (3) | 0.8886 (5) | 0.2821 (5) | 0.0482 (14) |
| H1 | 0.1148 | 0.8671 | 0.2551 | 0.072* |
| O2 | 0.3053 (4) | 0.7548 (6) | 0.3039 (6) | 0.077 (2) |
| C1 | 0.0291 (5) | 0.8487 (6) | 0.0472 (8) | 0.0343 (19) |
| C2 | 0.0025 (5) | 0.8833 (6) | 0.1793 (8) | 0.0367 (19) |

| | | | | |
|---|---|---|---|---|
| C3 | −0.0897 (5) | 0.9172 (7) | 0.2027 (8) | 0.043 (2) |
| C4 | −0.1538 (5) | 0.9133 (7) | 0.0998 (10) | 0.050 (2) |
| H4 | −0.2153 | 0.9350 | 0.1168 | 0.060* |
| C5 | −0.1283 (5) | 0.8778 (8) | −0.0279 (9) | 0.051 (2) |
| C6 | −0.0383 (5) | 0.8446 (6) | −0.0562 (8) | 0.044 (2) |
| H6 | −0.0222 | 0.8196 | −0.1439 | 0.053* |
| C7 | 0.1229 (5) | 0.8119 (7) | 0.0171 (8) | 0.0378 (19) |
| H7 | 0.1400 | 0.7937 | −0.0725 | 0.045* |
| C8 | 0.3274 (5) | 0.7392 (8) | 0.1856 (8) | 0.046 (2) |
| C9 | 0.4215 (5) | 0.6948 (8) | 0.1424 (8) | 0.044 (2) |
| C10 | 0.4584 (6) | 0.5931 (8) | 0.1939 (9) | 0.055 (2) |
| C11 | 0.5447 (7) | 0.5530 (10) | 0.1535 (11) | 0.074 (3) |
| H11 | 0.5699 | 0.4843 | 0.1882 | 0.088* |
| C12 | 0.5921 (7) | 0.6211 (15) | 0.0577 (14) | 0.102 (6) |
| H12 | 0.6510 | 0.5968 | 0.0293 | 0.122* |
| C13 | 0.5576 (8) | 0.7187 (12) | 0.0048 (12) | 0.090 (4) |
| H13 | 0.5915 | 0.7607 | −0.0599 | 0.108* |
| C14 | 0.4710 (6) | 0.7572 (9) | 0.0471 (9) | 0.068 (3) |
| H14 | 0.4462 | 0.8255 | 0.0109 | 0.082* |
| H2 | 0.290 (5) | 0.755 (8) | −0.005 (3) | 0.080* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I1 | 0.0465 (4) | 0.0791 (5) | 0.0737 (5) | −0.0086 (3) | −0.0190 (3) | 0.0115 (4) |
| I2 | 0.0617 (4) | 0.0786 (5) | 0.0663 (5) | 0.0136 (3) | 0.0127 (3) | −0.0186 (4) |
| Cl1 | 0.0839 (19) | 0.089 (2) | 0.099 (2) | −0.0011 (15) | −0.0081 (16) | 0.0317 (18) |
| N1 | 0.028 (4) | 0.052 (5) | 0.036 (4) | 0.004 (3) | −0.002 (3) | −0.002 (3) |
| N2 | 0.034 (4) | 0.063 (5) | 0.024 (4) | 0.008 (3) | 0.004 (3) | −0.005 (4) |
| O1 | 0.051 (3) | 0.058 (4) | 0.036 (3) | 0.008 (3) | 0.000 (3) | −0.002 (3) |
| O2 | 0.068 (4) | 0.143 (7) | 0.020 (3) | 0.042 (4) | 0.003 (3) | 0.003 (4) |
| C1 | 0.041 (5) | 0.031 (5) | 0.031 (5) | −0.001 (3) | 0.002 (3) | 0.008 (4) |
| C2 | 0.035 (4) | 0.035 (5) | 0.040 (5) | −0.011 (4) | 0.004 (3) | 0.008 (4) |
| C3 | 0.051 (5) | 0.034 (5) | 0.044 (6) | −0.007 (4) | 0.010 (4) | 0.002 (4) |
| C4 | 0.034 (5) | 0.048 (6) | 0.067 (7) | 0.000 (4) | 0.004 (4) | 0.006 (5) |
| C5 | 0.037 (5) | 0.061 (6) | 0.055 (6) | 0.000 (4) | −0.011 (4) | 0.014 (5) |
| C6 | 0.049 (5) | 0.038 (5) | 0.045 (5) | −0.003 (4) | −0.006 (4) | −0.008 (4) |
| C7 | 0.051 (5) | 0.038 (5) | 0.025 (5) | 0.002 (4) | 0.003 (4) | 0.009 (4) |
