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# 4-{[3-(4-Hydroxybenzylideneamino)-2,2-dimethylpropyl]iminiomethyl}phenolate dihydrate

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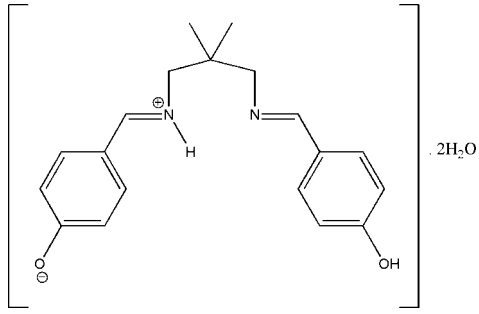
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.047; $wR$ factor = 0.116; data-to-parameter ratio = 9.5.

The asymmetric unit of the title compound, $C_{19}H_{22}N_2O_2\cdot 2H_2O$, comprises a zwitterionic form of the Schiff base compound and two water molecules of crystallization. Intermolecular N—H···O, C—H···O and O—H···N hydrogen bonds involving one of the water molecules in the asymmetric unit generate seven- and eight-membered rings, with $R_2^1(7)$ and $R_2^2(8)$ ring motifs, respectively. The dihedral angle beween the two aromatic rings is 86.5 (2)°. The imino and iminium groups are coplanar with the benzene rings to which they are attached, making dihedral angles (N—C—C—C) of −179.3 (5) and −179.2 (4)°, respectively. Validation software indicates the higher symmetry space group *Pnma* for this structure. However, this process ignores H atoms and the zwitterionic configuration of the main molecule breaks the higher symmetry. Solution in $Pna2_1$ provides a chemically sensible zwitterionic compound with improved residuals. In the crystal structure, molecules are linked together through intermolecular O—H···O, O—H···N, N—H···O and C—H···O interactions, forming a three-dimensional network. The crystal structure is further stabilized by intermolecular C—H···$\pi$ interactions.

## Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For information on Schiff base ligands and their complexes and applications, see: Calligaris & Randaccio (1987); Li *et al.* (2005); Bomfim *et al.* (2005); Glidewell *et al.* (2005, 2006); Sun *et al.* (2004). For details of the synthesis, see: Fun *et al.* (2008). For the stability of the temperature controller used for data collection, see: Cosier & Glazer (1986).




## Experimental

### Crystal data

$C_{19}H_{22}N_2O_2\cdot 2H_2O$
$M_r$ = 346.42
Orthorhombic, $Pna2_1$
$a$ = 13.0336 (4) Å
$b$ = 11.5242 (3) Å
$c$ = 12.4132 (4) Å
$V$ = 1864.49 (10) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 100 K
0.34 × 0.21 × 0.11 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.971, $T_{max}$ = 0.991
10183 measured reflections
2237 independent reflections
1753 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.056

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.116
$S$ = 1.05
2237 reflections
235 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C12–C17 benzene rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1*W*—H1*W*1···O1$^{i}$ | 0.93 | 1.91 | 2.830 (5) | 171 |
| O1*W*—H2*W*1···O2$^{ii}$ | 0.86 | 1.90 | 2.751 (4) | 168 |
| O2*W*—H1*W*2···O1*W*$^{iii}$ | 0.84 | 2.00 | 2.774 (3) | 153 |
| O2*W*—H2*W*2···N1 | 0.86 | 2.22 | 2.826 (4) | 127 |
| N2—H1N2···O2*W* | 0.95 (4) | 1.90 (4) | 2.832 (4) | 170 (3) |
| O1—H1O1···O2$^{ii}$ | 0.83 (4) | 1.74 (5) | 2.565 (5) | 174 (5) |
| C17—H17*A*···O2*W* | 0.93 | 2.52 | 3.408 (6) | 161 |
| C10—H10*B*···*Cg*1$^{iv}$ | 0.97 | 2.69 | 3.422 (5) | 133 |
| C8—H8*A*···*Cg*2$^{v}$ | 0.97 | 2.74 | 3.497 (5) | 136 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2615).

