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Bis(tetraethylammonium) oxalate dihydrate

Timothy J. McNeese[a]* and Robert D. Pike[b]

[a]Department of Chemistry, Loyola University Maryland, 4501 North Charles Street, Baltimore, MD 21210-2699, USA, and [b]Department of Chemistry, College of William and Mary, PO Box 8795, Williamsburg, VA 23187-8795, USA
Correspondence e-mail: tmcneese@loyola.edu

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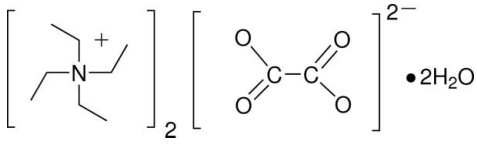
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.081; data-to-parameter ratio = 7.9.

The title compound, $2C_8H_{20}N^+\cdot C_2O_4^{2-}\cdot 2H_2O$, synthesized by neutralizing $H_2C_2O_4\cdot 2H_2O$ with $(C_2H_5)_4NOH$ in a 1:2 molar ratio, is a deliquescent solid. The oxalate ion is nonplanar, with a dihedral angle between carboxylate groups of 64.37 (2)°. O—H···O hydrogen bonds of moderate strength link the O atoms of the water molecules and the oxalate ions into rings parallel to the c axis. The rings exhibit the graph-set motif $R_4^4(12)$. In addition, there are weak C—H···O interactions in the crystal structure.

Related literature

For related compounds containing planar and nonplanar oxalate ions, see: Beagley & Small (1964); Robertson (1965); Jeffrey & Parry (1954). For general syntheses of tetraalkylammonium salts and their uses, see: Barthel & Kunz (1988); Heck (1982); Markowitz (1957); McNeese *et al.* (1984); Starks (1971). For uses of $[(C_2H_5)_4N)_2(C_2O_4)]\cdot 2H_2O$, see: Darensbourg *et al.* (1992); Demadis & Coucouvanis (1995); Diop *et al.* (1997); Engels *et al.* (1983). For classification of the graph-set motifs, see: Etter *et al.* (1990). For classification of the hydrogen bonds, see: Gilli & Gilli (2009). Oxalate was confirmed by the blue ring resorcinol test (Chernoff, 1920).



Experimental

Crystal data

$2C_8H_{20}N^+\cdot C_2O_4^{2-}\cdot 2H_2O$
M_r = 384.55
Orthorhombic, $Pca2_1$
a = 19.9302 (4) Å
b = 7.6627 (1) Å
c = 14.3253 (3) Å
V = 2187.75 (7) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.70 mm^{-1}
T = 100 K
0.32 × 0.25 × 0.05 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.808, T_{max} = 0.966
17374 measured reflections
2026 independent reflections
1953 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 1.07
2026 reflections
256 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H1W···O1^{i}	0.82 (4)	1.96 (4)	2.749 (2)	161 (3)
O6—H2W···O3^{ii}	0.87 (4)	2.00 (4)	2.842 (2)	162 (3)
O5—H3W···O2^{iii}	0.86 (4)	1.88 (4)	2.720 (2)	166 (4)
O5—H4W···O4^{iv}	0.85 (4)	1.89 (4)	2.732 (2)	167 (3)
C3—H3B···O2^{i}	0.99	2.40	3.300 (3)	151
C5—H5A···O1	0.99	2.43	3.324 (3)	149
C5—H5B···O4^{iii}	0.99	2.34	3.270 (3)	157
C7—H7A···O3^{ii}	0.99	2.44	3.332 (3)	150
C8—H8B···O4^{iii}	0.98	2.57	3.497 (3)	158
C10—H10A···O2^{i}	0.98	2.50	3.450 (3)	162
C11—H11B···O5^{v}	0.99	2.41	3.384 (3)	167
C13—H13A···O1^{vi}	0.99	2.37	3.334 (3)	165
C15—H15B···O4^{vi}	0.99	2.51	3.166 (3)	124
C16—H16C···O6^{vii}	0.98	2.59	3.559 (3)	170
C18—H18A···O3^{i}	0.98	2.59	3.296 (2)	129

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXLE* (Hübschle *et al.*, 2011); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2256).

