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# Tetramethylammonium hemi(terephthalate) dihydrate

**Yunxia Yang* and Laipeng Shi**

Key Laboratory of Polymer Materials of Gansu Province, Ministry of Education, College of Chemistry and Chemical Engineering, Northwest Normal University, Lanzhou 730070, Gansu, People's Republic of China
Correspondence e-mail: yangyx80@nwnu.edu.cn

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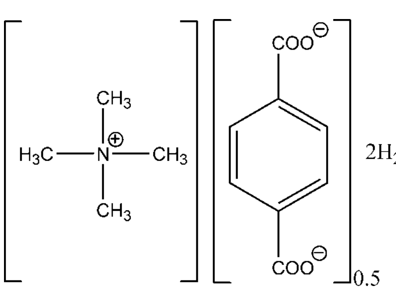
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.047; $wR$ factor = 0.140; data-to-parameter ratio = 18.9.

In the title compound, $(CH_3)_4N^+\cdot0.5C_8H_4O_4^{2-}\cdot2H_2O$, the complete terephthalate dianion is completed by twofold symmetry and has a dihedral angle of 23.5 (2)° between the carboxylate group and its parent ring. Two independent water molecules serve as both donors and acceptor in the construction of undulating hydrogen-bonded host layers with various O—H⋯O contacts ocurring between the anion and two water molecules. At the same time, the tetramethylammonium cations, as the sphere-like guest species, are arranged in two rows between neighboring host layers, with an approximate interlayer distance of 7.36 Å, forming a sandwich-like crystal structure.

## Related literature

Biphenyl-4,4′-dicarboxylic acid can be used as a host molecule in the construction of different host–guest crystal structures with various cations such as tetraethylammonium and choline ions, see: Furey *et al.* (1996); Xu *et al.* (2002).




## Experimental

### *Crystal data*

$C_4H_{12}N^+\cdot0.5C_8H_4O_4^{2-}\cdot2H_2O$
$M_r$ = 192.23
Monoclinic, $C2/c$
$a$ = 22.0950 (4) Å
$b$ = 11.2922 (2) Å
$c$ = 9.1101 (1) Å
$\beta$ = 109.613 (1)°
$V$ = 2141.10 (6) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 296 K
0.23 × 0.16 × 0.10 mm

### *Data collection*

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.979, $T_{max}$ = 0.991
6025 measured reflections
2227 independent reflections
1771 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.015

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.140
$S$ = 1.03
2227 reflections
118 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| O1$W$—H1$WA$⋯O2 | 0.87 | 1.87 | 2.7262 (17) | 168 |
| O1$W$—H1$WB$⋯O1$W^{i}$ | 0.84 | 2.41 | 2.812 (2) | 110 |
| O2$W$—H2$WA$⋯O1$^{ii}$ | 0.86 | 1.89 | 2.7291 (15) | 164 |
| O2$W$—H2$WB$⋯O1$^{iii}$ | 0.86 | 1.96 | 2.7999 (18) | 165 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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* and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5050).

## References

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconson, USA.
Furey, W. S., Sharma, C. V. K. & Zaworotko, M. J. (1996). *Supramol. Chem.* **8**, 9–11.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Xu, Z. T., Lee, S., Lobkovsky, E. B. & Kiang, Y.-H. (2002). *J. Am. Chem. Soc.* **124**, 121–135.

supporting information

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# Tetramethylammonium hemi(terephthalate) dihydrate

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## S1. Comment

Biphenyl-4,4′-dicarboxylic acid can be used as host molecule to construct different host–guest crystal structures with various cations such as tetraethylammonium and choline ions (Furey *et al.*, 1996; Xu *et al.*, 2002). In this structure, there is half a terephthalate anion disposed at the twofold axis, two water molecules, and one tetramethylammonium cation at general positions in the asymmetric unit. From the packing diagram (Fig. 2), it can be observed that terephthalate anion and two water molecules form hydrogen-bonded host layers along the *b* axis with the help of four various O—H···O hydrogen bonds between the anion and these two water molecules. The guest cations are doubly contained between the layers with an interlayer distance of $a/3 \simeq 7.36$ Å. Obviously, two independent water molecules, as the complementary host molecules, play a significant linking role in constructing the hydrogen-bonded host layer by generating four O—H···O hydrogen bonds (Fig. 3).