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# supporting information

*Acta Cryst.* (2009). E**65**, o1071–o1072 [doi:10.1107/S1600536809013804]

## 4-{[3-(4-Hydroxybenzylideneamino)-2,2-dimethylpropyl]iminiomethyl}-phenolate dihydrate

**Reza Kia, Hoong-Kun Fun and Hadi Kargar**

### S1. Comment

In the field of coordination chemistry, Schiff base compounds are among the most prevalent and versatile ligands. They have received much attention due to their important roles in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism, and supramolecular architectures. In comparison to the Schiff base metal complexes, only a relatively small number of free Schiff base ligands have been structurally characterized (Calligaris & Randaccio, 1987). Structures of Schiff bases derived from substituted benzaldehydes have been reported (Li *et al.*, 2005; Bomfim *et al.*, 2005; Glidewell *et al.*, 2005, 2006; Sun *et al.*, 2004).

The asymmetric unit of the title compound, Fig. 1, comprises a zwitterionic Schiff base compound and two water molecules of crystallization. The zwitterion results from protonation of the imine N2 atom with the O2 hydroxy group deprotonated resulting in the formation of iminium and phenolate groups. Intermolecular N—H···O and C—H···O hydrogen bonds involving the O2W O atom as a bifurcated acceptor generate an $R_2^1(7)$ ring motif (Bernstein *et al.*, 1995). Intermolecular N—H···O and O—H···N hydrogen bonds, again involving O2W, generate an $R_2^2(8)$ ring motif. The dihedral angle beween the two phenyl rings is 86.5 (2)°. The imino and iminium groups are coplanar with the benzene rings to which they are attached making dihedral angles of -179.3 (5) and -179.2 (4)° for N1—C7—C6—C5 and N2—C11—C12—C13, respectively. This structure requires differentiating between the *Pna*21 and *Pnma* space groups to achieve a correct solution. The solution based on the higher symmetry space group (Pnma) gives a chemically nonsensible polymeric chain as the program PLATON/ADDSYM has not taken into account H atoms in determining the symmetry elements. The solution in *Pna*21 gives a chemically sensible zwitterionic compound with lower R values. In the crystal structure, the molecules are linked together through intermolecular O—H···O, O—H···N, N—H···O and C—H···O interactions, forming a three-dimensional network (Fig. 2). The crystal structure is further stabilized by intermolecular C—H···$\pi$ interactions [*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C12–C17 benzene rings] (Table 1).

### S2. Experimental

The synthetic method has been described earlier (Fun *et al.*, 2008), except that 4-hydroxybenzaldehyde (2 mmol, 244 mg) and 2,2-dimethylpropane diamine (1 mmol, 102 mg) were used. Single crystals suitable for X-ray diffraction were obtained by evaporation of an ethanol solution at room temperature.

### S3. Refinement

The H atom of the hydroxy group was located from the difference Fourier map and refined freely. The H atoms of O2W were located from the difference Fourier map and constrained to refine with the carrier atom with $U_{iso}(H) = 1.5U_{eq}(O)$ with distance restraint of 0.85 (1) Å. The H atoms of O1W were located from the difference Fourier map and constrained

to refine with the carrier atom with $U_{iso}(H) = 1.5U_{eq}(O)$. Other N-bound and O-bound H atoms were located from the difference Fourier map and refined freely, see Table 1. The rest of the H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. In the absence of sufficient anomalous scattering, 904 Friedel pairs were merged.