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S1. Comment

Tetraalkylammonium salts are important compounds as phase-transfer catalysts (Starks, 1971), as electrolytes in electrochemical studies (Barthel & Kunz, 1988), in organic Heck-type synthetic reactions (Heck, 1982), and in preparing and isolating polynuclear organometallic complexes (McNeese *et al.*, 1984). A procedure for synthesizing quaternary ammonium salts involves neutralization of a tetraalkylammonium hydroxide with the acid of the desired anion (Markowitz, 1957).

The title compound, $(Et_4N)_2C_2O_4.2H_2O$, synthesized by reaction of Et_4NOH and $H_2C_2O_4.2H_2O$ in a 2:1 mole ratio, is deliquescent and rapidly absorbs moisture from air. Previously prepared and used *in situ* or as a noncrystalline solid, it has been employed as a reductant in aprotic electrochemical cells (Engels *et al.*, 1983) and in the synthesis of oxalate-containing organometallic complexes (Diop *et al.*, 1997; Demadis & Coucouvanis, 1995; Darensbourg *et al.*, 1992).

Oxalate bond distances (C—C and C—O) and angles (O—C—O and O—C—C) are comparable to other reported oxalate salts containing NH_4^+ and alkali metal ions. The oxalate ion is planar in $Li_2C_2O_4$ (Beagley & Small, 1964) and $Na_2C_2O_4$ (Jeffrey & Parry, 1954), but the dihedral angle between planes of symmetrical carboxylate groups of the oxalate fragment is 26.6° in $(NH_4)_2C_2O_4.H_2O$ (Robertson, 1965). The directional character of the hydrogen bonding pattern in the monohydrate is believed responsible for the observed non-planar stereochemistry of the oxalate ion. A dihedral angle of 64.37 (2)° is present in the title compound (Fig. 1). The non-planarity of the oxalate ion is maintained by moderate hydrogen bonds (Table 1) that link the oxygen atoms of the oxalate ion and the water molecules into a ring motif $R^4_4(12)$ (Etter *et al.*, 1990). (For the classification of the hydrogen bonds, see Gilli & Gilli, 2009). In addition, there are also present weak C-H···O interactions in the structure (Tab. 1). Fig. 2 illustrates the packing diagram for the structure of the title compound.

S2. Experimental

An aqueous 35 weight percent solution of Et_4NOH (6.69 g, 15.9 mmol OH^-) was added by syringe to a 50-ml Schlenk tube containing a 20-ml CH_3CN solution of $H_2C_2O_4.2H_2O$ (1.00 g, 7.93 mmol). The colorless solution was stirred under argon for 15 min followed by solvent removal under reduced pressure. Hot tetrahydrofuran (25 ml) was added to the greasy solid, the mixture was stirred for 10 min, and the solution was decanted from the product. The deliquescent white solid was dried under vacuum and crystallized from CH_3CN/ THF. Yield: 1.98 g (65%). IR (υ(CO), CH_3CN) 1565(*s*) cm^{-1}. Oxalate was confirmed by the blue ring resorcinol test (Chernoff, 1920). The analyzed crystal was rapidly transferred from vacuum to a 100 K stream of dry air for X-ray analysis.

S3. Refinement

Data were refined against F^2. Because of the relatively high R_{int} the determination of the absolute structure turned out to be meaningless and therefore 1509 Friedel pairs have been merged. All the hydrogen atoms appeared in the difference

electron density map, nevertheless, those pertinent to the methyl and the methylene carbons were situated into the idealized positions and refined in the riding atom formalism. The positional parameters of the water hydrogens were refined freely. The applied constraints: C_{methyl}-H_{methyl}=0.98, $C_{methylene}$-$H_{methylene}$=0.99 Å. $U_{iso}(H_{methylene}) = 1.2U_{eq}(C_{methylene})$, $U_{iso}(H_{methyl}) = 1.5U_{eq}(C_{methyl})$, $U_{iso}(H_{water}) = 1.5U_{eq}(O_{water})$.