## S2. Experimental

Biphenyl-4,4′-dicarboxylic acid (0.042 g, 0.25 mmol) was dissolved in a water-ethanol (1:2 *v*/*v*) mixture and tetramethylammonium hydroxide was added to neutralize the acid. Colorless block crystals formed after several days.

## S3. Refinement

All hydrogen atoms bonded to carbon were introduced to idealized positions and allowed to ride on their parent atoms. Hydrogen atoms bonded to oxygen were located in difference Fourier syntheses with O—H distance of 0.86 Å.

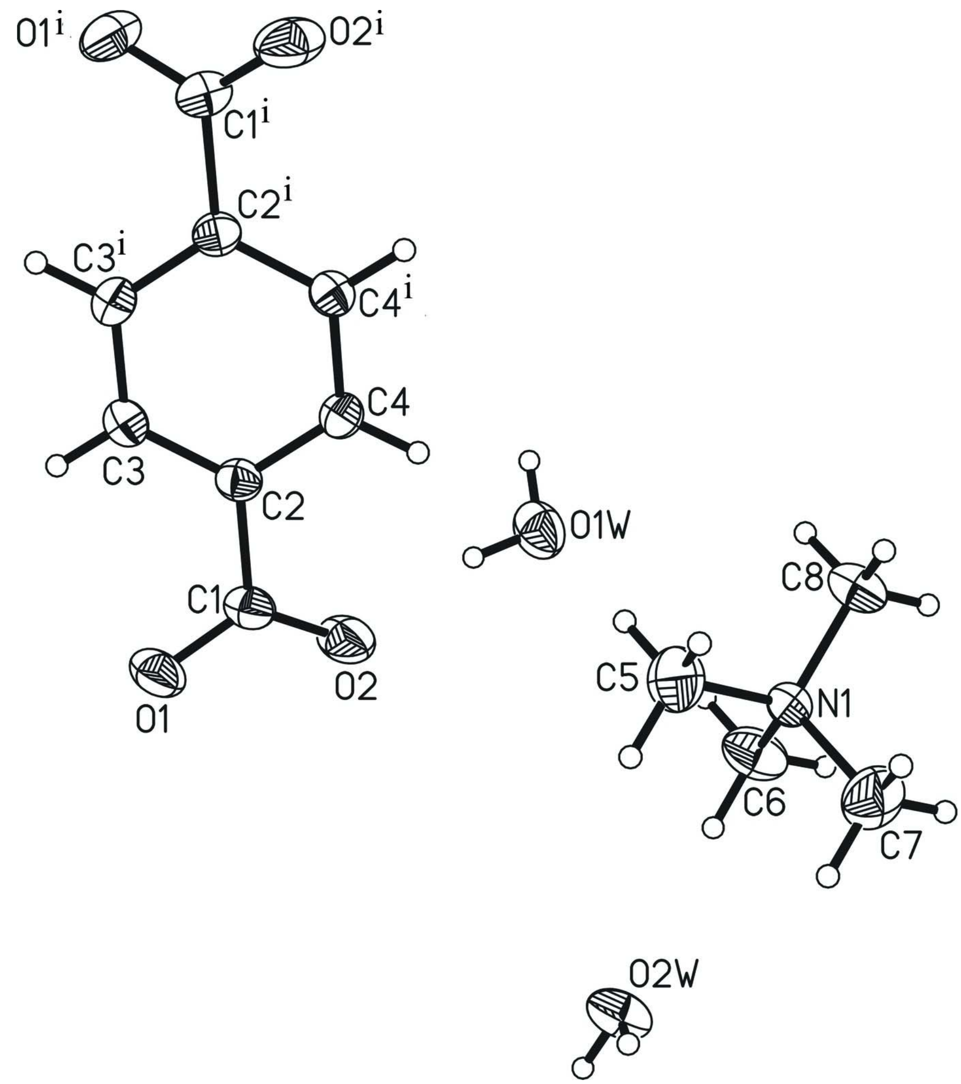


**Figure 1**

Thermal ellipsoid plot of the title compound at the 30% probability level; hydrogen atoms are drawn as spheres of arbitrary radius [Symmetry code: (i) -*x*, *y*, -*z* + 1/2.].

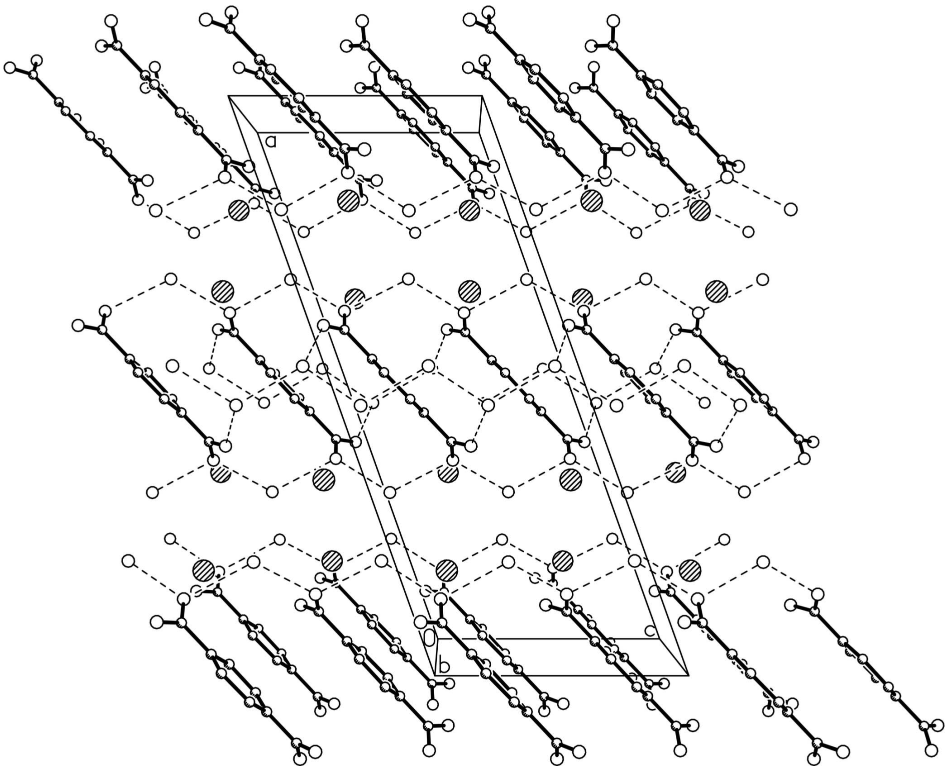


**Figure 2**

Packing diagram of the title compound; all hydrogen atoms are omitted for clarity and the cations are represented with the hatched spheres.

