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# *N*′-(2-Chloro-5-nitrobenzylidene)-3-hydroxybenzohydrazide methanol solvate

**Zhi Zhou**

Department of Chemistry, Kaili College, Kaili Guizhou 556000, People's Republic of China
Correspondence e-mail: zhou82zhi@126.com

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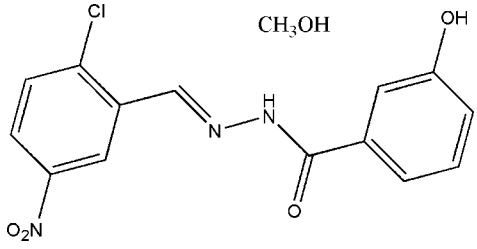
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.045; $wR$ factor = 0.122; data-to-parameter ratio = 14.9.

In the title compound, $C_{14}H_{10}ClN_3O_4\cdot CH_3OH$, the dihedral angle between the two benzene rings is 33.9 (2)°. In the crystal structure, the methanol solvent molecules are linked to the Schiff base molecules through intermolecular N—H···O and O—H···O hydrogen bonds. Molecules are further linked through intermolecular O—H···O and O—H···N hydrogen bonds, forming chains running along the $b$ axis.

## Related literature

For related structures, see: Zhou & Tang (2007); Zhou & Xiao (2007). For related literature, see: Ali *et al.* (2007); Butcher *et al.* (2007); He (2008); Jing & Yu (2007); Nie (2008).




## Experimental

### *Crystal data*

$C_{14}H_{10}ClN_3O_4\cdot CH_4O$
$M_r$ = 351.74
Monoclinic, $P2_1/c$
$a$ = 7.716 (3) Å
$b$ = 11.945 (2) Å
$c$ = 17.650 (3) Å
$\beta$ = 99.886 (2)°
$V$ = 1602.6 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.27 mm$^{-1}$
$T$ = 298 (2) K
0.27 × 0.23 × 0.22 mm

### *Data collection*

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.931, $T_{max}$ = 0.943
12656 measured reflections
3316 independent reflections
2396 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.121
$S$ = 1.01
3316 reflections
223 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4—H4···O3$^{i}$ | 0.82 | 1.91 | 2.7237 (19) | 169 |
| O4—H4···N2$^{i}$ | 0.82 | 2.62 | 3.118 (2) | 120 |
| O5—H5···O3$^{i}$ | 0.82 | 2.00 | 2.817 (2) | 175 |
| N3—H3*B*···O5 | 0.81 (3) | 2.05 (3) | 2.854 (2) | 173 (3) |

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

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*.

The author thanks Kaili College for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2226).

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# supporting information

*Acta Cryst.* (2008). E**64**, o1383 [doi:10.1107/S1600536808019636]

## *N*′-(2-Chloro-5-nitrobenzylidene)-3-hydroxybenzohydrazide methanol solvate

**Zhi Zhou**

### S1. Comment

Recently, we have reported two metal complexes with Schiff base ligands (Zhou & Tang, 2007; Zhou & Xiao, 2007). We report herein the crystal structure of the title Schiff base compound (Fig. 1).

The title compound consists of a Schiff base molecule and a methanol molecule of crystallization. The dihedral angle between the two benzene rings is 33.9 (2)°. All bond lengths are comparable to those found in similar compounds (Ali *et al.*, 2007; Nie, 2008; He, 2008; Butcher *et al.*, 2007; Jing & Yu, 2007). In the crystal structure, the methanol molecules are linked to the Schiff base molecules through intermolecular N–H···O and O–H···O hydrogen bonds (Table 1). Molecules are further linked through intermolecular O—H···O and O—H···N hydrogen bonds to form chains running along the *b* axis (Fig. 2).

### S2. Experimental

2-Chloro-5-nitrobenzaldehyde (1.0 mmol, 167.1 mg) and 3-hydroxybenzohydrazide (1.0 mmol, 152.1 mg) were dissolved in methanol (30 ml). The mixture was stirred at reflux for 30 min to give a colourless solution. After keeping the solution in air for a few days, colourless block-shaped crystals were formed.