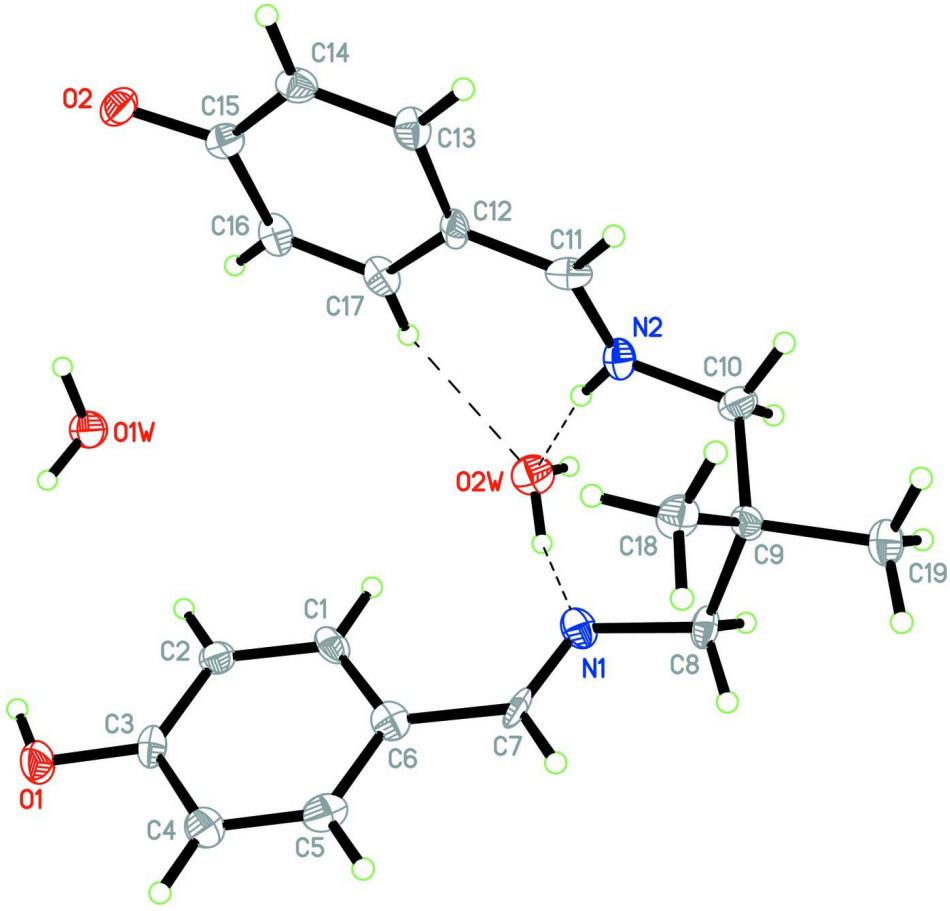


**Figure 1**

The molecular structure of the title compound with atom labels and 50% probability ellipsoids for non-H atoms. Intermolecular interactions involving the O2*W* water molecule are shown as dashed lines.

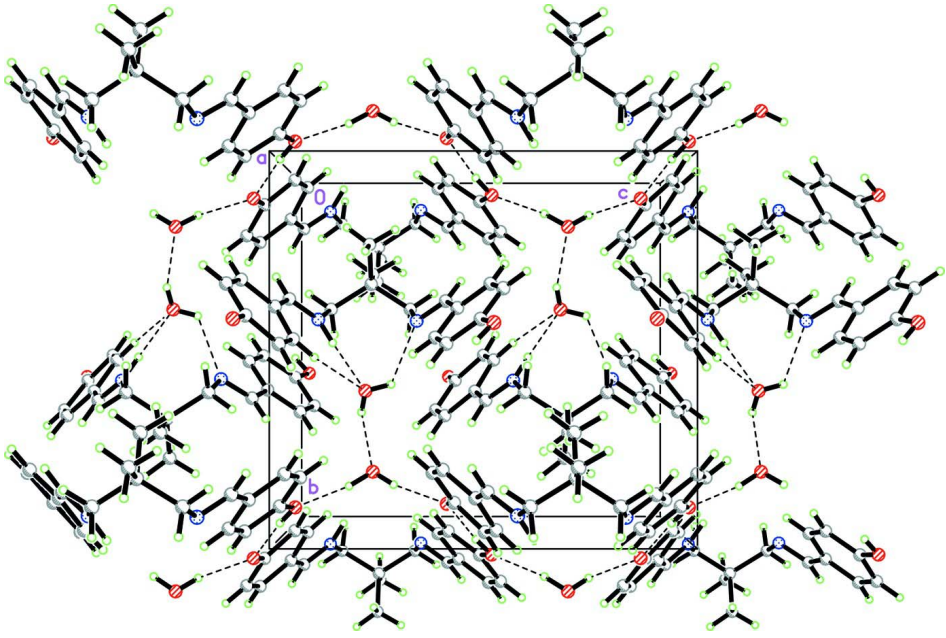


**Figure 2**

The crystal packing of the title compound, viewed down the *a* axis showing three-dimensional network formation through intermolecular interactions, shown as dashed lines.

