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# (*E*)-2-Methoxy-*N*′-(4-nitrobenzylidene)-benzohydrazide

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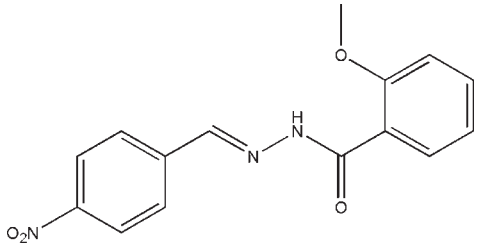
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.047; $wR$ factor = 0.138; data-to-parameter ratio = 15.0.

In the title compound, $C_{15}H_{13}N_3O_4$, the molecule exists in a *trans* configuration with respect to the methylidene unit. The dihedral angle between the two benzene rings is 6.8 (2)°. The C—N—NH—C torsion angle is 3.4 (3)°. The molecule possesses an intramolecular N—H···O hydrogen bond. In the crystal structure, adjacent molecules are linked through intermolecular C—H···O hydrogen bonds, forming dimers

## Related literature

For the biological activity of hydrazones, see: Zhong *et al.* (2007); Raj *et al.* (2007); Jimenez-Pulido *et al.* (2008). For related structures, see: Ban & Li (2008*a*,*b*); Li & Ban (2009*a*,*b*); Yehye *et al.* (2008); Fun, Patil, Jebas *et al.* (2008); Fun, Patil, Rao *et al.* (2008); Yang *et al.* (2008); Ejsmont *et al.* (2008).



## Experimental

### *Crystal data*

$C_{15}H_{13}N_3O_4$
$M_r$ = 299.28
Monoclinic, $P2_1/c$
$a$ = 11.1843 (2) Å
$b$ = 11.3718 (3) Å
$c$ = 13.0519 (2) Å
$\beta$ = 121.792 (2)°
$V$ = 1410.96 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 298 K
0.15 × 0.13 × 0.12 mm

### *Data collection*

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.985, $T_{max}$ = 0.988
8270 measured reflections
3048 independent reflections
1964 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.138
$S$ = 1.02
3048 reflections
203 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$A$···O1 | 0.91 (1) | 1.94 (2) | 2.644 (2) | 133 (2) |
| C3—H3···O2$^{i}$ | 0.93 | 2.50 | 3.260 (2) | 140 |

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2369).

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# supporting information

*Acta Cryst.* (2009). E**65**, o3272 [doi:10.1107/S160053680905079X]

## (*E*)-2-Methoxy-*N*′-(4-nitrobenzylidene)benzohydrazide

**Hong-Yan Ban and Cong-Ming Li**

### S1. Comment

Hydrazones derived from the condensation of aldehydes with hydrazides have been shown to possess excellent biological activities (Zhong *et al.*, 2007; Raj *et al.*, 2007; Jimenez-Pulido *et al.*, 2008). Due to the easy synthesis of such compounds, a great deal of hydrazones have been synthesized and structurally characterized (Yehye *et al.*, 2008; Fun, Patil, Jebas *et al.*, 2008; Fun, Patil, Rao *et al.*, 2008; Yang *et al.*, 2008; Ejsmont *et al.*, 2008). Recently, we have also reported the crystal structures of a few hydrazones (Ban & Li, 2008a,b; Li & Ban, 2009a,b). In this paper, we report the crystal structure of the title compound.

In the structure of the title compound (Fig. 1) the molecule exists in a *trans* configuration with respect to the methylidene unit. The dihedral angle between the two benzene rings is 6.8 (2)°. In the 2-methoxyphenyl unit, the methoxy group is nearly coplanar with the mean plane of the C9–C14 ring; the atom C15 deviates from this plane by 0.002 (2) Å. The torsion angle C7—N1—N2—C8 is 3.4 (3)°. The molecule possesses an intramolecular N—H···O hydrogen bond (Table 1, Fig. 1).

In the crystal structure, adjacent molecules are linked through intermolecular C—H···O hydrogen bonds (Table 1), forming dimers (Fig. 2).

### S2. Experimental

The compound was prepared by refluxing 4-nitrobenzaldehyde (1.0 mol) with 2-methoxybenzohydrazide (1.0 mol) in methanol (100 ml). Excess methanol was removed from the mixture by distillation. The colorless solid product was filtered, and washed three times with methanol. Colorless block crystals of the title compound were obtained from a methanol solution by slow evaporation in air.