| C8 | 0.047 (5) | 0.070 (7) | 0.020 (5) | 0.007 (4) | 0.002 (4) | 0.000 (4) |
| C9 | 0.030 (4) | 0.072 (7) | 0.029 (5) | 0.000 (4) | −0.002 (3) | −0.008 (5) |
| C10 | 0.048 (6) | 0.066 (7) | 0.052 (6) | −0.001 (5) | −0.007 (4) | −0.003 (5) |
| C11 | 0.054 (7) | 0.100 (10) | 0.067 (8) | 0.026 (6) | −0.018 (5) | −0.025 (7) |
| C12 | 0.039 (6) | 0.182 (16) | 0.083 (10) | 0.015 (8) | −0.013 (6) | −0.079 (11) |
| C13 | 0.066 (8) | 0.141 (13) | 0.063 (8) | −0.034 (8) | 0.029 (6) | −0.016 (8) |
| C14 | 0.050 (6) | 0.112 (9) | 0.043 (6) | −0.006 (6) | 0.010 (4) | 0.007 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| I1—C5 | 2.086 (7) | C4—H4 | 0.9300 |
| I2—C3 | 2.076 (8) | C5—C6 | 1.372 (10) |
| Cl1—C10 | 1.703 (9) | C6—H6 | 0.9300 |
| N1—C7 | 1.261 (8) | C7—H7 | 0.9300 |
| N1—N2 | 1.373 (7) | C8—C9 | 1.501 (10) |
| N2—C8 | 1.328 (9) | C9—C14 | 1.369 (11) |
| N2—H2 | 0.91 (4) | C9—C10 | 1.374 (12) |
| O1—C2 | 1.328 (8) | C10—C11 | 1.376 (11) |
| O1—H1 | 0.8200 | C11—C12 | 1.393 (16) |
| O2—C8 | 1.208 (9) | C11—H11 | 0.9300 |
| C1—C6 | 1.395 (10) | C12—C13 | 1.328 (16) |
| C1—C2 | 1.398 (10) | C12—H12 | 0.9300 |
| C1—C7 | 1.438 (9) | C13—C14 | 1.379 (13) |
| C2—C3 | 1.396 (10) | C13—H13 | 0.9300 |
| C3—C4 | 1.358 (10) | C14—H14 | 0.9300 |
| C4—C5 | 1.358 (11) | | |
| | | | |
| C7—N1—N2 | 118.6 (6) | N1—C7—H7 | 120.2 |
| C8—N2—N1 | 118.5 (6) | C1—C7—H7 | 120.2 |
| C8—N2—H2 | 119 (5) | O2—C8—N2 | 121.8 (7) |
| N1—N2—H2 | 122 (5) | O2—C8—C9 | 123.5 (7) |
| C2—O1—H1 | 109.5 | N2—C8—C9 | 114.6 (7) |
| C6—C1—C2 | 119.0 (7) | C14—C9—C10 | 119.5 (8) |
| C6—C1—C7 | 119.2 (7) | C14—C9—C8 | 118.5 (8) |
| C2—C1—C7 | 121.7 (6) | C10—C9—C8 | 122.0 (8) |
| O1—C2—C3 | 118.9 (7) | C9—C10—C11 | 121.6 (9) |
| O1—C2—C1 | 121.8 (7) | C9—C10—Cl1 | 121.9 (7) |
| C3—C2—C1 | 119.2 (7) | C11—C10—Cl1 | 116.5 (8) |
| C4—C3—C2 | 120.5 (8) | C10—C11—C12 | 116.2 (10) |
| C4—C3—I2 | 120.8 (6) | C10—C11—H11 | 121.9 |
| C2—C3—I2 | 118.6 (6) | C12—C11—H11 | 121.9 |
| C5—C4—C3 | 120.3 (8) | C13—C12—C11 | 123.5 (12) |
| C5—C4—H4 | 119.9 | C13—C12—H12 | 118.3 |
| C3—C4—H4 | 119.9 | C11—C12—H12 | 118.3 |
| C4—C5—C6 | 121.3 (7) | C12—C13—C14 | 119.2 (12) |
| C4—C5—I1 | 120.0 (6) | C12—C13—H13 | 120.4 |
| C6—C5—I1 | 118.7 (7) | C14—C13—H13 | 120.4 |
| C5—C6—C1 | 119.7 (8) | C9—C14—C13 | 120.0 (10) |
| C5—C6—H6 | 120.2 | C9—C14—H14 | 120.0 |
| C1—C6—H6 | 120.2 | C13—C14—H14 | 120.0 |
| N1—C7—C1 | 119.7 (7) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N1 | 0.82 | 1.83 | 2.556 (8) | 146 |

| | | | | |
|---|---|---|---|---|
| N2—H2···O2$^{i}$ | 0.91 (4) | 1.88 (2) | 2.768 (8) | 168 (8) |

Symmetry code: (i) $x$, $-y+3/2$, $z-1/2$.