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# 1,10-Phenanthrolin-1-ium hydrogen D,L-tartrate dihydrate

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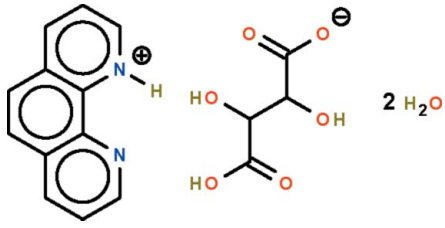
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.047; $wR$ factor = 0.160; data-to-parameter ratio = 13.6.

In the title hydrated molecular salt, $C_{12}H_9N_2^+\cdot C_4H_5O_6^-\cdot 2H_2O$, the cation is almost planar (r.m.s. deviation = 0.014 Å); the carbon skeleton of the anion assumes a *trans* conformation [C—C—C—C torsion angle = −179.86 (14)°]. The carboxyl end of one hydrogen tartrate anion forms a short hydrogen bond to the carboxylate end of another anion [O···O = 2.508 (2) Å] in a head-to-tail manner, forming a chain; the chains and water molecules interact, generating an O—H···O hydrogen-bonded layer. The cation binds to the layer by an N—H···O hydrogen bond.

## Related literature

For the trihydrated 1,10-phenanthrolin-1-ium salts of D- and L-tartaric acid, see: Derikvand & Olmstead (2010); Wang *et al.* (2006).




## Experimental

### Crystal data

$C_{12}H_9N_2^+\cdot C_4H_5O_6^-\cdot 2H_2O$
$M_r$ = 366.32
Triclinic, $P\overline{1}$
$a$ = 7.0933 (7) Å
$b$ = 10.5849 (11) Å
$c$ = 11.4694 (11) Å
$\alpha$ = 98.081 (1)°
$\beta$ = 100.350 (1)°
$\gamma$ = 103.903 (1)°
$V$ = 806.95 (14) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.12 mm$^{-1}$
$T$ = 100 K
0.40 × 0.10 × 0.10 mm

### Data collection

Bruker SMART APEX diffractometer
7610 measured reflections
3635 independent reflections
2880 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.160
$S$ = 1.04
3635 reflections
267 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O1w$^{i}$ | 0.89 (3) | 1.82 (3) | 2.709 (2) | 175 (3) |
| O4—H4···O2w$^{ii}$ | 0.90 (3) | 1.84 (3) | 2.739 (2) | 172 (3) |
| O5—H5···O2$^{iii}$ | 0.99 (3) | 1.52 (3) | 2.508 (2) | 169 (3) |
| O1w—H12···O1 | 0.93 (3) | 1.97 (3) | 2.846 (2) | 157 (3) |
| O1w—H11···O1$^{iv}$ | 0.90 (4) | 1.86 (4) | 2.753 (2) | 173 (4) |
| O2w—H21···O2 | 0.84 (4) | 1.93 (4) | 2.764 (2) | 168 (3) |
| O2w—H22···O6$^{v}$ | 0.87 (3) | 1.97 (3) | 2.835 (2) | 176 (3) |
| N1—H1···O1w | 0.93 (3) | 1.89 (3) | 2.753 (2) | 153 (3) |

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

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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5536).

## References

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# supporting information

*Acta Cryst.* (2011). E**67**, o1306 [doi:10.1107/S1600536811015972]