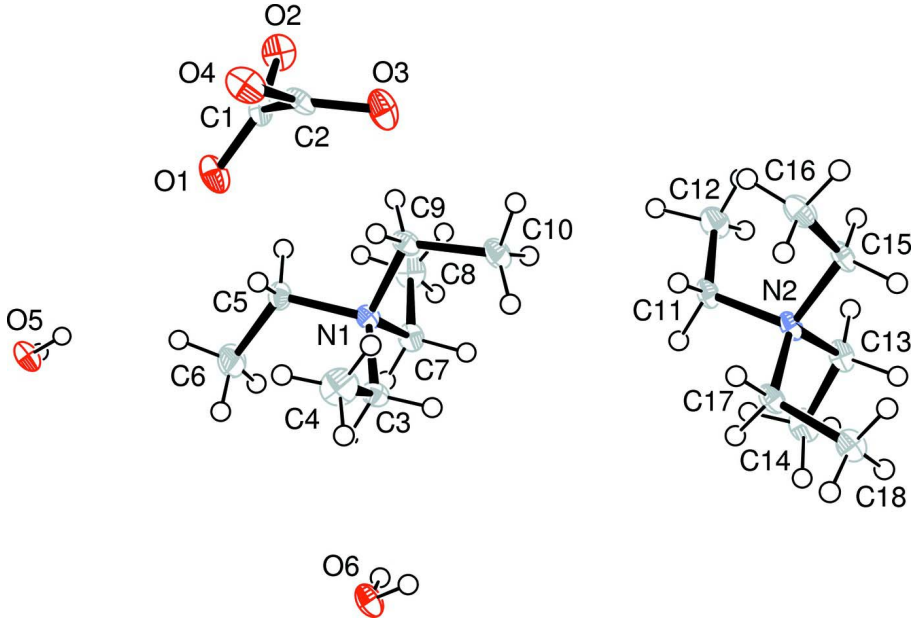

Figure 1

Ellipsoid plot of the title molecules. Displacement ellipsoids are drawn at the 50% probability level.

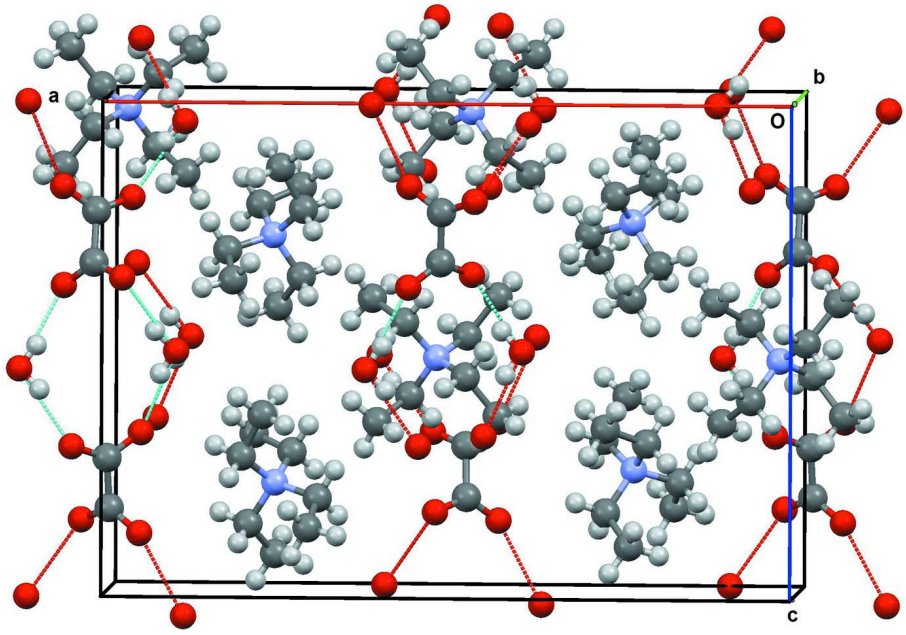

Figure 2

Mercury (Macrae *et al.*, 2006) packing diagram of the title compound viewed down the *b* axis.