### S3. Refinement

H3B was located in a difference Fourier map and refined isotropically, with $U_{iso}$(H) fixed at 0.08 Å$^2$. Other H atoms were positioned geometrically and refined using a riding model with O–H = 0.92 Å, C–H = 0.93-0.96 Å, and with $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C, O).

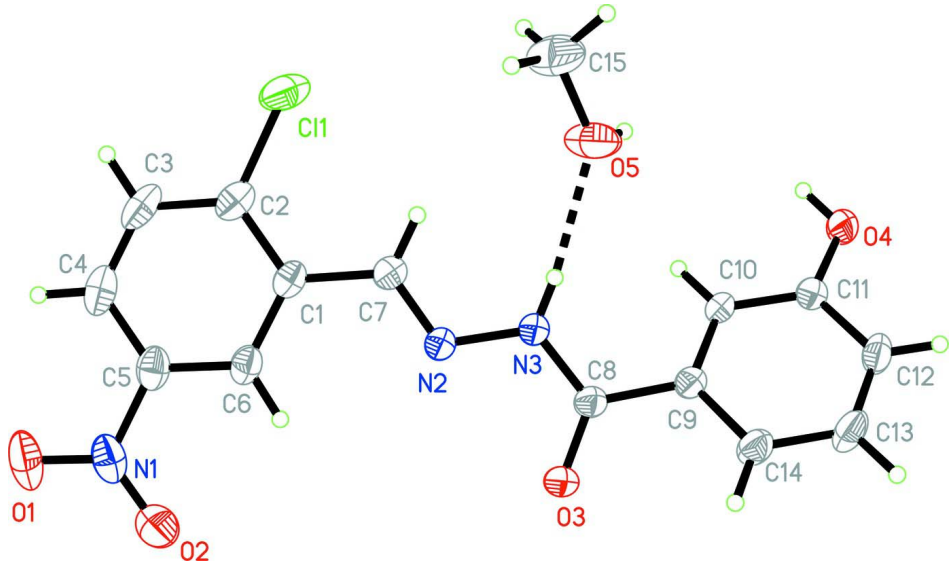


**Figure 1**

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. Hydrogen bond is shown as a dashed line.