## 1,10-Phenanthrolin-1-ium hydrogen D,L-tartrate dihydrate

**Sit Foon Cheng and Seik Weng Ng**

### S1. Comment

**D**-tartaric acid transfers one proton to 1,10-phenanthroline to yield 1,10-phenanthroline hydrogen **D**-tartrate, which separates from solution as a trihydrate. The hydrogen **D**-tartrate anions are connected in a head-to-tail fashion by an *O*–$H_{carboxylic\ acid}\cdots O_{carboxyl}$ hydrogen bond [O···O 2.455 (1) Å] (Derikvand & Olmstead, 2010). The identical feature should be presented in the **L** analog (Wang *et al.*, 2006). The anion and water molecules are linked by extensive O–H···O hydrogen bonds into a three-dimensional network, with the cations occupying the cavities. Racemic tartaric furnishes the corresponding dihydrate. In $C_{12}H_9N_2^+\ C_4H_5O_4^-2H_2O$ (Scheme I, Fig. 1), the carboxylic acid $–CO_2H$ end of one hydrogen (**D**,**L**)-tartrate anion forms a short hydrogen bond to the carboxylate $–CO_2^-$ end of another anion [O···O 2.508 (2) Å] in a head-to-tail manner to form a chain; the chains and water molecules interact to generate an O–H···O hydrogen-bonded layer. The cation binds to the layer by an N–H···O hydrogen bond (Table 1).

### S2. Experimental

**D**,**L**-Tartaric acid (2 mmol, 0.30 g) and 1,10-phenanthroline (0.33 mmol, 0.06 g) were dissolved in water (5 ml). The solution was heated briefly to dissolve the reactants. The solution was set aside for the growth of colorless crystals, which were isolated after 10 days. The bulk crystals were faintly tinted a shade of pink.

### S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 1.00 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 *U*(C).

The ammonium and water H-atoms were located in a difference Fourier map, and were freely refined.

Omitted from the refinement owing to bad disagreements were these reflections: (-2 - 3 1), (-8 8 2), (2 2 4), (-7 6 7) and (3 - 3 4).

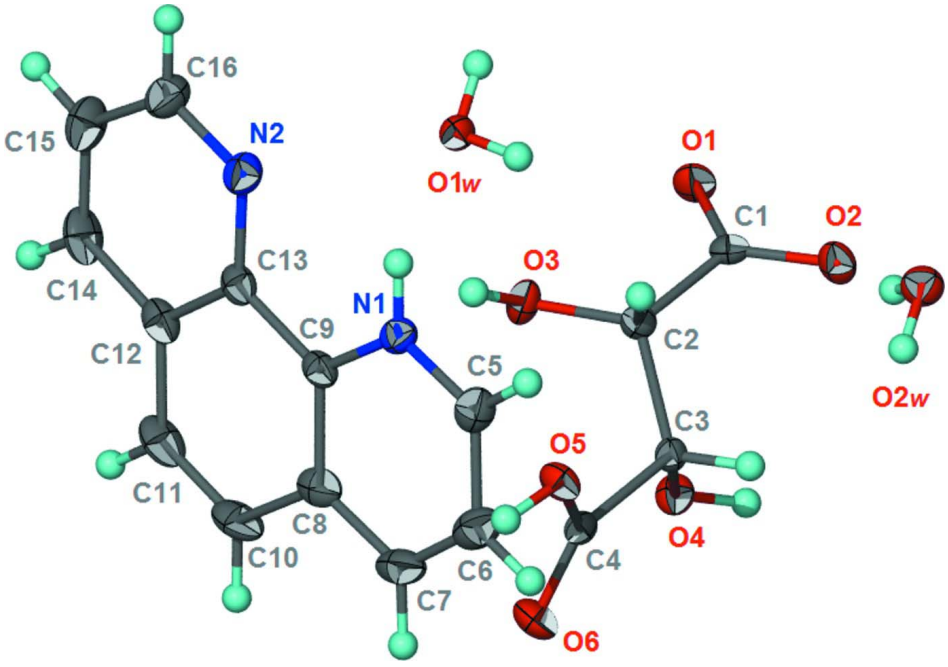


**Figure 1**

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{12}H_9N_2^+$ $C_4H_5O_6^-2H_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