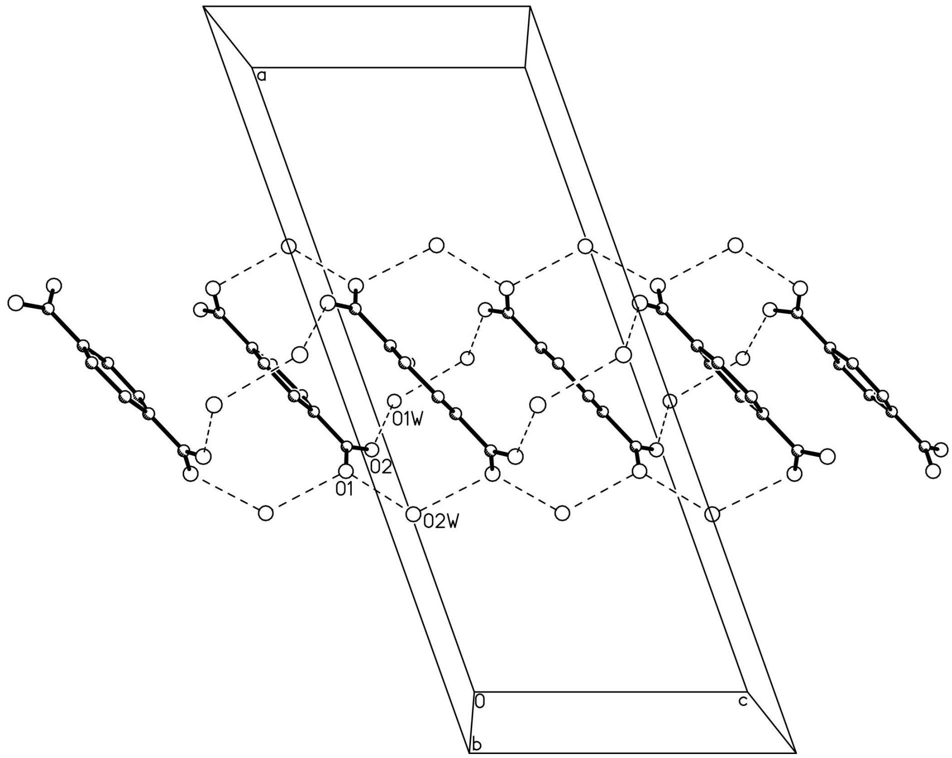


**Figure 3**

Hydrogen bond pattern in the host layer of the title compound; all hydrogen atoms are omitted for clarity.

**Tetramethylammonium hemi(terephthalate) dihydrate**

*Crystal data*

$C_4H_{12}N^+\cdot 0.5C_8H_4O_4^{2-}\cdot 2H_2O$
$M_r = 192.23$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 22.0950$ (4) Å
$b = 11.2922$ (2) Å
$c = 9.1101$ (1) Å
$\beta = 109.613$ (1)°
$V = 2141.10$ (6) Å$^3$
$Z = 8$
$F(000) = 840$
$D_x = 1.193$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2337 reflections
$\theta = 2.9$–26.4°
$\mu = 0.09$ mm$^{-1}$
$T = 296$ K
Block, colorless
0.23 × 0.16 × 0.10 mm

*Data collection*

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.979$, $T_{max} = 0.991$
6025 measured reflections
2227 independent reflections
1771 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 26.6°$, $\theta_{min} = 2.1°$
$h = -15\rightarrow 27$
$k = -12\rightarrow 14$
$l = -11\rightarrow 11$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.140$
$S = 1.03$
2227 reflections
118 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 0.7137P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.13625 (6) | 0.07836 (11) | 0.13458 (15) | 0.0647 (4) |
| C1 | 0.10126 (7) | 0.16860 (14) | 0.11158 (17) | 0.0484 (4) |
| N1 | 0.16707 (5) | 0.69678 (10) | 0.18126 (13) | 0.0424 (3) |
| O1W | 0.03390 (7) | 0.44906 (11) | −0.09000 (15) | 0.0711 (4) |
| H1WA | 0.0527 | 0.3822 | −0.0561 | 0.107* |
| H1WB | −0.0051 | 0.4402 | −0.1035 | 0.107* |
| O2 | 0.10772 (6) | 0.25714 (12) | 0.03731 (16) | 0.0753 (4) |
| C2 | 0.04837 (6) | 0.16903 (12) | 0.18277 (15) | 0.0414 (3) |
| O2W | 0.30308 (6) | 0.52166 (12) | 0.05088 (15) | 0.0716 (4) |
| H2WA | 0.3259 | 0.4825 | 0.0078 | 0.107* |
| H2WB | 0.3274 | 0.5319 | 0.1455 | 0.107* |
| C3 | 0.02429 (8) | 0.06406 (14) | 0.2185 (2) | 0.0593 (4) |
| H3A | 0.0411 | −0.0076 | 0.1997 | 0.071* |
| C4 | 0.02375 (7) | 0.27422 (12) | 0.21607 (15) | 0.0399 (3) |
| H4A | 0.0392 | 0.3458 | 0.1927 | 0.048* |
| C5 | 0.16656 (11) | 0.58985 (19) | 0.2752 (3) | 0.0846 (6) |
| H5A | 0.2052 | 0.5452 | 0.2905 | 0.127* |
| H5B | 0.1299 | 0.5419 | 0.2214 | 0.127* |
| H5C | 0.1642 | 0.6132 | 0.3745 | 0.127* |
| C6 | 0.17003 (10) | 0.66356 (19) | 0.0258 (2) | 0.0727 (6) |
| H6A | 0.2087 | 0.6198 | 0.0389 | 0.109* |
| H6B | 0.1698 | 0.7339 | −0.0335 | 0.109* |
| H6C | 0.1335 | 0.6155 | −0.0283 | 0.109* |
| C7 | 0.22409 (10) | 0.7706 (2) | 0.2642 (3) | 0.0810 (6) |
| H7A | 0.2626 | 0.7260 | 0.2777 | 0.121* |