### 4-{[3-(4-Hydroxybenzylideneamino)-2,2-dimethylpropyl]iminiomethyl}phenolate dihydrate

*Crystal data*

$C_{19}H_{22}N_2O_2\cdot 2H_2O$
$M_r = 346.42$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 13.0336\ (4)$ Å
$b = 11.5242\ (3)$ Å
$c = 12.4132\ (4)$ Å
$V = 1864.49\ (10)$ Å$^3$
$Z = 4$
$F(000) = 744$
$D_x = 1.234$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1477 reflections
$\theta = 2.4$–$29.9°$
$\mu = 0.09$ mm$^{-1}$
$T = 100$ K
Block, orange
0.34 × 0.21 × 0.11 mm

*Data collection*

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.971$, $T_{max} = 0.991$
10183 measured reflections
2237 independent reflections
1753 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -15\rightarrow16$
$k = -14\rightarrow14$
$l = -16\rightarrow7$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.116$
$S = 1.05$
2237 reflections
235 parameters
3 restraints
Primary atom site location: structure-invariant direct methods

| | |
|---|---|
| Secondary atom site location: difference Fourier map | $w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.5548P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| Hydrogen site location: inferred from neighbouring sites | $(\Delta/\sigma)_{max} < 0.001$ |
| H atoms treated by a mixture of independent and constrained refinement | $\Delta\rho_{max} = 0.27$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.20$ e Å$^{-3}$ |

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.0498 (3) | 0.5696 (3) | 1.0250 (3) | 0.0254 (8) |
| O2 | 0.0475 (3) | 0.5792 (3) | 0.4102 (2) | 0.0217 (7) |
| N1 | 0.5104 (3) | 0.5803 (3) | 0.8323 (3) | 0.0192 (8) |
| N2 | 0.5136 (3) | 0.5842 (3) | 0.5796 (3) | 0.0186 (8) |
| C1 | 0.2946 (4) | 0.5328 (4) | 0.8831 (4) | 0.0181 (10) |
| H1A | 0.3225 | 0.4895 | 0.8270 | 0.022* |
| C2 | 0.1925 (4) | 0.5165 (4) | 0.9112 (4) | 0.0178 (10) |
| H2A | 0.1528 | 0.4627 | 0.8741 | 0.021* |
| C3 | 0.1496 (4) | 0.5802 (4) | 0.9947 (4) | 0.0184 (10) |
| C4 | 0.2104 (4) | 0.6615 (4) | 1.0498 (4) | 0.0221 (10) |
| H4A | 0.1828 | 0.7050 | 1.1059 | 0.026* |
| C5 | 0.3115 (4) | 0.6764 (4) | 1.0197 (4) | 0.0211 (10) |
| H5A | 0.3510 | 0.7310 | 1.0560 | 0.025* |
| C6 | 0.3563 (4) | 0.6130 (4) | 0.9374 (4) | 0.0181 (10) |
| C7 | 0.4645 (4) | 0.6339 (4) | 0.9073 (4) | 0.0186 (10) |
| H7A | 0.5010 | 0.6896 | 0.9457 | 0.022* |
| C8 | 0.6178 (4) | 0.6143 (4) | 0.8131 (4) | 0.0172 (9) |
| H8A | 0.6594 | 0.5447 | 0.8092 | 0.021* |
| H8B | 0.6416 | 0.6593 | 0.8742 | 0.021* |
| C9 | 0.63477 (18) | 0.6854 (2) | 0.7101 (5) | 0.0166 (5) |
| C10 | 0.6196 (4) | 0.6143 (4) | 0.6059 (4) | 0.0207 (10) |
| H10A | 0.6481 | 0.6580 | 0.5463 | 0.025* |
| H10B | 0.6588 | 0.5431 | 0.6122 | 0.025* |
| C11 | 0.4577 (4) | 0.6396 (3) | 0.5096 (3) | 0.0173 (9) |
| H11A | 0.4891 | 0.7009 | 0.4737 | 0.021* |
| C12 | 0.3548 (4) | 0.6168 (4) | 0.4823 (4) | 0.0160 (9) |
| C13 | 0.3087 (4) | 0.6874 (4) | 0.4023 (4) | 0.0186 (10) |
| H13A | 0.3476 | 0.7432 | 0.3668 | 0.022* |