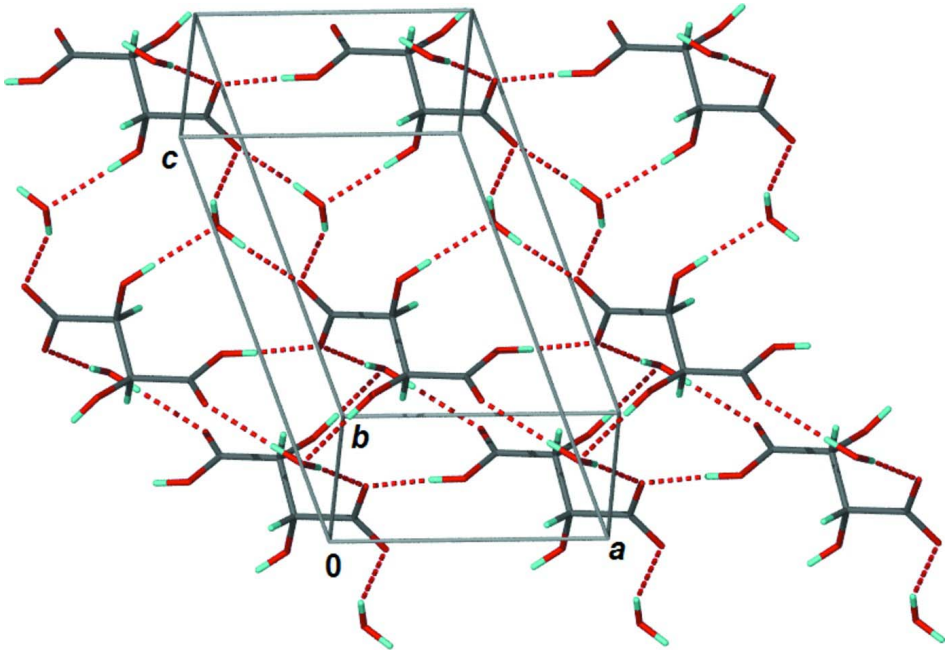


**Figure 2**

Hydrogen-bonded layer structure arising from tartrate–water interactions.

## 1,10-Phenanthrolin-1-ium hydrogen D,L-tartrate dihydrate

*Crystal data*

$C_{12}H_9N_2^+·C_4H_5O_6^-·2H_2O$
$M_r$ = 366.32
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 7.0933 (7) Å
$b$ = 10.5849 (11) Å
$c$ = 11.4694 (11) Å
$\alpha$ = 98.081 (1)°
$\beta$ = 100.350 (1)°
$\gamma$ = 103.903 (1)°
$V$ = 806.95 (14) Å$^3$
$Z$ = 2
$F(000)$ = 384
$D_x$ = 1.508 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2722 reflections
$\theta$ = 2.5–28.2°
$\mu$ = 0.12 mm$^{-1}$
$T$ = 100 K
Prism, colorless
0.40 × 0.10 × 0.10 mm

*Data collection*

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
7610 measured reflections
3635 independent reflections
2880 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.8°
$h$ = −9→9
$k$ = −13→13
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.160
$S$ = 1.04
3635 reflections
267 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
$w = 1/[\sigma^2(F_o^2) + (0.1012P)^2 + 0.1854P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.0272 (2) | 0.97612 (14) | 0.33939 (12) | 0.0172 (3) |
| O2 | 0.00294 (19) | 1.04199 (14) | 0.16223 (12) | 0.0167 (3) |
| O3 | 0.3922 (2) | 0.95680 (14) | 0.34847 (12) | 0.0163 (3) |
| O4 | 0.1832 (2) | 0.78094 (13) | 0.12086 (12) | 0.0154 (3) |
| O5 | 0.6410 (2) | 1.02428 (13) | 0.15147 (12) | 0.0153 (3) |
| O6 | 0.5609 (2) | 0.80301 (14) | 0.09605 (13) | 0.0192 (3) |
| O1W | 0.2228 (2) | 0.90933 (13) | 0.55174 (12) | 0.0145 (3) |
| O2W | 0.1595 (2) | 1.23818 (15) | 0.04324 (13) | 0.0174 (3) |
| N1 | 0.2289 (2) | 0.65380 (16) | 0.46822 (15) | 0.0143 (3) |
| N2 | 0.2713 (2) | 0.66417 (16) | 0.71074 (15) | 0.0171 (4) |
| C1 | 0.0952 (3) | 1.00580 (17) | 0.25142 (16) | 0.0125 (4) |
| C2 | 0.3085 (3) | 1.00188 (18) | 0.24666 (16) | 0.0124 (4) |