### S3. Refinement

H2A, attached to N2, was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. Other H atoms were placed in calculated positions (C—H = 0.93 - 0.96 Å) and refined as riding with $U_{iso}(H) = 1.5U_{eq}$(methyl C) and $1.2U_{eq}$(other C). A rotating group model was used for the methyl group.

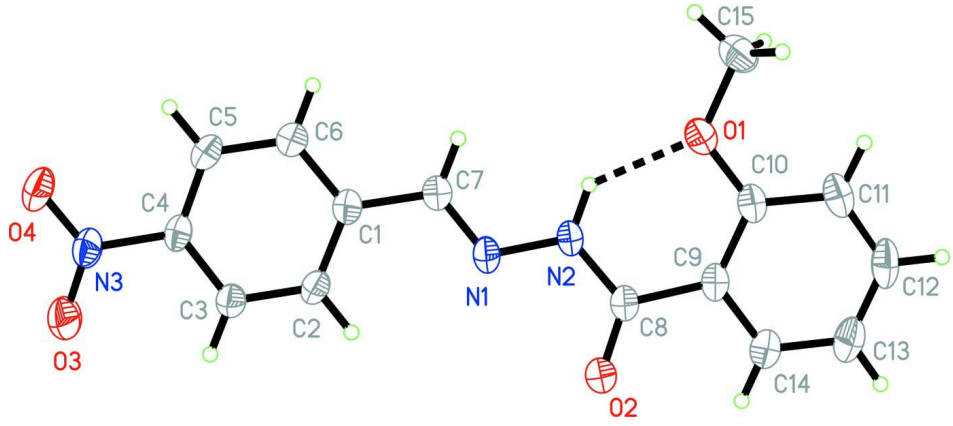


**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids for the non-hydrogen atoms. Hydrogen atoms are shown as spheres of arbitrary radius. The intramolecular N—H···O hydrogen bond is shown as a dashed line.