| | | | | |
|---|---|---|---|---|
| H7B | 0.2223 | 0.7932 | 0.3642 | 0.121* |
| H7C | 0.2242 | 0.8404 | 0.2040 | 0.121* |
| C8 | 0.10738 (8) | 0.76601 (17) | 0.1586 (2) | 0.0628 (5) |
| H8A | 0.1051 | 0.7884 | 0.2583 | 0.094* |
| H8B | 0.0707 | 0.7185 | 0.1041 | 0.094* |
| H8C | 0.1078 | 0.8358 | 0.0989 | 0.094* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0632 (7) | 0.0710 (8) | 0.0713 (8) | 0.0227 (6) | 0.0374 (6) | 0.0106 (6) |
| C1 | 0.0430 (8) | 0.0600 (9) | 0.0446 (8) | 0.0082 (7) | 0.0177 (6) | 0.0054 (7) |
| N1 | 0.0390 (6) | 0.0414 (6) | 0.0456 (6) | 0.0048 (5) | 0.0126 (5) | 0.0025 (5) |
| O1W | 0.0854 (9) | 0.0637 (8) | 0.0681 (8) | 0.0174 (7) | 0.0311 (7) | 0.0150 (6) |
| O2 | 0.0725 (8) | 0.0844 (9) | 0.0881 (9) | 0.0251 (7) | 0.0522 (7) | 0.0361 (7) |
| C2 | 0.0374 (7) | 0.0464 (8) | 0.0395 (7) | 0.0025 (6) | 0.0118 (6) | 0.0016 (6) |
| O2W | 0.0567 (7) | 0.0910 (9) | 0.0671 (8) | 0.0201 (7) | 0.0206 (6) | −0.0087 (7) |
| C3 | 0.0581 (9) | 0.0401 (8) | 0.0893 (12) | 0.0052 (7) | 0.0375 (9) | −0.0021 (8) |
| C4 | 0.0432 (7) | 0.0410 (7) | 0.0355 (6) | −0.0016 (5) | 0.0134 (6) | 0.0027 (5) |
| C5 | 0.0846 (14) | 0.0652 (12) | 0.1042 (16) | 0.0117 (10) | 0.0319 (12) | 0.0371 (11) |
| C6 | 0.0670 (11) | 0.0936 (14) | 0.0606 (10) | 0.0172 (10) | 0.0255 (9) | −0.0115 (10) |
| C7 | 0.0596 (11) | 0.0790 (13) | 0.0844 (14) | −0.0132 (10) | −0.0021 (10) | −0.0082 (11) |
| C8 | 0.0541 (10) | 0.0708 (11) | 0.0661 (10) | 0.0205 (8) | 0.0235 (8) | 0.0041 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.2534 (18) | C4—C4$^{i}$ | 1.385 (3) |
| C1—O2 | 1.2420 (19) | C4—H4A | 0.9300 |
| C1—C2 | 1.5151 (19) | C5—H5A | 0.9600 |
| N1—C5 | 1.482 (2) | C5—H5B | 0.9600 |
| N1—C8 | 1.4867 (18) | C5—H5C | 0.9600 |
| N1—C6 | 1.487 (2) | C6—H6A | 0.9600 |
| N1—C7 | 1.488 (2) | C6—H6B | 0.9600 |
| O1W—H1WA | 0.8668 | C6—H6C | 0.9600 |
| O1W—H1WB | 0.8351 | C7—H7A | 0.9600 |
| C2—C4 | 1.3818 (19) | C7—H7B | 0.9600 |
| C2—C3 | 1.382 (2) | C7—H7C | 0.9600 |
| O2W—H2WA | 0.8580 | C8—H8A | 0.9600 |
| O2W—H2WB | 0.8570 | C8—H8B | 0.9600 |
| C3—C3$^{i}$ | 1.377 (3) | C8—H8C | 0.9600 |
| C3—H3A | 0.9300 | | |
| | | | |
| O2—C1—O1 | 124.64 (14) | H5A—C5—H5B | 109.5 |
| O2—C1—C2 | 118.47 (13) | N1—C5—H5C | 109.5 |
| O1—C1—C2 | 116.88 (13) | H5A—C5—H5C | 109.5 |
| C5—N1—C8 | 109.28 (14) | H5B—C5—H5C | 109.5 |
| C5—N1—C6 | 110.81 (16) | N1—C6—H6A | 109.5 |
| C8—N1—C6 | 108.73 (13) | N1—C6—H6B | 109.5 |

