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# *N*′-[4-(Dimethylamino)benzylidene]-3-hydroxybenzohydrazide

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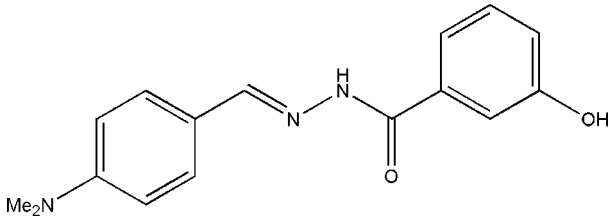
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.045; $wR$ factor = 0.131; data-to-parameter ratio = 15.8.

The title compound, $C_{16}H_{17}N_3O_2$, was synthesized by the reaction of 4-dimethylaminobenzaldehyde with 3-hydroxybenzoic acid hydrazide in methanol. The dihedral angle between the two benzene rings in the molecule is 9.2 (2)°. In the crystal structure, molecules are linked through intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds, forming layers parallel to the *bc* plane.

## Related literature

For related literature, see: Akitsu & Einaga (2006); Bahner *et al.* (1968); Butcher *et al.* (2005); Hodnett & Mooney (1970); Merchant & Chothia (1970); Pradeep (2005); Sigman & Jacobsen (1998).




## Experimental

### *Crystal data*

$C_{16}H_{17}N_3O_2$
$M_r$ = 283.33
Monoclinic, $P2_1/c$
$a$ = 13.397 (3) Å
$b$ = 9.663 (2) Å
$c$ = 11.183 (2) Å
$\beta$ = 101.97 (3)°
$V$ = 1416.2 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 298 (2) K
0.28 × 0.27 × 0.27 mm

### *Data collection*

Bruker SMART APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.975, $T_{max}$ = 0.976
11531 measured reflections
3094 independent reflections
2579 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.021

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
$S$ = 1.05
3094 reflections
196 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 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···$A$ |
|---|---|---|---|---|
| O2—H2···O1$^{i}$ | 0.82 | 2.18 | 2.8470 (14) | 138 |
| O2—H2···N2$^{i}$ | 0.82 | 2.36 | 3.1008 (16) | 150 |
| N3—H3*A*···O1$^{ii}$ | 0.895 (9) | 2.561 (11) | 3.4172 (16) | 160.4 (16) |

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2040).

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# supporting information

*Acta Cryst.* (2008). E**64**, o471 [doi:10.1107/S160053680800130X]

## *N*′-[4-(Dimethylamino)benzylidene]-3-hydroxybenzohydrazide

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### S1. Comment

Schiff base compounds have been widely investigated due to their easy synthesis, versatile structures and wide applications (Sigman & Jacobsen, 1998; Akitsu & Einaga, 2006; Pradeep, 2005; Butcher *et al.*, 2005). The excellent antibacterial and antitumor properties of such compounds have attracted much interest in recent years (Hodnett & Mooney, 1970; Bahner *et al.*, 1968; Merchant & Chothia, 1970). In order to investigate further the structures of such compounds, the new title Schiff base compound is reported on here.

The dihedral angle between the two benzene rings in the molecule (Fig. 1) of the title compound is 9.2 (2)°. In the crystal structure, molecules are linked through intermolecular O–H···O, O–H···N and N–H···O hydrogen bonds (Table 1), forming layers parallel to the *bc* plane (Fig. 2).

### S2. Experimental

The title compound was obtained by stirring of 4-dimethylaminobenzaldehyde (0.1 mmol, 14.9 mg) and 3-hydroxy-benzoic acid hydrazide (0.1 mmol, 15.2 mg) in a methanol solution (10 ml) at room temperature. Yellow block-shaped single crystals suitable for X-ray diffraction were formed from the solution after seven days.

### S3. Refinement

H3A was located from a difference Fourier map and refined with the N–H distance restrained to 0.90 (1) Å, and $U_{iso}$(H) = 0.08 Å$^2$. Other H atoms were positioned geometrically (C–H = 0.93–0.96Å and O–H = 0.82 Å) and treated as riding atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(C) and 1.5$U_{eq}$(O and methyl-C).