| | | | | |
|---|---|---|---|---|
| H2A | 0.3902 | 1.0934 | 0.2475 | 0.015* |
| C3 | 0.3050 (3) | 0.91028 (18) | 0.12952 (16) | 0.0115 (4) |
| H3A | 0.2505 | 0.9473 | 0.0594 | 0.014* |
| C4 | 0.5161 (3) | 0.90552 (18) | 0.12418 (16) | 0.0122 (4) |
| C5 | 0.2130 (3) | 0.6578 (2) | 0.35202 (18) | 0.0184 (4) |
| H5A | 0.2027 | 0.7366 | 0.3240 | 0.022* |
| C6 | 0.2114 (3) | 0.5459 (2) | 0.27087 (19) | 0.0220 (5) |
| H6 | 0.2015 | 0.5487 | 0.1876 | 0.026* |
| C7 | 0.2243 (3) | 0.4319 (2) | 0.31181 (19) | 0.0213 (4) |
| H7 | 0.2216 | 0.3552 | 0.2567 | 0.026* |
| C8 | 0.2417 (3) | 0.42852 (19) | 0.43589 (19) | 0.0178 (4) |
| C9 | 0.2446 (3) | 0.54419 (18) | 0.51418 (17) | 0.0138 (4) |
| C10 | 0.2550 (3) | 0.3129 (2) | 0.4856 (2) | 0.0220 (5) |
| H10 | 0.2530 | 0.2340 | 0.4337 | 0.026* |
| C11 | 0.2704 (3) | 0.3145 (2) | 0.6049 (2) | 0.0224 (5) |
| H11A | 0.2778 | 0.2366 | 0.6356 | 0.027* |
| C12 | 0.2757 (3) | 0.43249 (19) | 0.68601 (19) | 0.0173 (4) |
| C13 | 0.2645 (3) | 0.54791 (18) | 0.64134 (18) | 0.0149 (4) |
| C14 | 0.2935 (3) | 0.4390 (2) | 0.8110 (2) | 0.0227 (5) |
| H14 | 0.3004 | 0.3633 | 0.8456 | 0.027* |
| C15 | 0.3006 (3) | 0.5558 (2) | 0.8818 (2) | 0.0236 (5) |
| H15 | 0.3135 | 0.5626 | 0.9665 | 0.028* |
| C16 | 0.2888 (3) | 0.6661 (2) | 0.82806 (18) | 0.0210 (4) |
| H16 | 0.2936 | 0.7464 | 0.8787 | 0.025* |
| H3 | 0.517 (5) | 1.006 (3) | 0.381 (3) | 0.048 (9)* |
| H4 | 0.066 (4) | 0.767 (3) | 0.067 (3) | 0.032 (7)* |
| H5 | 0.781 (5) | 1.020 (3) | 0.156 (3) | 0.057 (10)* |
| H11 | 0.145 (5) | 0.944 (4) | 0.593 (4) | 0.067 (11)* |
| H12 | 0.195 (4) | 0.938 (3) | 0.480 (3) | 0.039 (8)* |
| H21 | 0.120 (5) | 1.172 (4) | 0.074 (3) | 0.052 (10)* |
| H22 | 0.247 (5) | 1.223 (3) | 0.003 (3) | 0.037 (8)* |
| H1 | 0.233 (5) | 0.731 (3) | 0.520 (3) | 0.046 (8)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0162 (7) | 0.0231 (7) | 0.0161 (7) | 0.0078 (6) | 0.0087 (5) | 0.0054 (5) |
| O2 | 0.0131 (7) | 0.0224 (7) | 0.0178 (7) | 0.0070 (5) | 0.0060 (5) | 0.0070 (6) |
| O3 | 0.0120 (7) | 0.0243 (7) | 0.0118 (7) | 0.0032 (6) | 0.0015 (5) | 0.0055 (5) |
| O4 | 0.0139 (7) | 0.0137 (7) | 0.0165 (7) | 0.0017 (5) | 0.0018 (5) | 0.0020 (5) |
| O5 | 0.0123 (7) | 0.0156 (7) | 0.0197 (7) | 0.0047 (5) | 0.0058 (5) | 0.0036 (5) |
| O6 | 0.0187 (7) | 0.0173 (7) | 0.0250 (8) | 0.0097 (6) | 0.0070 (6) | 0.0042 (6) |
| O1W | 0.0160 (7) | 0.0181 (7) | 0.0111 (7) | 0.0070 (5) | 0.0038 (5) | 0.0037 (5) |
| O2W | 0.0167 (7) | 0.0191 (7) | 0.0168 (7) | 0.0047 (6) | 0.0053 (6) | 0.0034 (6) |