| | | | |
|---|---|---|---|
| C5—N1—C7 | 109.44 (15) | H6A—C6—H6B | 109.5 |
| C8—N1—C7 | 109.64 (14) | N1—C6—H6C | 109.5 |
| C6—N1—C7 | 108.92 (15) | H6A—C6—H6C | 109.5 |
| H1WA—O1W—H1WB | 107.2 | H6B—C6—H6C | 109.5 |
| C4—C2—C3 | 118.33 (13) | N1—C7—H7A | 109.5 |
| C4—C2—C1 | 120.92 (12) | N1—C7—H7B | 109.5 |
| C3—C2—C1 | 120.75 (13) | H7A—C7—H7B | 109.5 |
| H2WA—O2W—H2WB | 105.3 | N1—C7—H7C | 109.5 |
| C3$^{i}$—C3—C2 | 120.92 (8) | H7A—C7—H7C | 109.5 |
| C3$^{i}$—C3—H3A | 119.5 | H7B—C7—H7C | 109.5 |
| C2—C3—H3A | 119.5 | N1—C8—H8A | 109.5 |
| C2—C4—C4$^{i}$ | 120.73 (8) | N1—C8—H8B | 109.5 |
| C2—C4—H4A | 119.6 | H8A—C8—H8B | 109.5 |
| C4$^{i}$—C4—H4A | 119.6 | N1—C8—H8C | 109.5 |
| N1—C5—H5A | 109.5 | H8A—C8—H8C | 109.5 |
| N1—C5—H5B | 109.5 | H8B—C8—H8C | 109.5 |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*WA*···O2 | 0.87 | 1.87 | 2.7262 (17) | 168 |
| O1*W*—H1*WB*···O1*W*$^{ii}$ | 0.84 | 2.41 | 2.812 (2) | 110 |
| O2*W*—H2*WA*···O1$^{iii}$ | 0.86 | 1.89 | 2.7291 (15) | 164 |
| O2*W*—H2*WB*···O1$^{iv}$ | 0.86 | 1.96 | 2.7999 (18) | 165 |

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