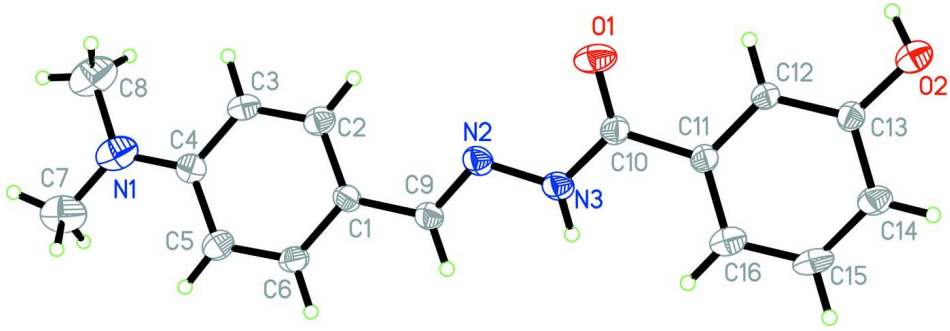


**Figure 1**

The molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids drawn at the 30% probability level.

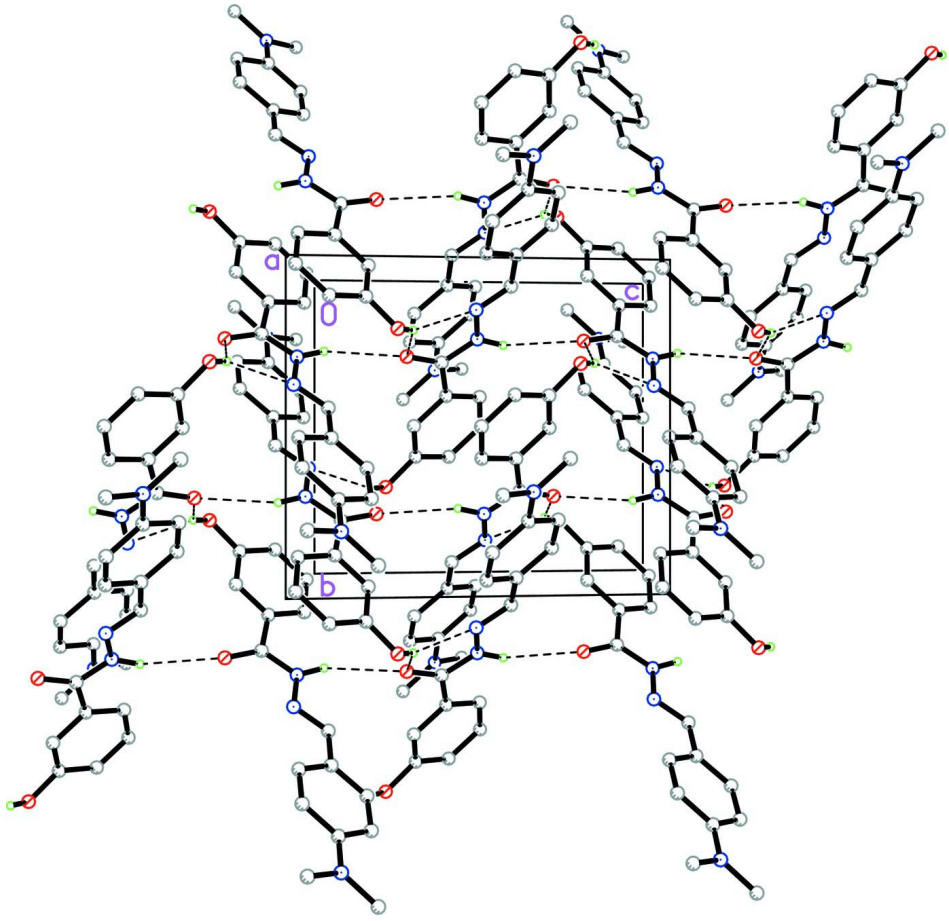


**Figure 2**

Crystal packing of the title compound view along the *a* axis [hydrogen bonds are drawn as dotted lines].