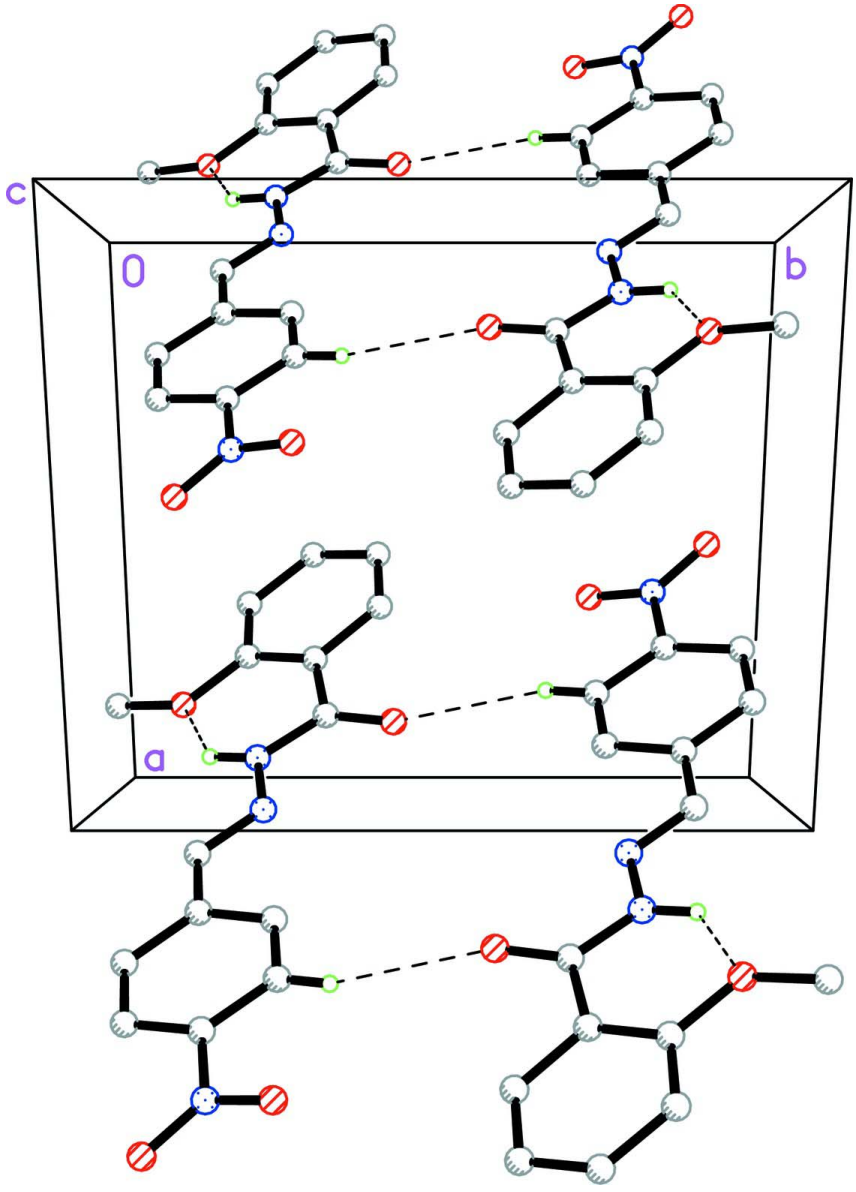


**Figure 2**

The molecular packing of the title compound, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in these hydrogen bonds have been omitted.

### (*E*)-2-Methoxy-*N*′-(4-nitrobenzylidene)benzohydrazide

*Crystal data*

$C_{15}H_{13}N_3O_4$
$M_r = 299.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.1843\ (2)$ Å
$b = 11.3718\ (3)$ Å
$c = 13.0519\ (2)$ Å
$\beta = 121.792\ (2)°$
$V = 1410.96\ (6)$ Å$^3$
$Z = 4$
$F(000) = 624$
$D_x = 1.409$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2137 reflections
$\theta = 2.6–27.7°$
$\mu = 0.11$ mm$^{-1}$

$T$ = 298 K
Block, colorless

0.15 × 0.13 × 0.12 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.985, $T_{max}$ = 0.988

8270 measured reflections
3048 independent reflections
1964 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026
$\theta_{max}$ = 27.0°, $\theta_{min}$ = 2.1°
$h$ = −14→13
$k$ = −14→9
$l$ = −16→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.138
$S$ = 1.02
3048 reflections
203 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
$w = 1/[\sigma^2(F_o^2) + (0.0691P)^2 + 0.1341P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.02468 (14) | 0.74628 (13) | 0.11097 (11) | 0.0533 (4) |
| N2 | 0.10276 (15) | 0.75892 (13) | 0.23349 (11) | 0.0542 (4) |
| N3 | −0.37828 (15) | 0.80034 (14) | −0.46112 (12) | 0.0584 (4) |
| O1 | 0.18128 (13) | 0.87506 (11) | 0.43486 (10) | 0.0632 (4) |
| O2 | 0.16350 (19) | 0.56837 (12) | 0.25823 (12) | 0.0989 (6) |
| O3 | −0.36814 (16) | 0.71210 (14) | −0.50895 (11) | 0.0841 (5) |
| O4 | −0.45977 (16) | 0.87945 (13) | −0.51687 (11) | 0.0874 (5) |
| C1 | −0.12755 (16) | 0.82699 (14) | −0.08248 (13) | 0.0477 (4) |
| C2 | −0.13227 (17) | 0.72335 (14) | −0.14102 (14) | 0.0513 (4) |
| H2 | −0.0804 | 0.6587 | −0.0958 | 0.062* |
| C3 | −0.21222 (17) | 0.71486 (15) | −0.26456 (14) | 0.0518 (4) |
| H3 | −0.2148 | 0.6454 | −0.3032 | 0.062* |
| C4 | −0.28832 (16) | 0.81139 (14) | −0.32966 (13) | 0.0477 (4) |