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Crystal data

$2C_8H_{20}N^+·C_2O_4^{2-}·2H_2O$
M_r = 384.55
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
a = 19.9302 (4) Å
b = 7.6627 (1) Å
c = 14.3253 (3) Å
V = 2187.75 (7) Å^3
Z = 4
$F(000)$ = 856
D_x = 1.168 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 6094 reflections
θ = 3.8–68.4°
μ = 0.70 mm^{-1}
T = 100 K
Plate, colorless
0.32 × 0.25 × 0.05 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and ψ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.808, T_{max} = 0.966
17374 measured reflections
2026 independent reflections
1953 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 67.0°, θ_{min} = 4.4°
h = −23→23
k = −8→9
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 1.07
2026 reflections
256 parameters
1 restraint
152 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.3359P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXLE* (Hübschle *et al.*, 2011), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0018 (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	0.44416 (8)	0.9370 (2)	0.66940 (11)	0.0260 (4)
O2	0.54192 (8)	1.0741 (2)	0.69474 (11)	0.0245 (4)
O3	0.53292 (7)	0.8233 (2)	0.84977 (11)	0.0263 (4)

O4	0.44453 (8)	0.9997 (2)	0.87126 (11)	0.0254 (4)
C1	0.49202 (10)	0.9852 (3)	0.71976 (14)	0.0162 (4)
C2	0.48956 (10)	0.9310 (3)	0.82269 (14)	0.0167 (4)
N1	0.52560 (8)	0.5362 (2)	0.52871 (12)	0.0155 (4)
C3	0.48056 (10)	0.3892 (3)	0.56294 (15)	0.0191 (4)
H3A	0.4527	0.3484	0.5100	0.023*
H3B	0.5092	0.2903	0.5826	0.023*
C4	0.43481 (12)	0.4368 (3)	0.64282 (19)	0.0321 (6)
H4A	0.4618	0.4682	0.6975	0.048*
H4B	0.4061	0.3369	0.6581	0.048*
H4C	0.4067	0.5363	0.6248	0.048*
C5	0.48413 (10)	0.6851 (3)	0.48890 (15)	0.0194 (4)
H5A	0.4561	0.7342	0.5396	0.023*
H5B	0.5151	0.7782	0.4678	0.023*
C6	0.43882 (13)	0.6362 (3)	0.40826 (19)	0.0331 (6)
H6A	0.4200	0.7423	0.3804	0.050*
H6B	0.4023	0.5620	0.4310	0.050*
H6C	0.4648	0.5726	0.3612	0.050*
C7	0.57042 (10)	0.4560 (3)	0.45509 (15)	0.0191 (4)
H7A	0.5418	0.4061	0.4053	0.023*
H7B	0.5958	0.3587	0.4837	0.023*
C8	0.61999 (11)	0.5824 (3)	0.41095 (17)	0.0267 (5)
H8A	0.6519	0.6226	0.4584	0.040*
H8B	0.5956	0.6828	0.3854	0.040*
H8C	0.6444	0.5233	0.3606	0.040*
C9	0.56591 (10)	0.6129 (3)	0.60804 (14)	0.0180 (4)
H9A	0.5347	0.6644	0.6542	0.022*
H9B	0.5943	0.7084	0.5833	0.022*
C10	0.61047 (11)	0.4815 (3)	0.65726 (16)	0.0231 (5)
H10A	0.5836	0.3801	0.6759	0.035*
H10B	0.6303	0.5355	0.7128	0.035*
H10C	0.6463	0.4440	0.6149	0.035*
N2	0.74829 (8)	0.0031 (2)	0.76685 (12)	0.0164 (4)
C11	0.72670 (10)	0.1585 (3)	0.70941 (15)	0.0189 (4)
H11A	0.6954	0.2301	0.7470	0.023*
H11B	0.7019	0.1160	0.6540	0.023*
C12	0.78410 (11)	0.2737 (3)	0.67665 (17)	0.0257 (5)
H12A	0.8076	0.3218	0.7310	0.039*
H12B	0.7663	0.3694	0.6386	0.039*
H12C	0.8154	0.2043	0.6392	0.039*
C13	0.79440 (10)	−0.1148 (3)	0.71088 (15)	0.0195 (4)
H13A	0.8343	−0.0467	0.6918	0.023*
H13B	0.8100	−0.2106	0.7518	0.023*
C14	0.76283 (11)	−0.1938 (3)	0.62449 (16)	0.0243 (5)
H14A	0.7287	−0.2792	0.6430	0.036*
H14B	0.7975	−0.2519	0.5873	0.036*
H14C	0.7418	−0.1014	0.5873	0.036*
C15	0.78792 (10)	0.0602 (3)	0.85239 (15)	0.0204 (4)

