

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(E)-N'-(2,4-Dichlorobenzylidene)-3-nitrobenzohydrazide

Fu-Lin Mao* and Chun-Hua Dai

Jiangsu Provincial Key Laboratory of Coastal Wetland Bioresources and Environmental Protection, Department of Chemistry, Yancheng Teachers University, Yancheng 224002, People's Republic of China

Correspondence e-mail: xpzhougroup@163.com

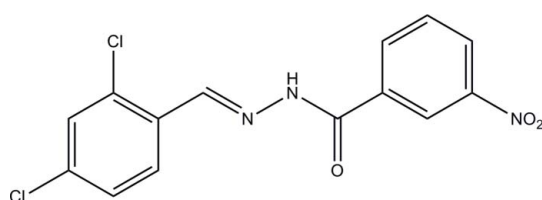
Received 25 November 2010; accepted 26 November 2010

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.043; wR factor = 0.107; data-to-parameter ratio = 15.7.

The title compound, $\text{C}_{14}\text{H}_9\text{Cl}_2\text{N}_3\text{O}_3$, was prepared by the reaction of 3-nitrobenzohydrazide with 2,4-dichlorobenzaldehyde. The molecule adopts an *E* configuration about the $\text{C}=\text{N}$ bond. The dihedral angle between the two benzene rings is $4.6(2)^\circ$. In the crystal, the hydrazone molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming chains along the *c* axis.

Related literature

For medical applications of hydrazones, see: Ajani *et al.* (2010); Zhang *et al.* (2010); Angelusiu *et al.* (2010). For related structures, see: Huang & Wu (2010); Khaledi *et al.* (2010); Zhou & Yang (2010); Ji & Lu (2010); Singh & Singh (2010); Ahmad *et al.* (2010). For similar compounds that we have reported recently, see: Dai & Mao (2010*a,b*).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_9\text{Cl}_2\text{N}_3\text{O}_3$ $M_r = 338.14$ Monoclinic, $P2_1/c$ $a = 12.004(3)$ Å $b = 14.384(3)$ Å $c = 8.465(2)$ Å $\beta = 96.302(2)^\circ$ $V = 1452.8(6)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.46$ mm⁻¹ $T = 298$ K $0.20 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

 $T_{\min} = 0.913$, $T_{\max} = 0.921$

11555 measured reflections

3163 independent reflections

2128 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.038$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.107$ $S = 1.05$

3163 reflections

202 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N2}-\text{H2}\cdots\text{O1}^i$ | 0.89 (1) | 2.04 (2) | 2.859 (2) | 152 (2) |

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

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.

We acknowledge the Jiangsu Provincial Key Laboratory of Coastal Wetland Bioresources and Environmental Protection for financial support (project No. JLCBE07026).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5069).

References

- Ahmad, T., Zia-ur-Rehman, M., Siddiqui, H. L., Mahmud, S. & Parvez, M. (2010). *Acta Cryst.* **E66**, o1022.
- Ajani, O. O., Obafemi, C. A., Nwinyi, O. C. & Akinpelu, D. A. (2010). *Bioorg. Med. Chem.* **18**, 214–221.
- Angelusiu, M. V., Barbuceanu, S. F., Draghici, C. & Almajian, G. L. (2010). *Eur. J. Med. Chem.* **45**, 2055–2062.
- Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2007). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dai, C.-H. & Mao, F.-L. (2010*a*). *Acta Cryst.* **E66**, o2942.
- Dai, C.-H. & Mao, F.-L. (2010*b*). *Acta Cryst.* **E66**, o3004–o3005.
- Huang, H.-T. & Wu, H.-Y. (2010). *Acta Cryst.* **E66**, o2729–o2730.
- Ji, X.-H. & Lu, J.-F. (2010). *Acta Cryst.* **E66**, o1514.
- Khaledi, H., Alhadi, A. A., Mohd Ali, H., Robinson, W. T. & Abdulla, M. A. (2010). *Acta Cryst.* **E66**, o105–o106.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Singh, V. P. & Singh, S. (2010). *Acta Cryst.* **E66**, o1172.
- Zhang, Y.-H., Zhang, L., Liu, L., Guo, J.-X., Wu, D.-L., Xu, G.-C., Wang, X.-H. & Jia, D.-Z. (2010). *Inorg. Chim. Acta*, **363**, 289–293.
- Zhou, C.-S. & Yang, T. (2010). *Acta Cryst.* **E66**, o290.