| N1 | 0.0141 (8) | 0.0146 (8) | 0.0132 (8) | 0.0028 (6) | 0.0023 (6) | 0.0017 (6) |
| N2 | 0.0195 (8) | 0.0153 (8) | 0.0154 (8) | 0.0040 (6) | 0.0017 (7) | 0.0033 (6) |
| C1 | 0.0139 (9) | 0.0108 (8) | 0.0123 (9) | 0.0027 (7) | 0.0044 (7) | −0.0003 (7) |
| C2 | 0.0105 (8) | 0.0153 (9) | 0.0120 (9) | 0.0034 (7) | 0.0039 (7) | 0.0027 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.0115 (8) | 0.0142 (8) | 0.0101 (8) | 0.0042 (7) | 0.0035 (7) | 0.0041 (7) |
| C4 | 0.0146 (9) | 0.0153 (8) | 0.0083 (8) | 0.0053 (7) | 0.0036 (7) | 0.0041 (7) |
| C5 | 0.0159 (9) | 0.0225 (10) | 0.0166 (10) | 0.0039 (8) | 0.0040 (7) | 0.0051 (8) |
| C6 | 0.0196 (10) | 0.0278 (11) | 0.0169 (10) | 0.0038 (8) | 0.0059 (8) | 0.0009 (8) |
| C7 | 0.0148 (9) | 0.0226 (10) | 0.0229 (11) | 0.0041 (8) | 0.0051 (8) | −0.0062 (8) |
| C8 | 0.0110 (9) | 0.0167 (9) | 0.0230 (10) | 0.0016 (7) | 0.0039 (7) | −0.0015 (8) |
| C9 | 0.0107 (8) | 0.0125 (8) | 0.0178 (10) | 0.0037 (7) | 0.0020 (7) | 0.0015 (7) |
| C10 | 0.0184 (10) | 0.0128 (9) | 0.0330 (12) | 0.0042 (8) | 0.0063 (9) | −0.0019 (8) |
| C11 | 0.0201 (10) | 0.0122 (9) | 0.0354 (12) | 0.0058 (8) | 0.0042 (9) | 0.0065 (8) |
| C12 | 0.0131 (9) | 0.0145 (9) | 0.0239 (11) | 0.0038 (7) | 0.0020 (8) | 0.0050 (8) |
| C13 | 0.0113 (8) | 0.0134 (9) | 0.0189 (10) | 0.0029 (7) | 0.0016 (7) | 0.0031 (7) |
| C14 | 0.0227 (10) | 0.0194 (10) | 0.0282 (11) | 0.0068 (8) | 0.0026 (9) | 0.0137 (9) |
| C15 | 0.0270 (11) | 0.0248 (11) | 0.0188 (10) | 0.0056 (9) | 0.0016 (8) | 0.0105 (8) |
| C16 | 0.0262 (11) | 0.0180 (10) | 0.0164 (10) | 0.0047 (8) | 0.0016 (8) | 0.0017 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.241 (2) | C3—H3A | 1.0000 |
| O2—C1 | 1.269 (2) | C5—C6 | 1.395 (3) |
| O3—C2 | 1.410 (2) | C5—H5A | 0.9500 |
| O3—H3 | 0.89 (3) | C6—C7 | 1.372 (3) |
| O4—C3 | 1.412 (2) | C6—H6 | 0.9500 |
| O4—H4 | 0.91 (3) | C7—C8 | 1.412 (3) |
| O5—C4 | 1.311 (2) | C7—H7 | 0.9500 |
| O5—H5 | 1.00 (3) | C8—C9 | 1.406 (3) |
| O6—C4 | 1.219 (2) | C8—C10 | 1.437 (3) |
| O1W—H11 | 0.90 (4) | C9—C13 | 1.434 (3) |
| O1W—H12 | 0.92 (3) | C10—C11 | 1.350 (3) |
| O2W—H21 | 0.84 (4) | C10—H10 | 0.9500 |
| O2W—H22 | 0.87 (3) | C11—C12 | 1.437 (3) |
| N1—C5 | 1.325 (3) | C11—H11A | 0.9500 |
| N1—C9 | 1.360 (3) | C12—C13 | 1.403 (3) |
| N1—H1 | 0.93 (3) | C12—C14 | 1.407 (3) |
| N2—C16 | 1.326 (3) | C14—C15 | 1.365 (3) |
| N2—C13 | 1.354 (2) | C14—H14 | 0.9500 |
| C1—C2 | 1.534 (2) | C15—C16 | 1.407 (3) |
| C2—C3 | 1.535 (2) | C15—H15 | 0.9500 |
| C2—H2A | 1.0000 | C16—H16 | 0.9500 |
| C3—C4 | 1.522 (2) | | |