H15A	0.8001	−0.0449	0.8888	0.024*
H15B	0.8302	0.1154	0.8312	0.024*
C16	0.75169 (11)	0.1864 (3)	0.91622 (15)	0.0247 (5)
H16A	0.7430	0.2955	0.8827	0.037*
H16B	0.7796	0.2103	0.9711	0.037*
H16C	0.7090	0.1349	0.9362	0.037*
C17	0.68437 (10)	−0.0925 (3)	0.79498 (14)	0.0177 (4)
H17A	0.6562	−0.0122	0.8324	0.021*
H17B	0.6590	−0.1220	0.7377	0.021*
C18	0.69534 (10)	−0.2576 (3)	0.85037 (15)	0.0213 (4)
H18A	0.6519	−0.3112	0.8646	0.032*
H18B	0.7187	−0.2296	0.9087	0.032*
H18C	0.7226	−0.3391	0.8138	0.032*
O5	0.38115 (7)	0.9648 (2)	0.03930 (11)	0.0216 (3)
H1W	0.4148 (16)	0.023 (4)	0.547 (3)	0.032*
H2W	0.4216 (17)	0.071 (4)	0.461 (3)	0.032*
O6	0.39111 (7)	0.0424 (2)	0.50150 (12)	0.0235 (3)
H3W	0.4112 (17)	0.951 (4)	0.082 (3)	0.035*
H4W	0.4061 (16)	0.968 (4)	−0.009 (3)	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0228 (8)	0.0430 (9)	0.0122 (8)	0.0059 (6)	−0.0052 (6)	−0.0044 (7)
O2	0.0276 (8)	0.0317 (8)	0.0143 (8)	−0.0002 (6)	0.0056 (6)	0.0056 (6)
O3	0.0217 (7)	0.0387 (9)	0.0185 (8)	0.0006 (6)	−0.0022 (6)	0.0122 (7)
O4	0.0247 (8)	0.0400 (9)	0.0114 (7)	−0.0035 (7)	0.0055 (6)	−0.0047 (6)
C1	0.0175 (10)	0.0226 (10)	0.0085 (9)	0.0064 (7)	0.0016 (8)	−0.0021 (8)
C2	0.0128 (9)	0.0265 (10)	0.0109 (10)	−0.0050 (7)	−0.0016 (8)	0.0008 (8)
N1	0.0153 (8)	0.0194 (8)	0.0120 (8)	−0.0007 (7)	−0.0004 (7)	−0.0002 (6)
C3	0.0176 (9)	0.0211 (10)	0.0187 (10)	−0.0045 (8)	−0.0009 (8)	0.0010 (8)
C4	0.0275 (12)	0.0354 (13)	0.0334 (14)	−0.0027 (10)	0.0150 (11)	0.0028 (10)
C5	0.0199 (9)	0.0203 (9)	0.0181 (10)	0.0036 (7)	−0.0028 (8)	0.0008 (8)
C6	0.0365 (13)	0.0322 (12)	0.0306 (13)	0.0062 (10)	−0.0179 (11)	−0.0034 (10)
C7	0.0188 (10)	0.0249 (10)	0.0134 (10)	0.0039 (8)	0.0026 (8)	−0.0021 (8)
C8	0.0243 (11)	0.0342 (11)	0.0216 (11)	0.0024 (9)	0.0090 (9)	0.0016 (9)
C9	0.0172 (9)	0.0242 (10)	0.0125 (10)	−0.0029 (8)	−0.0015 (8)	−0.0014 (8)
C10	0.0226 (11)	0.0291 (11)	0.0176 (11)	−0.0012 (8)	−0.0068 (9)	0.0012 (9)
N2	0.0121 (7)	0.0252 (9)	0.0119 (8)	−0.0003 (7)	−0.0011 (7)	0.0019 (7)
C11	0.0161 (9)	0.0250 (10)	0.0156 (9)	0.0006 (8)	−0.0026 (8)	0.0053 (9)
C12	0.0217 (10)	0.0316 (11)	0.0239 (11)	−0.0041 (9)	−0.0012 (9)	0.0084 (9)
C13	0.0141 (9)	0.0296 (10)	0.0149 (10)	0.0031 (8)	0.0032 (8)	0.0019 (9)
C14	0.0239 (11)	0.0331 (11)	0.0159 (10)	0.0019 (8)	0.0035 (8)	−0.0017 (9)
C15	0.0171 (9)	0.0300 (11)	0.0140 (10)	0.0002 (8)	−0.0055 (8)	0.0014 (8)
C16	0.0249 (10)	0.0346 (11)	0.0146 (10)	−0.0017 (9)	−0.0041 (8)	−0.0032 (9)
C17	0.0120 (9)	0.0279 (11)	0.0132 (10)	−0.0013 (8)	0.0007 (7)	0.0008 (8)
C18	0.0216 (10)	0.0296 (11)	0.0128 (10)	−0.0006 (8)	0.0014 (8)	0.0014 (8)
O5	0.0169 (7)	0.0371 (8)	0.0109 (7)	−0.0003 (6)	0.0004 (7)	0.0001 (6)