### *N*′-[4-(Dimethylamino)benzylidene]3-hydroxybenzohydrazide

*Crystal data*

$C_{16}H_{17}N_3O_2$
$M_r = 283.33$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.397\ (3)$ Å
$b = 9.663\ (2)$ Å
$c = 11.183\ (2)$ Å
$\beta = 101.97\ (3)°$
$V = 1416.2\ (5)$ Å$^3$
$Z = 4$
$F(000) = 600$
$D_x = 1.329$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4593 reflections
$\theta = 2.5–27.7°$
$\mu = 0.09$ mm$^{-1}$
$T = 298$ K
Block, yellow
0.28 × 0.27 × 0.27 mm

*Data collection*

Bruker SMART APEX area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.975$, $T_{max} = 0.976$
11531 measured reflections
3094 independent reflections
2579 reflections with $I > 2\sigma(I)$

$R_{int}$ = 0.021
$\theta_{max}$ = 27.0°, $\theta_{min}$ = 2.6°
$h$ = −16→17
$k$ = −12→12
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
$S$ = 1.05
3094 reflections
196 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
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 0.1729P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 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)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.51231 (8) | 0.27675 (11) | 0.29293 (9) | 0.0574 (3) |
| O2 | 0.28657 (7) | 0.69755 (10) | 0.22473 (9) | 0.0486 (3) |
| H2 | 0.3315 | 0.7006 | 0.1849 | 0.073* |
| N1 | 0.97117 (10) | −0.30631 (15) | 0.63913 (13) | 0.0654 (4) |
| N2 | 0.60441 (8) | 0.13189 (10) | 0.48763 (10) | 0.0416 (3) |
| N3 | 0.52359 (8) | 0.22033 (11) | 0.49046 (10) | 0.0420 (3) |
| C1 | 0.72192 (9) | −0.03254 (12) | 0.59700 (11) | 0.0379 (3) |
| C2 | 0.78863 (10) | −0.03161 (13) | 0.51647 (11) | 0.0430 (3) |
| H2A | 0.7774 | 0.0302 | 0.4513 | 0.052* |
| C3 | 0.87041 (10) | −0.11981 (14) | 0.53134 (12) | 0.0445 (3) |
| H3 | 0.9138 | −0.1157 | 0.4764 | 0.053* |
| C4 | 0.89036 (9) | −0.21613 (13) | 0.62715 (12) | 0.0425 (3) |
| C5 | 0.82405 (10) | −0.21577 (14) | 0.70911 (12) | 0.0456 (3) |
| H5 | 0.8352 | −0.2770 | 0.7747 | 0.055* |
| C6 | 0.74284 (10) | −0.12595 (14) | 0.69358 (11) | 0.0424 (3) |
| H6 | 0.7004 | −0.1277 | 0.7496 | 0.051* |
| C7 | 0.99119 (16) | −0.4061 (2) | 0.73523 (17) | 0.0793 (6) |
| H7A | 1.0129 | −0.3597 | 0.8121 | 0.119* |
| H7B | 1.0439 | −0.4679 | 0.7219 | 0.119* |
| H7C | 0.9302 | −0.4578 | 0.7363 | 0.119* |
| C8 | 1.04154 (15) | −0.2992 (2) | 0.5584 (2) | 0.0903 (7) |