| | | | |
|---|---|---|---|
| C2—O3—H3 | 111 (2) | C5—C6—H6 | 120.1 |
| C3—O4—H4 | 110.2 (17) | C6—C7—C8 | 119.99 (19) |
| C4—O5—H5 | 111.6 (19) | C6—C7—H7 | 120.0 |
| H11—O1W—H12 | 101 (3) | C8—C7—H7 | 120.0 |
| H21—O2W—H22 | 109 (3) | C9—C8—C7 | 118.09 (19) |
| C5—N1—C9 | 122.93 (18) | C9—C8—C10 | 118.62 (19) |
| C5—N1—H1 | 117.6 (19) | C7—C8—C10 | 123.30 (19) |
| C9—N1—H1 | 119.4 (19) | N1—C9—C8 | 119.30 (18) |

| | | | |
|---|---|---|---|
| C16—N2—C13 | 116.48 (17) | N1—C9—C13 | 119.82 (17) |
| O1—C1—O2 | 125.36 (18) | C8—C9—C13 | 120.88 (18) |
| O1—C1—C2 | 119.50 (17) | C11—C10—C8 | 121.05 (19) |
| O2—C1—C2 | 115.13 (16) | C11—C10—H10 | 119.5 |
| O3—C2—C1 | 109.60 (14) | C8—C10—H10 | 119.5 |
| O3—C2—C3 | 110.78 (15) | C10—C11—C12 | 120.86 (19) |
| C1—C2—C3 | 109.30 (14) | C10—C11—H11A | 119.6 |
| O3—C2—H2A | 109.0 | C12—C11—H11A | 119.6 |
| C1—C2—H2A | 109.0 | C13—C12—C14 | 117.29 (18) |
| C3—C2—H2A | 109.0 | C13—C12—C11 | 119.93 (19) |
| O4—C3—C4 | 109.91 (14) | C14—C12—C11 | 122.78 (18) |
| O4—C3—C2 | 111.42 (14) | N2—C13—C12 | 124.24 (18) |
| C4—C3—C2 | 109.66 (14) | N2—C13—C9 | 117.11 (17) |
| O4—C3—H3A | 108.6 | C12—C13—C9 | 118.65 (18) |
| C4—C3—H3A | 108.6 | C15—C14—C12 | 118.92 (19) |
| C2—C3—H3A | 108.6 | C15—C14—H14 | 120.5 |
| O6—C4—O5 | 124.72 (17) | C12—C14—H14 | 120.5 |
| O6—C4—C3 | 123.46 (17) | C14—C15—C16 | 119.4 (2) |
| O5—C4—C3 | 111.81 (15) | C14—C15—H15 | 120.3 |
| N1—C5—C6 | 119.94 (19) | C16—C15—H15 | 120.3 |
| N1—C5—H5A | 120.0 | N2—C16—C15 | 123.7 (2) |
| C6—C5—H5A | 120.0 | N2—C16—H16 | 118.1 |
| C7—C6—C5 | 119.74 (19) | C15—C16—H16 | 118.1 |
| C7—C6—H6 | 120.1 | | |