supporting information

Acta Cryst. (2010). E66, o3360 [https://doi.org/10.1107/S1600536810049470]

(*E*)-*N'*-(2,4-Dichlorobenzylidene)-3-nitrobenzohydrazide**Fu-Lin Mao and Chun-Hua Dai****S1. Comment**

In the last few years, medical applications of a number of hydrazone compounds have been received much attention (Ajani *et al.*, 2010; Zhang *et al.*, 2010; Angelusiu *et al.*, 2010). The structures of several hydrazone derivatives have also been determined (Huang & Wu, 2010; Khaledi *et al.*, 2010; Zhou & Yang, 2010; Ji & Lu, 2010; Singh & Singh, 2010; Ahmad *et al.*, 2010). As a continuation of our work on this area (Dai & Mao, 2010*a,b*), in this paper, we report the structure of the new derivative *EN'*-(2,4-dichlorobenzylidene)-3-nitrobenzohydrazide.

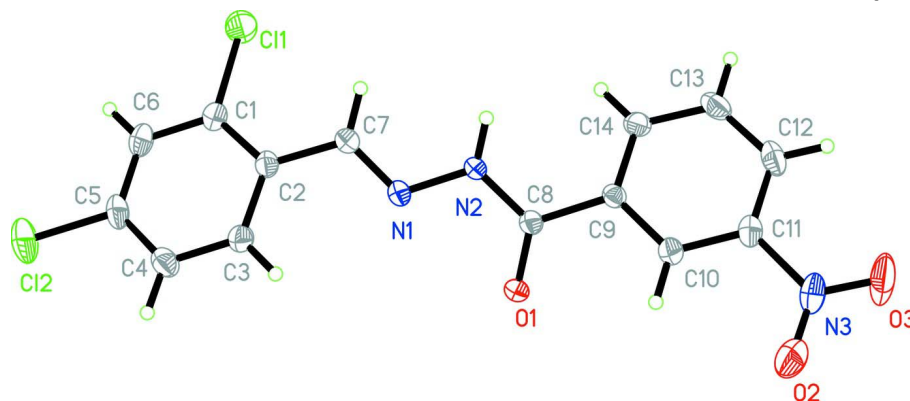
In the molecule of the title compound, the dihedral angle between the C1...C6 and C9...C14 benzene rings is 4.6 (2)°. The O2/N3/O3 plane forms a dihedral angle of 8.9 (2)° with the C9...C14 benzene ring. The bond lengths and angles are comparable to those found in the hydrazone compounds cited above. In the crystal structure, the hydrazone molecules are linked through intermolecular N—H...O hydrogen bonds (Table 1), to form one-dimensional chains along the *c* axis, as shown in Fig. 2.

S2. Experimental

The reaction of 3-nitrobenzohydrazide (0.181 g, 1 mmol) with 2,4-dichlorobenzaldehyde (0.174 g, 1 mmol) in 50 ml methanol at room temperature afforded the title compound. Yellow block-shaped single crystals were formed by slow evaporation of the clear solution in air.

S3. Refinement

The amino H atom was located in a difference Fourier map and refined with N—H = 0.90 (1) Å, and $U_{\text{iso}} = 0.08 \text{ \AA}^2$. Other H atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The molecular structure of the title compound showing 30% probability displacement ellipsoids and the atomic numbering.

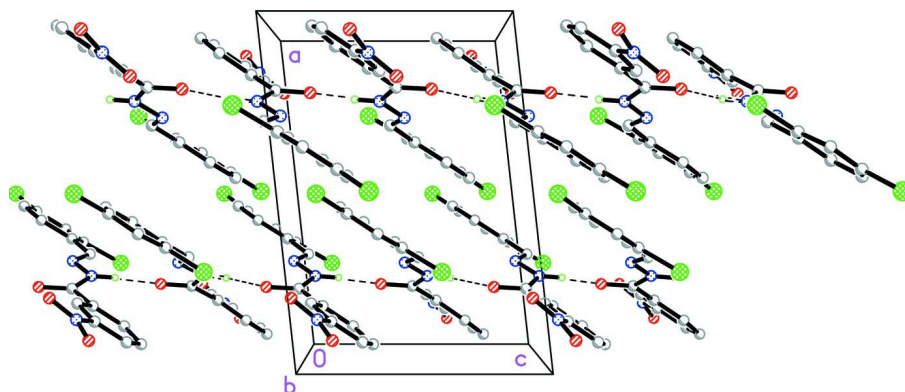


Figure 2

Crystal packing of the title compound, viewed down the *b* axis. Intermolecular interactions are drawn as dashed lines.