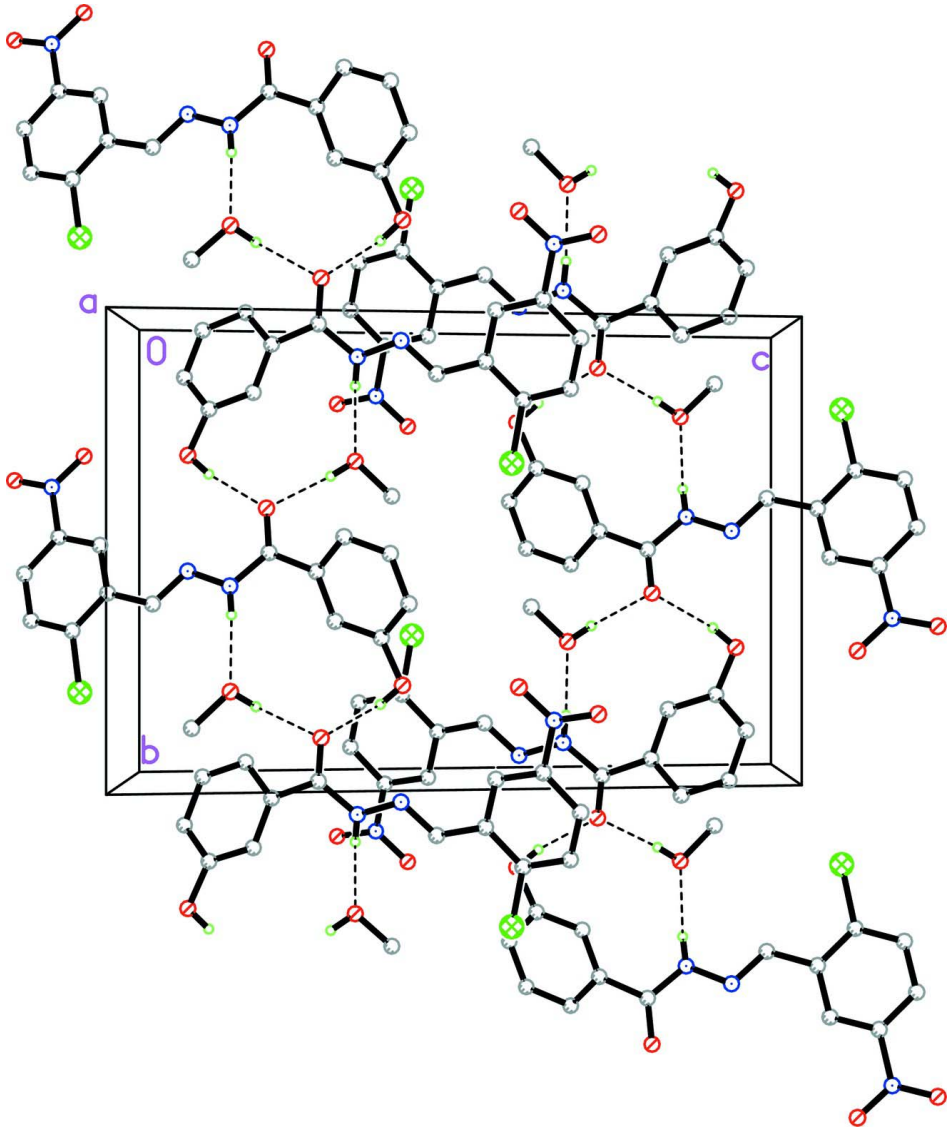


**Figure 2**

The crystal packing of the title copound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

### *N*′-(2-Chloro-5-nitrobenzylidene)-3-hydroxybenzohydrazide methanol solvate

*Crystal data*

$C_{14}H_{10}ClN_3O_4 \cdot CH_4O$
$M_r = 351.74$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.716$ (3) Å
$b = 11.945$ (2) Å
$c = 17.650$ (3) Å
$\beta = 99.886$ (2)°
$V = 1602.6$ (7) Å$^3$
$Z = 4$

$F(000) = 728$
$D_x = 1.458$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2947 reflections
$\theta = 2.3$–24.5°
$\mu = 0.27$ mm$^{-1}$
$T = 298$ K
Block, colourless
0.27 × 0.23 × 0.22 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.931, $T_{max}$ = 0.943
12656 measured reflections
3316 independent reflections
2396 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034
$\theta_{max}$ = 26.5°, $\theta_{min}$ = 2.1°
$h$ = −9→9
$k$ = −14→14
$l$ = −21→22

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.121
$S$ = 1.01
3316 reflections
223 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.2859P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.27705 (9) | 0.68944 (5) | 0.42450 (4) | 0.0774 (2) |
| O1 | −0.1188 (3) | 1.16001 (18) | 0.30340 (10) | 0.0978 (7) |
| O2 | 0.0033 (3) | 1.21390 (16) | 0.41618 (12) | 0.0859 (6) |
| O3 | 0.4707 (2) | 1.09327 (11) | 0.70885 (8) | 0.0623 (4) |
| O4 | 0.6995 (2) | 0.71117 (11) | 0.91382 (8) | 0.0607 (4) |
| H4 | 0.6479 | 0.6690 | 0.8808 | 0.091* |
| O5 | 0.6192 (3) | 0.69826 (13) | 0.66034 (11) | 0.0897 (6) |
| H5 | 0.5907 | 0.6645 | 0.6968 | 0.135* |
| N1 | −0.0240 (3) | 1.14400 (19) | 0.36561 (12) | 0.0652 (5) |
| N2 | 0.3857 (2) | 0.95587 (12) | 0.58923 (8) | 0.0428 (4) |
| N3 | 0.4964 (2) | 0.92397 (13) | 0.65533 (9) | 0.0424 (4) |
| C1 | 0.2405 (2) | 0.90699 (16) | 0.46380 (10) | 0.0439 (5) |
| C2 | 0.1934 (3) | 0.82357 (18) | 0.40857 (12) | 0.0534 (5) |
| C3 | 0.0788 (3) | 0.8448 (2) | 0.34054 (13) | 0.0684 (7) |
| H3 | 0.0502 | 0.7880 | 0.3045 | 0.082* |