| | | | | |
|---|---|---|---|---|
| C5 | −0.28638 (18) | 0.91571 (15) | −0.27587 (15) | 0.0572 (4) |
| H5 | −0.3389 | 0.9798 | −0.3219 | 0.069* |
| C6 | −0.20458 (18) | 0.92326 (15) | −0.15175 (15) | 0.0559 (4) |
| H6 | −0.2009 | 0.9936 | −0.1139 | 0.067* |
| C7 | −0.04485 (17) | 0.83471 (15) | 0.04892 (14) | 0.0523 (4) |
| H7 | −0.0432 | 0.9044 | 0.0870 | 0.063* |
| C8 | 0.17233 (18) | 0.66437 (16) | 0.30264 (14) | 0.0571 (4) |
| C9 | 0.26280 (16) | 0.68339 (15) | 0.43602 (13) | 0.0507 (4) |
| C10 | 0.26528 (17) | 0.78298 (15) | 0.49941 (13) | 0.0514 (4) |
| C11 | 0.35310 (19) | 0.78490 (18) | 0.62478 (15) | 0.0643 (5) |
| H11 | 0.3528 | 0.8500 | 0.6678 | 0.077* |
| C12 | 0.4398 (2) | 0.6917 (2) | 0.68511 (16) | 0.0730 (6) |
| H12 | 0.4982 | 0.6944 | 0.7686 | 0.088* |
| C13 | 0.4412 (2) | 0.5949 (2) | 0.62399 (16) | 0.0720 (6) |
| H13 | 0.5015 | 0.5326 | 0.6653 | 0.086* |
| C14 | 0.35239 (19) | 0.59045 (17) | 0.50043 (15) | 0.0621 (5) |
| H14 | 0.3523 | 0.5238 | 0.4591 | 0.075* |
| C15 | 0.1754 (2) | 0.97515 (17) | 0.49824 (18) | 0.0720 (6) |
| H15A | 0.1474 | 0.9507 | 0.5530 | 0.108* |
| H15B | 0.1083 | 1.0306 | 0.4417 | 0.108* |
| H15C | 0.2665 | 1.0114 | 0.5426 | 0.108* |
| H2A | 0.102 (2) | 0.8277 (12) | 0.2684 (17) | 0.080* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0562 (8) | 0.0618 (9) | 0.0314 (7) | 0.0037 (7) | 0.0159 (6) | −0.0002 (6) |
| N2 | 0.0613 (9) | 0.0581 (9) | 0.0295 (7) | 0.0065 (7) | 0.0145 (6) | −0.0012 (6) |
| N3 | 0.0601 (9) | 0.0703 (10) | 0.0373 (7) | 0.0068 (8) | 0.0204 (7) | 0.0109 (7) |
| O1 | 0.0706 (8) | 0.0654 (8) | 0.0440 (6) | 0.0069 (6) | 0.0237 (6) | −0.0082 (6) |
| O2 | 0.1488 (15) | 0.0625 (9) | 0.0423 (7) | 0.0271 (9) | 0.0207 (8) | −0.0018 (6) |
| O3 | 0.1009 (11) | 0.0921 (11) | 0.0426 (7) | 0.0188 (8) | 0.0264 (7) | −0.0010 (7) |
| O4 | 0.0927 (11) | 0.0905 (10) | 0.0455 (7) | 0.0317 (8) | 0.0135 (7) | 0.0184 (7) |
| C1 | 0.0465 (9) | 0.0548 (10) | 0.0384 (8) | 0.0013 (7) | 0.0200 (7) | 0.0037 (7) |
| C2 | 0.0539 (9) | 0.0515 (10) | 0.0405 (8) | 0.0102 (7) | 0.0195 (7) | 0.0098 (7) |
| C3 | 0.0561 (10) | 0.0537 (10) | 0.0395 (8) | 0.0074 (7) | 0.0209 (7) | 0.0023 (7) |
| C4 | 0.0454 (8) | 0.0590 (10) | 0.0342 (8) | 0.0042 (7) | 0.0178 (7) | 0.0091 (7) |
| C5 | 0.0594 (10) | 0.0542 (10) | 0.0471 (9) | 0.0126 (8) | 0.0204 (8) | 0.0150 (8) |
| C6 | 0.0601 (10) | 0.0521 (10) | 0.0456 (9) | 0.0072 (8) | 0.0210 (8) | 0.0021 (7) |
| C7 | 0.0546 (10) | 0.0542 (10) | 0.0400 (8) | 0.0029 (8) | 0.0194 (7) | −0.0003 (7) |
| C8 | 0.0660 (11) | 0.0585 (11) | 0.0359 (8) | 0.0066 (8) | 0.0193 (8) | 0.0009 (8) |
| C9 | 0.0494 (9) | 0.0630 (11) | 0.0341 (8) | −0.0003 (8) | 0.0183 (7) | 0.0033 (7) |
| C10 | 0.0471 (9) | 0.0657 (11) | 0.0367 (8) | −0.0049 (8) | 0.0189 (7) | −0.0020 (7) |
| C11 | 0.0622 (11) | 0.0859 (14) | 0.0389 (9) | −0.0129 (10) | 0.0225 (8) | −0.0119 (9) |