(E)-*N'*-(2,4-Dichlorobenzylidene)-3-nitrobenzohydrazide

Crystal data

$C_{14}H_9Cl_2N_3O_3$

$M_r = 338.14$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.004 (3) \text{ \AA}$

$b = 14.384 (3) \text{ \AA}$

$c = 8.465 (2) \text{ \AA}$

$\beta = 96.302 (2)^\circ$

$V = 1452.8 (6) \text{ \AA}^3$

$Z = 4$

$F(000) = 688$

$D_x = 1.546 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2237 reflections

$\theta = 2.3\text{--}24.5^\circ$

$\mu = 0.46 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, yellow

$0.20 \times 0.20 \times 0.18 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube
Graphite monochromator

ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2001)

$T_{\min} = 0.913$, $T_{\max} = 0.921$

11555 measured reflections

3163 independent reflections

2128 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -15 \rightarrow 13$

$k = -18 \rightarrow 18$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.107$

$S = 1.05$

3163 reflections

202 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

$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.1033P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.22 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|---------------|----------------------------------|
| C11 | 0.26406 (6) | -0.09815 (4) | 0.11977 (8) | 0.0651 (2) |
| C12 | 0.49631 (6) | -0.18532 (5) | -0.36654 (9) | 0.0760 (3) |
| N1 | 0.28128 (14) | 0.18486 (11) | -0.03316 (19) | 0.0344 (4) |
| N2 | 0.23633 (14) | 0.25093 (11) | 0.06051 (18) | 0.0342 (4) |
| N3 | 0.12644 (17) | 0.66091 (13) | 0.0801 (3) | 0.0542 (5) |
| O1 | 0.20635 (13) | 0.34676 (9) | -0.15308 (16) | 0.0454 (4) |
| O2 | 0.19086 (19) | 0.67781 (12) | -0.0175 (3) | 0.0808 (6) |
| O3 | 0.06980 (18) | 0.71959 (12) | 0.1368 (3) | 0.0922 (7) |
| C1 | 0.33504 (17) | -0.06183 (14) | -0.0373 (3) | 0.0385 (5) |
| C2 | 0.34878 (16) | 0.03282 (13) | -0.0659 (2) | 0.0330 (5) |
| C3 | 0.40982 (18) | 0.05744 (14) | -0.1909 (2) | 0.0394 (5) |
| H3 | 0.4201 | 0.1200 | -0.2128 | 0.047* |
| C4 | 0.45528 (18) | -0.00914 (16) | -0.2829 (2) | 0.0449 (5) |
| H4 | 0.4973 | 0.0084 | -0.3640 | 0.054* |
| C5 | 0.43747 (18) | -0.10158 (15) | -0.2525 (3) | 0.0446 (6) |
| C6 | 0.37757 (18) | -0.12984 (15) | -0.1318 (3) | 0.0451 (6) |
| H6 | 0.3657 | -0.1926 | -0.1135 | 0.054* |
| C7 | 0.30075 (17) | 0.10478 (13) | 0.0282 (2) | 0.0353 (5) |
| H7 | 0.2847 | 0.0923 | 0.1311 | 0.042* |
| C8 | 0.20044 (16) | 0.33100 (13) | -0.0112 (2) | 0.0320 (5) |
| C9 | 0.15054 (16) | 0.40225 (13) | 0.0894 (2) | 0.0296 (4) |
| C10 | 0.16380 (16) | 0.49489 (13) | 0.0476 (2) | 0.0340 (5) |