| | | | | |
|---|---|---|---|---|
| C4 | 0.0082 (3) | 0.9485 (2) | 0.32639 (12) | 0.0663 (7) |
| H4A | −0.0690 | 0.9633 | 0.2810 | 0.080* |
| C5 | 0.0531 (3) | 1.03136 (19) | 0.38052 (11) | 0.0533 (5) |
| C6 | 0.1661 (3) | 1.01296 (17) | 0.44857 (10) | 0.0468 (5) |
| H6 | 0.1927 | 1.0705 | 0.4841 | 0.056* |
| C7 | 0.3601 (3) | 0.88397 (16) | 0.53602 (11) | 0.0460 (5) |
| H7 | 0.4179 | 0.8154 | 0.5428 | 0.055* |
| C8 | 0.5280 (2) | 0.99647 (15) | 0.71415 (10) | 0.0429 (4) |
| C9 | 0.6354 (2) | 0.95567 (15) | 0.78653 (10) | 0.0409 (4) |
| C10 | 0.6158 (2) | 0.84831 (15) | 0.81342 (10) | 0.0414 (4) |
| H10 | 0.5366 | 0.7992 | 0.7849 | 0.050* |
| C11 | 0.7141 (3) | 0.81385 (16) | 0.88285 (11) | 0.0451 (5) |
| C12 | 0.8325 (3) | 0.88781 (18) | 0.92424 (11) | 0.0536 (5) |
| H12 | 0.9003 | 0.8652 | 0.9704 | 0.064* |
| C13 | 0.8499 (3) | 0.99430 (19) | 0.89738 (12) | 0.0591 (6) |
| H13 | 0.9295 | 1.0432 | 0.9259 | 0.071* |
| C14 | 0.7518 (3) | 1.03021 (17) | 0.82898 (11) | 0.0511 (5) |
| H14 | 0.7633 | 1.1029 | 0.8116 | 0.061* |
| C15 | 0.6536 (4) | 0.6218 (2) | 0.60542 (17) | 0.0899 (9) |
| H15A | 0.6769 | 0.6613 | 0.5609 | 0.135* |
| H15B | 0.5535 | 0.5739 | 0.5911 | 0.135* |
| H15C | 0.7542 | 0.5775 | 0.6263 | 0.135* |
| H3B | 0.538 (3) | 0.862 (2) | 0.6593 (14) | 0.080* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0721 (4) | 0.0661 (4) | 0.0918 (5) | 0.0006 (3) | 0.0077 (3) | −0.0362 (3) |
| O1 | 0.1037 (15) | 0.1191 (16) | 0.0612 (11) | 0.0065 (12) | −0.0124 (10) | 0.0357 (11) |
| O2 | 0.0977 (14) | 0.0643 (11) | 0.0857 (13) | 0.0020 (10) | −0.0123 (11) | 0.0069 (10) |
| O3 | 0.0989 (12) | 0.0348 (8) | 0.0464 (8) | 0.0086 (7) | −0.0068 (8) | −0.0028 (6) |
| O4 | 0.0881 (12) | 0.0414 (8) | 0.0441 (8) | −0.0011 (7) | −0.0130 (7) | 0.0061 (6) |
| O5 | 0.1570 (19) | 0.0489 (10) | 0.0750 (12) | 0.0195 (10) | 0.0527 (12) | 0.0041 (8) |
| N1 | 0.0607 (12) | 0.0783 (14) | 0.0543 (12) | −0.0043 (10) | 0.0038 (10) | 0.0231 (11) |
| N2 | 0.0519 (10) | 0.0387 (8) | 0.0355 (8) | −0.0009 (7) | 0.0013 (7) | −0.0009 (7) |