| C12 | 0.0604 (11) | 0.1071 (17) | 0.0325 (9) | −0.0109 (11) | 0.0115 (8) | 0.0056 (10) |
| C13 | 0.0623 (12) | 0.0882 (15) | 0.0474 (10) | 0.0045 (10) | 0.0165 (9) | 0.0172 (10) |
| C14 | 0.0630 (11) | 0.0681 (12) | 0.0453 (9) | 0.0049 (9) | 0.0217 (8) | 0.0073 (8) |
| C15 | 0.0915 (14) | 0.0618 (12) | 0.0640 (12) | −0.0047 (10) | 0.0418 (11) | −0.0136 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C7 | 1.268 (2) | C5—C6 | 1.381 (2) |
| N1—N2 | 1.3672 (17) | C5—H5 | 0.9300 |
| N2—C8 | 1.355 (2) | C6—H6 | 0.9300 |
| N2—H2A | 0.908 (9) | C7—H7 | 0.9300 |
| N3—O4 | 1.2127 (18) | C8—C9 | 1.498 (2) |
| N3—O3 | 1.2183 (18) | C9—C14 | 1.393 (2) |
| N3—C4 | 1.467 (2) | C9—C10 | 1.394 (2) |
| O1—C10 | 1.3619 (19) | C10—C11 | 1.396 (2) |
| O1—C15 | 1.429 (2) | C11—C12 | 1.371 (3) |
| O2—C8 | 1.215 (2) | C11—H11 | 0.9300 |
| C1—C2 | 1.390 (2) | C12—C13 | 1.364 (3) |
| C1—C6 | 1.392 (2) | C12—H12 | 0.9300 |
| C1—C7 | 1.460 (2) | C13—C14 | 1.379 (2) |
| C2—C3 | 1.375 (2) | C13—H13 | 0.9300 |
| C2—H2 | 0.9300 | C14—H14 | 0.9300 |
| C3—C4 | 1.375 (2) | C15—H15A | 0.9600 |
| C3—H3 | 0.9300 | C15—H15B | 0.9600 |
| C4—C5 | 1.373 (2) | C15—H15C | 0.9600 |
| | | | |
| C7—N1—N2 | 117.55 (14) | C1—C7—H7 | 120.1 |
| C8—N2—N1 | 118.97 (14) | O2—C8—N2 | 121.41 (15) |
| C8—N2—H2A | 120.2 (13) | O2—C8—C9 | 121.28 (15) |
| N1—N2—H2A | 120.6 (13) | N2—C8—C9 | 117.30 (15) |
| O4—N3—O3 | 123.15 (15) | C14—C9—C10 | 118.37 (14) |
| O4—N3—C4 | 118.28 (15) | C14—C9—C8 | 114.99 (15) |
| O3—N3—C4 | 118.56 (14) | C10—C9—C8 | 126.64 (15) |
| C10—O1—C15 | 118.79 (13) | O1—C10—C9 | 117.77 (13) |
| C2—C1—C6 | 118.59 (14) | O1—C10—C11 | 122.89 (16) |
| C2—C1—C7 | 120.89 (14) | C9—C10—C11 | 119.34 (16) |
| C6—C1—C7 | 120.52 (15) | C12—C11—C10 | 120.60 (18) |
| C3—C2—C1 | 121.13 (14) | C12—C11—H11 | 119.7 |
| C3—C2—H2 | 119.4 | C10—C11—H11 | 119.7 |
| C1—C2—H2 | 119.4 | C13—C12—C11 | 120.70 (16) |
| C4—C3—C2 | 118.51 (15) | C13—C12—H12 | 119.7 |
| C4—C3—H3 | 120.7 | C11—C12—H12 | 119.7 |
| C2—C3—H3 | 120.7 | C12—C13—C14 | 119.37 (18) |
| C5—C4—C3 | 122.42 (14) | C12—C13—H13 | 120.3 |
| C5—C4—N3 | 119.23 (14) | C14—C13—H13 | 120.3 |
| C3—C4—N3 | 118.31 (15) | C13—C14—C9 | 121.58 (18) |
| C4—C5—C6 | 118.42 (15) | C13—C14—H14 | 119.2 |
| C4—C5—H5 | 120.8 | C9—C14—H14 | 119.2 |
| C6—C5—H5 | 120.8 | O1—C15—H15A | 109.5 |
| C5—C6—C1 | 120.92 (16) | O1—C15—H15B | 109.5 |
| C5—C6—H6 | 119.5 | H15A—C15—H15B | 109.5 |
| C1—C6—H6 | 119.5 | O1—C15—H15C | 109.5 |
| N1—C7—C1 | 119.88 (16) | H15A—C15—H15C | 109.5 |

| | | | |
|---|---|---|---|
| N1—C7—H7 | 120.1 | H15B—C15—H15C | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···O1 | 0.91 (1) | 1.94 (2) | 2.644 (2) | 133 (2) |
| C3—H3···O2$^{i}$ | 0.93 | 2.50 | 3.260 (2) | 140 |

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