| H10 | 0.2056 | 0.5105 | -0.0347 | 0.041* |
| C11 | 0.11400 (18) | 0.56310 (14) | 0.1300 (2) | 0.0386 (5) |
| C12 | 0.05041 (19) | 0.54271 (16) | 0.2516 (3) | 0.0490 (6) |
| H12 | 0.0173 | 0.5898 | 0.3054 | 0.059* |
| C13 | 0.03713 (19) | 0.45074 (17) | 0.2916 (3) | 0.0477 (6) |
| H13 | -0.0056 | 0.4356 | 0.3732 | 0.057* |
| C14 | 0.08693 (16) | 0.38009 (15) | 0.2110 (2) | 0.0376 (5) |
| H14 | 0.0774 | 0.3183 | 0.2388 | 0.045* |
| H2 | 0.231 (2) | 0.2398 (18) | 0.1627 (14) | 0.080* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|-------------|------------|------------|
| C11 | 0.0776 (5) | 0.0440 (4) | 0.0797 (5) | -0.0040 (3) | 0.0359 (4) | 0.0085 (3) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C12 | 0.0832 (5) | 0.0620 (4) | 0.0845 (5) | 0.0183 (4) | 0.0174 (4) | -0.0351 (4) |
| N1 | 0.0418 (10) | 0.0307 (9) | 0.0318 (9) | 0.0035 (8) | 0.0095 (8) | -0.0038 (7) |
| N2 | 0.0493 (11) | 0.0294 (9) | 0.0259 (8) | 0.0063 (8) | 0.0126 (8) | 0.0000 (7) |
| N3 | 0.0544 (14) | 0.0335 (11) | 0.0733 (15) | 0.0013 (10) | 0.0004 (11) | -0.0153 (10) |
| O1 | 0.0735 (11) | 0.0371 (8) | 0.0285 (8) | 0.0118 (7) | 0.0187 (7) | 0.0037 (6) |
| O2 | 0.1069 (17) | 0.0392 (10) | 0.1022 (16) | -0.0029 (10) | 0.0370 (14) | 0.0029 (10) |
| O3 | 0.0933 (15) | 0.0387 (10) | 0.151 (2) | 0.0116 (10) | 0.0411 (14) | -0.0234 (12) |
| C1 | 0.0363 (12) | 0.0334 (11) | 0.0455 (12) | 0.0019 (9) | 0.0034 (10) | 0.0002 (9) |
| C2 | 0.0334 (11) | 0.0326 (11) | 0.0324 (11) | 0.0044 (9) | 0.0011 (9) | -0.0038 (8) |
| C3 | 0.0489 (14) | 0.0332 (11) | 0.0365 (12) | 0.0037 (10) | 0.0059 (10) | -0.0011 (9) |
| C4 | 0.0486 (14) | 0.0508 (14) | 0.0365 (12) | 0.0040 (11) | 0.0098 (10) | -0.0078 (10) |
| C5 | 0.0425 (13) | 0.0420 (13) | 0.0484 (13) | 0.0102 (10) | 0.0013 (11) | -0.0168 (10) |
| C6 | 0.0474 (14) | 0.0297 (11) | 0.0568 (14) | 0.0037 (10) | -0.0002 (12) | -0.0072 (10) |
| C7 | 0.0415 (13) | 0.0348 (11) | 0.0301 (11) | 0.0036 (9) | 0.0068 (9) | -0.0008 (8) |
| C8 | 0.0382 (12) | 0.0286 (10) | 0.0299 (11) | -0.0011 (9) | 0.0066 (9) | -0.0021 (8) |
| C9 | 0.0300 (11) | 0.0327 (11) | 0.0258 (10) | 0.0025 (8) | 0.0024 (8) | -0.0012 (8) |
| C10 | 0.0338 (11) | 0.0334 (11) | 0.0351 (11) | -0.0003 (9) | 0.0048 (9) | -0.0041 (8) |
| C11 | 0.0376 (12) | 0.0341 (11) | 0.0426 (12) | 0.0050 (9) | -0.0017 (10) | -0.0076 (9) |
| C12 | 0.0478 (14) | 0.0534 (15) | 0.0460 (13) | 0.0155 (11) | 0.0054 (11) | -0.0160 (11) |
| C13 | 0.0452 (14) | 0.0649 (16) | 0.0355 (12) | 0.0095 (12) | 0.0155 (10) | -0.0011 (11) |
| C14 | 0.0391 (12) | 0.0430 (12) | 0.0315 (11) | 0.0030 (10) | 0.0070 (9) | 0.0031 (9) |