| N3 | 0.0523 (10) | 0.0363 (8) | 0.0357 (8) | 0.0010 (7) | −0.0005 (7) | −0.0004 (7) |
| C1 | 0.0437 (11) | 0.0531 (12) | 0.0356 (10) | −0.0079 (8) | 0.0087 (8) | −0.0059 (8) |
| C2 | 0.0507 (12) | 0.0624 (13) | 0.0489 (12) | −0.0078 (10) | 0.0132 (10) | −0.0157 (10) |
| C3 | 0.0644 (15) | 0.0928 (19) | 0.0463 (13) | −0.0128 (14) | 0.0050 (11) | −0.0305 (13) |
| C4 | 0.0610 (14) | 0.097 (2) | 0.0378 (11) | −0.0051 (13) | 0.0000 (10) | −0.0035 (12) |
| C5 | 0.0506 (12) | 0.0699 (14) | 0.0396 (11) | −0.0102 (10) | 0.0080 (9) | 0.0053 (10) |
| C6 | 0.0508 (11) | 0.0549 (12) | 0.0343 (10) | −0.0117 (9) | 0.0061 (8) | −0.0001 (8) |
| C7 | 0.0541 (12) | 0.0415 (10) | 0.0413 (10) | −0.0005 (9) | 0.0054 (9) | −0.0035 (8) |
| C8 | 0.0526 (11) | 0.0343 (10) | 0.0403 (10) | −0.0041 (8) | 0.0041 (9) | −0.0002 (8) |
| C9 | 0.0476 (11) | 0.0382 (10) | 0.0359 (9) | −0.0012 (8) | 0.0041 (8) | −0.0030 (7) |
| C10 | 0.0477 (11) | 0.0377 (10) | 0.0359 (10) | −0.0013 (8) | −0.0012 (8) | −0.0065 (8) |
| C11 | 0.0540 (12) | 0.0415 (11) | 0.0376 (10) | 0.0039 (8) | 0.0020 (9) | −0.0016 (8) |
| C12 | 0.0553 (13) | 0.0621 (13) | 0.0379 (10) | −0.0032 (10) | −0.0075 (9) | −0.0019 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C13 | 0.0626 (14) | 0.0634 (14) | 0.0462 (12) | −0.0232 (11) | −0.0049 (10) | −0.0094 (10) |
| C14 | 0.0627 (13) | 0.0434 (11) | 0.0449 (11) | −0.0141 (9) | 0.0026 (10) | −0.0020 (9) |
| C15 | 0.109 (2) | 0.0755 (18) | 0.095 (2) | 0.0083 (16) | 0.0456 (18) | −0.0195 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C2 | 1.732 (2) | C4—C5 | 1.378 (3) |
| O1—N1 | 1.225 (2) | C4—H4A | 0.9300 |
| O2—N1 | 1.214 (3) | C5—C6 | 1.375 (3) |
| O3—C8 | 1.236 (2) | C6—H6 | 0.9300 |
| O4—C11 | 1.355 (2) | C7—H7 | 0.9300 |
| O4—H4 | 0.8200 | C8—C9 | 1.481 (2) |
| O5—C15 | 1.390 (3) | C9—C10 | 1.385 (3) |
| O5—H5 | 0.8200 | C9—C14 | 1.389 (3) |
| N1—C5 | 1.477 (3) | C10—C11 | 1.388 (3) |
| N2—C7 | 1.263 (2) | C10—H10 | 0.9300 |
| N2—N3 | 1.376 (2) | C11—C12 | 1.385 (3) |
| N3—C8 | 1.342 (2) | C12—C13 | 1.372 (3) |