| | | | | |
|---|---|---|---|---|
| C14 | 0.2061 (4) | 0.6736 (4) | 0.3767 (4) | 0.0183 (9) |
| H14A | 0.1768 | 0.7206 | 0.3241 | 0.022* |
| C15 | 0.1448 (4) | 0.5894 (4) | 0.4291 (4) | 0.0172 (9) |
| C16 | 0.1949 (4) | 0.5154 (3) | 0.5056 (4) | 0.0182 (10) |
| H16A | 0.1574 | 0.4564 | 0.5382 | 0.022* |
| C17 | 0.2949 (4) | 0.5287 (3) | 0.5318 (4) | 0.0181 (10) |
| H17A | 0.3246 | 0.4798 | 0.5826 | 0.022* |
| C18 | 0.56782 (19) | 0.7944 (2) | 0.7087 (5) | 0.0209 (6) |
| H18A | 0.5770 | 0.8364 | 0.7748 | 0.031* |
| H18B | 0.4971 | 0.7725 | 0.7014 | 0.031* |
| H18C | 0.5873 | 0.8426 | 0.6491 | 0.031* |
| C19 | 0.74958 (18) | 0.7180 (2) | 0.7099 (6) | 0.0230 (6) |
| H19A | 0.7659 | 0.7590 | 0.7750 | 0.034* |
| H19B | 0.7640 | 0.7666 | 0.6490 | 0.034* |
| H19C | 0.7903 | 0.6487 | 0.7057 | 0.034* |
| O1W | 0.02979 (14) | 0.33777 (16) | 0.7189 (3) | 0.0223 (5) |
| H1W1 | 0.0021 | 0.3754 | 0.6592 | 0.033* |
| H2W1 | 0.0012 | 0.3713 | 0.7731 | 0.033* |
| O2W | 0.45692 (15) | 0.38775 (16) | 0.7030 (3) | 0.0247 (5) |
| H1W2 | 0.4922 | 0.3279 | 0.6916 | 0.037* |
| H2W2 | 0.4735 | 0.4056 | 0.7681 | 0.037* |
| H1N2 | 0.487 (3) | 0.520 (3) | 0.618 (3) | 0.020 (9)* |
| H1O1 | 0.020 (3) | 0.524 (4) | 0.984 (4) | 0.040 (14)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0152 (16) | 0.0329 (16) | 0.0281 (17) | −0.0051 (14) | 0.0031 (13) | −0.0086 (14) |
| O2 | 0.0171 (16) | 0.0261 (15) | 0.0219 (16) | −0.0001 (13) | −0.0042 (13) | 0.0062 (12) |
| N1 | 0.020 (2) | 0.0172 (18) | 0.0207 (18) | −0.0025 (17) | 0.0044 (15) | 0.0045 (16) |
| N2 | 0.0145 (19) | 0.0181 (18) | 0.0231 (19) | −0.0025 (16) | 0.0018 (15) | −0.0003 (15) |
| C1 | 0.019 (2) | 0.019 (2) | 0.016 (2) | 0.001 (2) | 0.0061 (19) | 0.0063 (18) |
| C2 | 0.018 (3) | 0.020 (2) | 0.015 (2) | −0.0032 (19) | −0.0010 (19) | 0.0004 (17) |