| | | | | |
|---|---|---|---|---|
| H8A | 1.0063 | −0.3194 | 0.4764 | 0.135* |
| H8B | 1.0952 | −0.3655 | 0.5835 | 0.135* |
| H8C | 1.0702 | −0.2079 | 0.5612 | 0.135* |
| C9 | 0.63422 (9) | 0.05897 (13) | 0.58348 (11) | 0.0411 (3) |
| H9 | 0.5988 | 0.0645 | 0.6466 | 0.049* |
| C10 | 0.48449 (9) | 0.29458 (12) | 0.38962 (12) | 0.0397 (3) |
| C11 | 0.40510 (9) | 0.39961 (12) | 0.40150 (11) | 0.0375 (3) |
| C12 | 0.38258 (9) | 0.49733 (12) | 0.30887 (11) | 0.0375 (3) |
| H12 | 0.4168 | 0.4954 | 0.2446 | 0.045* |
| C13 | 0.30939 (9) | 0.59770 (12) | 0.31174 (11) | 0.0380 (3) |
| C14 | 0.25681 (10) | 0.59887 (14) | 0.40629 (12) | 0.0453 (3) |
| H14 | 0.2067 | 0.6651 | 0.4080 | 0.054* |
| C15 | 0.27927 (11) | 0.50121 (16) | 0.49776 (13) | 0.0530 (4) |
| H15 | 0.2440 | 0.5021 | 0.5611 | 0.064* |
| C16 | 0.35350 (10) | 0.40181 (14) | 0.49689 (12) | 0.0469 (3) |
| H16 | 0.3686 | 0.3372 | 0.5596 | 0.056* |
| H3A | 0.5095 (14) | 0.2388 (19) | 0.5637 (11) | 0.080* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0646 (6) | 0.0592 (6) | 0.0572 (6) | 0.0197 (5) | 0.0327 (5) | 0.0044 (5) |
| O2 | 0.0480 (5) | 0.0487 (5) | 0.0530 (6) | 0.0081 (4) | 0.0192 (4) | 0.0094 (4) |
| N1 | 0.0552 (7) | 0.0751 (9) | 0.0698 (8) | 0.0289 (7) | 0.0219 (6) | 0.0220 (7) |
| N2 | 0.0369 (5) | 0.0388 (6) | 0.0501 (6) | 0.0050 (4) | 0.0114 (4) | −0.0082 (4) |
| N3 | 0.0380 (5) | 0.0417 (6) | 0.0485 (6) | 0.0072 (4) | 0.0137 (5) | −0.0065 (5) |
| C1 | 0.0368 (6) | 0.0382 (6) | 0.0391 (6) | −0.0001 (5) | 0.0087 (5) | −0.0057 (5) |
| C2 | 0.0452 (7) | 0.0451 (7) | 0.0400 (6) | 0.0056 (5) | 0.0115 (5) | 0.0069 (5) |
| C3 | 0.0427 (7) | 0.0535 (8) | 0.0407 (7) | 0.0056 (5) | 0.0162 (5) | 0.0024 (5) |
| C4 | 0.0388 (6) | 0.0447 (7) | 0.0432 (7) | 0.0046 (5) | 0.0067 (5) | 0.0001 (5) |
| C5 | 0.0467 (7) | 0.0504 (7) | 0.0397 (7) | 0.0023 (6) | 0.0085 (5) | 0.0086 (5) |
| C6 | 0.0428 (7) | 0.0499 (7) | 0.0373 (6) | −0.0026 (5) | 0.0149 (5) | −0.0027 (5) |
| C7 | 0.0834 (12) | 0.0834 (13) | 0.0713 (11) | 0.0421 (10) | 0.0162 (9) | 0.0196 (9) |
| C8 | 0.0656 (11) | 0.1028 (15) | 0.1139 (16) | 0.0410 (11) | 0.0449 (11) | 0.0305 (13) |
| C9 | 0.0394 (6) | 0.0403 (6) | 0.0456 (7) | 0.0004 (5) | 0.0131 (5) | −0.0074 (5) |
| C10 | 0.0359 (6) | 0.0369 (6) | 0.0497 (7) | 0.0000 (5) | 0.0165 (5) | −0.0046 (5) |
| C11 | 0.0332 (6) | 0.0371 (6) | 0.0442 (7) | −0.0012 (5) | 0.0122 (5) | −0.0055 (5) |
| C12 | 0.0352 (6) | 0.0394 (6) | 0.0412 (6) | −0.0024 (5) | 0.0156 (5) | −0.0044 (5) |
| C13 | 0.0345 (6) | 0.0378 (6) | 0.0423 (6) | −0.0024 (5) | 0.0095 (5) | −0.0018 (5) |
| C14 | 0.0393 (6) | 0.0472 (7) | 0.0530 (7) | 0.0094 (5) | 0.0182 (5) | −0.0002 (6) |
| C15 | 0.0532 (8) | 0.0630 (9) | 0.0511 (8) | 0.0150 (6) | 0.0302 (6) | 0.0071 (6) |
| C16 | 0.0486 (7) | 0.0502 (7) | 0.0464 (7) | 0.0102 (6) | 0.0199 (6) | 0.0079 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C10 | 1.2267 (15) | C6—H6 | 0.9300 |
| O2—C13 | 1.3593 (15) | C7—H7A | 0.9600 |
| O2—H2 | 0.8200 | C7—H7B | 0.9600 |