| | | | |
|---|---|---|---|
| O1—C1—C2—O3 | 2.5 (2) | C10—C8—C9—C13 | 1.3 (3) |
| O2—C1—C2—O3 | −178.33 (15) | C9—C8—C10—C11 | −0.1 (3) |
| O1—C1—C2—C3 | 124.08 (18) | C7—C8—C10—C11 | −179.65 (19) |
| O2—C1—C2—C3 | −56.7 (2) | C8—C10—C11—C12 | −0.6 (3) |
| O3—C2—C3—O4 | 62.92 (19) | C10—C11—C12—C13 | 0.1 (3) |
| C1—C2—C3—O4 | −57.94 (19) | C10—C11—C12—C14 | −179.4 (2) |
| O3—C2—C3—C4 | −58.99 (19) | C16—N2—C13—C12 | −0.3 (3) |
| C1—C2—C3—C4 | −179.86 (14) | C16—N2—C13—C9 | 179.70 (17) |
| O4—C3—C4—O6 | 11.4 (2) | C14—C12—C13—N2 | 0.6 (3) |
| C2—C3—C4—O6 | 134.17 (19) | C11—C12—C13—N2 | −178.96 (18) |
| O4—C3—C4—O5 | −169.64 (14) | C14—C12—C13—C9 | −179.49 (17) |
| C2—C3—C4—O5 | −46.8 (2) | C11—C12—C13—C9 | 1.0 (3) |
| C9—N1—C5—C6 | 0.1 (3) | N1—C9—C13—N2 | −1.5 (3) |
| N1—C5—C6—C7 | 0.7 (3) | C8—C9—C13—N2 | 178.28 (17) |
| C5—C6—C7—C8 | −0.8 (3) | N1—C9—C13—C12 | 178.54 (17) |
| C6—C7—C8—C9 | 0.2 (3) | C8—C9—C13—C12 | −1.7 (3) |
| C6—C7—C8—C10 | 179.72 (19) | C13—C12—C14—C15 | −0.6 (3) |
| C5—N1—C9—C8 | −0.8 (3) | C11—C12—C14—C15 | 178.92 (19) |
| C5—N1—C9—C13 | 179.01 (18) | C12—C14—C15—C16 | 0.4 (3) |
| C7—C8—C9—N1 | 0.6 (3) | C13—N2—C16—C15 | 0.1 (3) |
| C10—C8—C9—N1 | −178.97 (17) | C14—C15—C16—N2 | −0.2 (3) |
| C7—C8—C9—C13 | −179.19 (17) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O1w$^{i}$ | 0.89 (3) | 1.82 (3) | 2.709 (2) | 175 (3) |
| O4—H4···O2w$^{ii}$ | 0.90 (3) | 1.84 (3) | 2.739 (2) | 172 (3) |
| O5—H5···O2$^{iii}$ | 0.99 (3) | 1.52 (3) | 2.508 (2) | 169 (3) |
| O1w—H12···O1 | 0.93 (3) | 1.97 (3) | 2.846 (2) | 157 (3) |
| O1w—H11···O1$^{iv}$ | 0.90 (4) | 1.86 (4) | 2.753 (2) | 173 (4) |
| O2w—H21···O2 | 0.84 (4) | 1.93 (4) | 2.764 (2) | 168 (3) |
| O2w—H22···O6$^{v}$ | 0.87 (3) | 1.97 (3) | 2.835 (2) | 176 (3) |
| N1—H1···O1w | 0.93 (3) | 1.89 (3) | 2.753 (2) | 153 (3) |

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