| C3 | 0.012 (2) | 0.0189 (19) | 0.025 (2) | −0.0004 (17) | 0.0006 (17) | 0.0025 (18) |
| C4 | 0.022 (2) | 0.0227 (19) | 0.022 (2) | 0.0024 (19) | 0.0011 (19) | −0.0022 (18) |
| C5 | 0.026 (3) | 0.0182 (18) | 0.019 (2) | −0.0060 (19) | −0.004 (2) | 0.0028 (18) |
| C6 | 0.021 (2) | 0.0139 (18) | 0.019 (2) | 0.0006 (19) | 0.0014 (18) | 0.0042 (19) |
| C7 | 0.0090 (19) | 0.024 (2) | 0.023 (2) | −0.0051 (17) | −0.0087 (18) | 0.0076 (18) |
| C8 | 0.010 (2) | 0.017 (2) | 0.025 (2) | 0.0022 (19) | −0.0017 (18) | 0.004 (2) |
| C9 | 0.0150 (11) | 0.0190 (11) | 0.0157 (13) | −0.0035 (10) | 0.002 (2) | −0.004 (2) |
| C10 | 0.021 (2) | 0.025 (2) | 0.016 (2) | −0.004 (2) | −0.0033 (19) | 0.000 (2) |
| C11 | 0.029 (2) | 0.0100 (16) | 0.013 (2) | 0.0029 (18) | −0.0015 (18) | −0.0009 (15) |
| C12 | 0.012 (2) | 0.0166 (18) | 0.020 (2) | 0.0025 (18) | 0.0026 (17) | −0.0080 (19) |
| C13 | 0.018 (2) | 0.0142 (17) | 0.024 (2) | −0.0012 (17) | 0.0025 (19) | 0.0039 (17) |
| C14 | 0.023 (2) | 0.0161 (18) | 0.016 (2) | 0.0023 (18) | 0.0008 (19) | 0.0027 (17) |
| C15 | 0.021 (2) | 0.0144 (16) | 0.017 (2) | 0.0029 (18) | −0.0014 (18) | −0.0027 (17) |
| C16 | 0.018 (3) | 0.0117 (18) | 0.025 (3) | 0.0003 (18) | 0.0044 (19) | 0.0002 (18) |
| C17 | 0.021 (2) | 0.0116 (17) | 0.022 (2) | 0.0041 (18) | 0.0054 (19) | 0.0051 (18) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C18 | 0.0220 (12) | 0.0170 (12) | 0.0238 (15) | −0.0011 (10) | −0.001 (3) | −0.006 (2) |
| C19 | 0.0181 (12) | 0.0244 (13) | 0.0265 (16) | −0.0061 (11) | 0.002 (3) | 0.001 (3) |
| O1W | 0.0244 (10) | 0.0209 (9) | 0.0216 (13) | 0.0046 (8) | −0.0003 (16) | −0.0010 (13) |
| O2W | 0.0286 (11) | 0.0201 (9) | 0.0255 (14) | 0.0002 (8) | −0.0010 (18) | −0.0041 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C3 | 1.359 (6) | C9—C10 | 1.543 (7) |