| | | | |
|---|---|---|---|
| N1—C4 | 1.3745 (17) | C7—H7C | 0.9600 |
| N1—C7 | 1.427 (2) | C8—H8A | 0.9600 |
| N1—C8 | 1.436 (2) | C8—H8B | 0.9600 |
| N2—C9 | 1.2757 (16) | C8—H8C | 0.9600 |
| N2—N3 | 1.3848 (14) | C9—H9 | 0.9300 |
| N3—C10 | 1.3475 (17) | C10—C11 | 1.4958 (16) |
| N3—H3A | 0.895 (9) | C11—C16 | 1.3864 (18) |
| C1—C6 | 1.3907 (17) | C11—C12 | 1.3878 (17) |
| C1—C2 | 1.3942 (17) | C12—C13 | 1.3842 (17) |
| C1—C9 | 1.4531 (17) | C12—H12 | 0.9300 |
| C2—C3 | 1.3706 (17) | C13—C14 | 1.3868 (17) |
| C2—H2A | 0.9300 | C14—C15 | 1.3783 (19) |
| C3—C4 | 1.4025 (18) | C14—H14 | 0.9300 |
| C3—H3 | 0.9300 | C15—C16 | 1.3841 (18) |
| C4—C5 | 1.4025 (19) | C15—H15 | 0.9300 |
| C5—C6 | 1.3744 (18) | C16—H16 | 0.9300 |
| C5—H5 | 0.9300 | | |

| | | | |
|---|---|---|---|
| C13—O2—H2 | 109.5 | H7B—C7—H7C | 109.5 |
| C4—N1—C7 | 121.53 (13) | N1—C8—H8A | 109.5 |
| C4—N1—C8 | 120.99 (13) | N1—C8—H8B | 109.5 |
| C7—N1—C8 | 117.43 (13) | H8A—C8—H8B | 109.5 |
| C9—N2—N3 | 115.57 (10) | N1—C8—H8C | 109.5 |
| C10—N3—N2 | 118.66 (10) | H8A—C8—H8C | 109.5 |
| C10—N3—H3A | 122.7 (12) | H8B—C8—H8C | 109.5 |
| N2—N3—H3A | 117.2 (12) | N2—C9—C1 | 121.94 (11) |
| C6—C1—C2 | 116.93 (11) | N2—C9—H9 | 119.0 |
| C6—C1—C9 | 120.27 (11) | C1—C9—H9 | 119.0 |
| C2—C1—C9 | 122.80 (11) | O1—C10—N3 | 121.76 (11) |
| C3—C2—C1 | 121.39 (12) | O1—C10—C11 | 121.68 (12) |
| C3—C2—H2A | 119.3 | N3—C10—C11 | 116.56 (10) |
| C1—C2—H2A | 119.3 | C16—C11—C12 | 119.87 (11) |
| C2—C3—C4 | 121.75 (11) | C16—C11—C10 | 123.77 (11) |
| C2—C3—H3 | 119.1 | C12—C11—C10 | 116.35 (10) |
| C4—C3—H3 | 119.1 | C13—C12—C11 | 120.32 (10) |
| N1—C4—C5 | 122.05 (12) | C13—C12—H12 | 119.8 |
| N1—C4—C3 | 121.11 (12) | C11—C12—H12 | 119.8 |
| C5—C4—C3 | 116.84 (11) | O2—C13—C12 | 122.41 (10) |
| C6—C5—C4 | 120.73 (12) | O2—C13—C14 | 117.75 (11) |
| C6—C5—H5 | 119.6 | C12—C13—C14 | 119.84 (11) |
| C4—C5—H5 | 119.6 | C15—C14—C13 | 119.55 (11) |
| C5—C6—C1 | 122.33 (11) | C15—C14—H14 | 120.2 |
| C5—C6—H6 | 118.8 | C13—C14—H14 | 120.2 |
| C1—C6—H6 | 118.8 | C14—C15—C16 | 121.09 (12) |
| N1—C7—H7A | 109.5 | C14—C15—H15 | 119.5 |
| N1—C7—H7B | 109.5 | C16—C15—H15 | 119.5 |
| H7A—C7—H7B | 109.5 | C15—C16—C11 | 119.32 (12) |
| N1—C7—H7C | 109.5 | C15—C16—H16 | 120.3 |

| | | | |
|---|---|---|---|
| H7A—C7—H7C | 109.5 | C11—C16—H16 | 120.3 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···O1$^{i}$ | 0.82 | 2.18 | 2.8470 (14) | 138 |
| O2—H2···N2$^{i}$ | 0.82 | 2.36 | 3.1008 (16) | 150 |
| N3—H3*A*···O1$^{ii}$ | 0.90 (1) | 2.56 (1) | 3.4172 (16) | 160 (2) |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*+1/2.