| N3—H3B | 0.81 (3) | C12—H12 | 0.9300 |
| C1—C6 | 1.397 (3) | C13—C14 | 1.379 (3) |
| C1—C2 | 1.398 (3) | C13—H13 | 0.9300 |
| C1—C7 | 1.466 (3) | C14—H14 | 0.9300 |
| C2—C3 | 1.387 (3) | C15—H15A | 0.9600 |
| C3—C4 | 1.358 (4) | C15—H15B | 0.9600 |
| C3—H3 | 0.9300 | C15—H15C | 0.9600 |
| | | | |
| C11—O4—H4 | 109.5 | C1—C7—H7 | 119.5 |
| C15—O5—H5 | 109.5 | O3—C8—N3 | 122.02 (17) |
| O2—N1—O1 | 123.6 (2) | O3—C8—C9 | 120.89 (16) |
| O2—N1—C5 | 118.83 (18) | N3—C8—C9 | 117.09 (16) |
| O1—N1—C5 | 117.5 (2) | C10—C9—C14 | 120.50 (17) |
| C7—N2—N3 | 115.93 (16) | C10—C9—C8 | 121.50 (16) |
| C8—N3—N2 | 118.75 (15) | C14—C9—C8 | 117.94 (17) |
| C8—N3—H3B | 120.6 (18) | C9—C10—C11 | 120.11 (17) |
| N2—N3—H3B | 120.6 (18) | C9—C10—H10 | 119.9 |
| C6—C1—C2 | 117.57 (18) | C11—C10—H10 | 119.9 |
| C6—C1—C7 | 120.92 (17) | O4—C11—C12 | 117.23 (17) |
| C2—C1—C7 | 121.49 (18) | O4—C11—C10 | 123.60 (17) |
| C3—C2—C1 | 121.6 (2) | C12—C11—C10 | 119.16 (18) |
| C3—C2—Cl1 | 118.32 (17) | C13—C12—C11 | 120.26 (18) |
| C1—C2—Cl1 | 120.09 (16) | C13—C12—H12 | 119.9 |
| C4—C3—C2 | 120.1 (2) | C11—C12—H12 | 119.9 |
| C4—C3—H3 | 119.9 | C12—C13—C14 | 121.29 (18) |
| C2—C3—H3 | 119.9 | C12—C13—H13 | 119.4 |
| C3—C4—C5 | 118.8 (2) | C14—C13—H13 | 119.4 |
| C3—C4—H4A | 120.6 | C13—C14—C9 | 118.66 (19) |
| C5—C4—H4A | 120.6 | C13—C14—H14 | 120.7 |
| C6—C5—C4 | 122.5 (2) | C9—C14—H14 | 120.7 |

| | | | |
|---|---|---|---|
| C6—C5—N1 | 118.48 (19) | O5—C15—H15A | 109.5 |
| C4—C5—N1 | 119.0 (2) | O5—C15—H15B | 109.5 |
| C5—C6—C1 | 119.39 (19) | H15A—C15—H15B | 109.5 |
| C5—C6—H6 | 120.3 | O5—C15—H15C | 109.5 |
| C1—C6—H6 | 120.3 | H15A—C15—H15C | 109.5 |
| N2—C7—C1 | 120.94 (18) | H15B—C15—H15C | 109.5 |
| N2—C7—H7 | 119.5 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H4···O3$^{i}$ | 0.82 | 1.91 | 2.7237 (19) | 169 |
| O4—H4···N2$^{i}$ | 0.82 | 2.62 | 3.118 (2) | 120 |
| O5—H5···O3$^{i}$ | 0.82 | 2.00 | 2.817 (2) | 175 |
| N3—H3*B*···O5 | 0.81 (3) | 2.05 (3) | 2.854 (2) | 173 (3) |

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