| O1—H1O1 | 0.83 (4) | C10—H10A | 0.9700 |
| O2—C15 | 1.295 (6) | C10—H10B | 0.9700 |
| N1—C7 | 1.267 (6) | C11—C12 | 1.407 (7) |
| N1—C8 | 1.473 (6) | C11—H11A | 0.9300 |
| N2—C11 | 1.302 (6) | C12—C13 | 1.418 (6) |
| N2—C10 | 1.461 (6) | C12—C17 | 1.421 (6) |
| N2—H1N2 | 0.95 (4) | C13—C14 | 1.384 (8) |
| C1—C2 | 1.388 (7) | C13—H13A | 0.9300 |
| C1—C6 | 1.398 (7) | C14—C15 | 1.415 (7) |
| C1—H1A | 0.9300 | C14—H14A | 0.9300 |
| C2—C3 | 1.387 (6) | C15—C16 | 1.434 (6) |
| C2—H2A | 0.9300 | C16—C17 | 1.352 (7) |
| C3—C4 | 1.405 (7) | C16—H16A | 0.9300 |
| C4—C5 | 1.380 (8) | C17—H17A | 0.9300 |
| C4—H4A | 0.9300 | C18—H18A | 0.9600 |
| C5—C6 | 1.385 (7) | C18—H18B | 0.9600 |
| C5—H5A | 0.9300 | C18—H18C | 0.9600 |
| C6—C7 | 1.480 (6) | C19—H19A | 0.9600 |
| C7—H7A | 0.9300 | C19—H19B | 0.9600 |
| C8—C9 | 1.535 (7) | C19—H19C | 0.9600 |
| C8—H8A | 0.9700 | O1W—H1W1 | 0.9309 |
| C8—H8B | 0.9700 | O1W—H2W1 | 0.8614 |
| C9—C18 | 1.529 (3) | O2W—H1W2 | 0.8404 |
| C9—C19 | 1.543 (3) | O2W—H2W2 | 0.8612 |
| | | | |
| C3—O1—H1O1 | 110 (4) | C9—C10—H10A | 108.3 |
| C7—N1—C8 | 115.9 (4) | N2—C10—H10B | 108.3 |
| C11—N2—C10 | 124.2 (4) | C9—C10—H10B | 108.3 |
| C11—N2—H1N2 | 121 (2) | H10A—C10—H10B | 107.4 |
| C10—N2—H1N2 | 115 (2) | N2—C11—C12 | 127.0 (4) |
| C2—C1—C6 | 121.3 (4) | N2—C11—H11A | 116.5 |
| C2—C1—H1A | 119.4 | C12—C11—H11A | 116.5 |
| C6—C1—H1A | 119.4 | C11—C12—C13 | 117.7 (4) |
| C3—C2—C1 | 120.2 (4) | C11—C12—C17 | 123.6 (4) |
| C3—C2—H2A | 119.9 | C13—C12—C17 | 118.7 (4) |
| C1—C2—H2A | 119.9 | C14—C13—C12 | 120.3 (4) |
| O1—C3—C2 | 123.0 (4) | C14—C13—H13A | 119.8 |
| O1—C3—C4 | 117.7 (4) | C12—C13—H13A | 119.8 |
| C2—C3—C4 | 119.3 (4) | C13—C14—C15 | 121.3 (4) |
| C5—C4—C3 | 119.3 (5) | C13—C14—H14A | 119.4 |