Geometric parameters (Å, °)

| | | | |
|-----------|-------------|------------|-------------|
| C11—C1 | 1.736 (2) | C4—C5 | 1.376 (3) |
| C12—C5 | 1.741 (2) | C4—H4 | 0.9300 |
| N1—C7 | 1.275 (2) | C5—C6 | 1.374 (3) |
| N1—N2 | 1.384 (2) | C6—H6 | 0.9300 |
| N2—C8 | 1.350 (2) | C7—H7 | 0.9300 |
| N2—H2 | 0.889 (10) | C8—C9 | 1.499 (3) |
| N3—O3 | 1.215 (2) | C9—C14 | 1.384 (3) |
| N3—O2 | 1.216 (3) | C9—C10 | 1.392 (3) |
| N3—C11 | 1.481 (3) | C10—C11 | 1.378 (3) |
| O1—C8 | 1.232 (2) | C10—H10 | 0.9300 |
| C1—C6 | 1.395 (3) | C11—C12 | 1.379 (3) |
| C1—C2 | 1.396 (3) | C12—C13 | 1.379 (3) |
| C2—C3 | 1.397 (3) | C12—H12 | 0.9300 |
| C2—C7 | 1.463 (3) | C13—C14 | 1.395 (3) |
| C3—C4 | 1.383 (3) | C13—H13 | 0.9300 |
| C3—H3 | 0.9300 | C14—H14 | 0.9300 |
| C7—N1—N2 | 116.89 (16) | N1—C7—C2 | 118.86 (18) |
| C8—N2—N1 | 116.96 (15) | N1—C7—H7 | 120.6 |
| C8—N2—H2 | 122.3 (17) | C2—C7—H7 | 120.6 |
| N1—N2—H2 | 120.7 (17) | O1—C8—N2 | 123.05 (17) |
| O3—N3—O2 | 123.7 (2) | O1—C8—C9 | 119.84 (17) |
| O3—N3—C11 | 117.9 (2) | N2—C8—C9 | 117.11 (17) |
| O2—N3—C11 | 118.37 (19) | C14—C9—C10 | 119.82 (18) |

| | | | |
|-----------|-------------|-------------|-------------|
| C6—C1—C2 | 121.8 (2) | C14—C9—C8 | 123.54 (17) |
| C6—C1—C11 | 117.98 (16) | C10—C9—C8 | 116.48 (17) |
| C2—C1—C11 | 120.26 (16) | C11—C10—C9 | 118.99 (19) |
| C1—C2—C3 | 117.44 (18) | C11—C10—H10 | 120.5 |
| C1—C2—C7 | 122.28 (19) | C9—C10—H10 | 120.5 |
| C3—C2—C7 | 120.28 (18) | C10—C11—C12 | 122.2 (2) |
| C4—C3—C2 | 121.5 (2) | C10—C11—N3 | 118.0 (2) |
| C4—C3—H3 | 119.2 | C12—C11—N3 | 119.8 (2) |
| C2—C3—H3 | 119.2 | C11—C12—C13 | 118.4 (2) |
| C5—C4—C3 | 119.0 (2) | C11—C12—H12 | 120.8 |
| C5—C4—H4 | 120.5 | C13—C12—H12 | 120.8 |
| C3—C4—H4 | 120.5 | C12—C13—C14 | 120.8 (2) |
| C6—C5—C4 | 122.0 (2) | C12—C13—H13 | 119.6 |
| C6—C5—C12 | 119.02 (17) | C14—C13—H13 | 119.6 |
| C4—C5—C12 | 118.97 (19) | C9—C14—C13 | 119.8 (2) |
| C5—C6—C1 | 118.3 (2) | C9—C14—H14 | 120.1 |
| C5—C6—H6 | 120.9 | C13—C14—H14 | 120.1 |
| C1—C6—H6 | 120.9 | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H2...O1 ⁱ | 0.89 (1) | 2.04 (2) | 2.859 (2) | 152 (2) |

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