| | | | |
|---|---|---|---|
| C5—C4—H4A | 120.3 | C15—C14—H14A | 119.4 |
| C3—C4—H4A | 120.3 | O2—C15—C14 | 122.1 (4) |
| C4—C5—C6 | 122.4 (4) | O2—C15—C16 | 120.8 (4) |
| C4—C5—H5A | 118.8 | C14—C15—C16 | 117.1 (4) |
| C6—C5—H5A | 118.8 | C17—C16—C15 | 122.0 (4) |
| C5—C6—C1 | 117.5 (4) | C17—C16—H16A | 119.0 |
| C5—C6—C7 | 120.1 (4) | C15—C16—H16A | 119.0 |
| C1—C6—C7 | 122.3 (4) | C16—C17—C12 | 120.5 (4) |
| N1—C7—C6 | 123.8 (4) | C16—C17—H17A | 119.8 |
| N1—C7—H7A | 118.1 | C12—C17—H17A | 119.8 |
| C6—C7—H7A | 118.1 | C9—C18—H18A | 109.5 |
| N1—C8—C9 | 114.5 (3) | C9—C18—H18B | 109.5 |
| N1—C8—H8A | 108.6 | H18A—C18—H18B | 109.5 |
| C9—C8—H8A | 108.6 | C9—C18—H18C | 109.5 |
| N1—C8—H8B | 108.6 | H18A—C18—H18C | 109.5 |
| C9—C8—H8B | 108.6 | H18B—C18—H18C | 109.5 |
| H8A—C8—H8B | 107.6 | C9—C19—H19A | 109.5 |
| C18—C9—C8 | 111.4 (4) | C9—C19—H19B | 109.5 |
| C18—C9—C19 | 110.7 (2) | H19A—C19—H19B | 109.5 |
| C8—C9—C19 | 105.7 (4) | C9—C19—H19C | 109.5 |
| C18—C9—C10 | 110.7 (4) | H19A—C19—H19C | 109.5 |
| C8—C9—C10 | 113.3 (2) | H19B—C19—H19C | 109.5 |
| C19—C9—C10 | 104.6 (4) | H1W1—O1W—H2W1 | 104.2 |
| N2—C10—C9 | 115.8 (4) | H1W2—O2W—H2W2 | 102.5 |
| N2—C10—H10A | 108.3 | | |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | −0.1 (7) | C11—N2—C10—C9 | 99.7 (5) |
| C1—C2—C3—O1 | 178.9 (4) | C18—C9—C10—N2 | −53.5 (5) |
| C1—C2—C3—C4 | 0.4 (7) | C8—C9—C10—N2 | 72.5 (4) |
| O1—C3—C4—C5 | −178.6 (4) | C19—C9—C10—N2 | −172.8 (4) |
| C2—C3—C4—C5 | 0.0 (7) | C10—N2—C11—C12 | −178.4 (4) |
| C3—C4—C5—C6 | −0.7 (7) | N2—C11—C12—C13 | −179.2 (4) |
| C4—C5—C6—C1 | 0.9 (7) | N2—C11—C12—C17 | 1.9 (7) |
| C4—C5—C6—C7 | 179.0 (4) | C11—C12—C13—C14 | −176.2 (4) |
| C2—C1—C6—C5 | −0.6 (7) | C17—C12—C13—C14 | 2.8 (7) |
| C2—C1—C6—C7 | −178.6 (4) | C12—C13—C14—C15 | −0.1 (7) |
| C8—N1—C7—C6 | 179.1 (4) | C13—C14—C15—O2 | 176.4 (4) |
| C5—C6—C7—N1 | −179.3 (4) | C13—C14—C15—C16 | −3.1 (7) |
| C1—C6—C7—N1 | −1.3 (7) | O2—C15—C16—C17 | −175.9 (5) |
| C7—N1—C8—C9 | −106.9 (4) | C14—C15—C16—C17 | 3.6 (7) |
| N1—C8—C9—C18 | 56.0 (4) | C15—C16—C17—C12 | −1.0 (7) |
| N1—C8—C9—C19 | 176.4 (4) | C11—C12—C17—C16 | 176.6 (4) |
| N1—C8—C9—C10 | −69.6 (4) | C13—C12—C17—C16 | −2.3 (7) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*W*1···O1$^{i}$ | 0.93 | 1.91 | 2.830 (5) | 171 |

| | | | | |
|---|---|---|---|---|
| O1*W*—H2*W*1···O2$^{ii}$ | 0.86 | 1.90 | 2.751 (4) | 168 |
| O2*W*—H1*W*2···O1*W*$^{iii}$ | 0.84 | 2.00 | 2.774 (3) | 153 |
| O2*W*—H2*W*2···N1 | 0.86 | 2.22 | 2.826 (4) | 127 |
| N2—H1*N*2···O2*W* | 0.95 (4) | 1.90 (4) | 2.832 (4) | 170 (3) |
| O1—H1*O*1···O2$^{ii}$ | 0.83 (4) | 1.74 (5) | 2.565 (5) | 174 (5) |
| C17—H17*A*···O2*W* | 0.93 | 2.52 | 3.408 (6) | 161 |
| C10—H10*B*···*Cg*1$^{iv}$ | 0.97 | 2.69 | 3.422 (5) | 133 |
| C8—H8*A*···*Cg*2$^{v}$ | 0.97 | 2.74 | 3.497 (5) | 136 |

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