O6	0.0195 (8)	0.0351 (8)	0.0159 (8)	0.0004 (6)	−0.0043 (7)	0.0009 (7)

Geometric parameters (Å, º)

O1—C1	1.252 (3)	N2—C11	1.510 (3)
O2—C1	1.258 (3)	N2—C13	1.518 (3)
O3—C2	1.256 (3)	N2—C15	1.522 (3)
O4—C2	1.252 (3)	N2—C17	1.524 (2)
C1—C2	1.533 (3)	C11—C12	1.520 (3)
N1—C9	1.511 (2)	C11—H11A	0.9900
N1—C7	1.513 (2)	C11—H11B	0.9900
N1—C5	1.520 (3)	C12—H12A	0.9800
N1—C3	1.522 (3)	C12—H12B	0.9800
C3—C4	1.508 (3)	C12—H12C	0.9800
C3—H3A	0.9900	C13—C14	1.514 (3)
C3—H3B	0.9900	C13—H13A	0.9900
C4—H4A	0.9800	C13—H13B	0.9900
C4—H4B	0.9800	C14—H14A	0.9800
C4—H4C	0.9800	C14—H14B	0.9800
C5—C6	1.514 (3)	C14—H14C	0.9800
C5—H5A	0.9900	C15—C16	1.514 (3)
C5—H5B	0.9900	C15—H15A	0.9900
C6—H6A	0.9800	C15—H15B	0.9900
C6—H6B	0.9800	C16—H16A	0.9800
C6—H6C	0.9800	C16—H16B	0.9800
C7—C8	1.521 (3)	C16—H16C	0.9800
C7—H7A	0.9900	C17—C18	1.509 (3)
C7—H7B	0.9900	C17—H17A	0.9900
C8—H8A	0.9800	C17—H17B	0.9900
C8—H8B	0.9800	C18—H18A	0.9800
C8—H8C	0.9800	C18—H18B	0.9800
C9—C10	1.517 (3)	C18—H18C	0.9800
C9—H9A	0.9900	O5—H3W	0.87 (4)
C9—H9B	0.9900	O5—H4W	0.86 (4)
C10—H10A	0.9800	O6—H1W	0.82 (4)
C10—H10B	0.9800	O6—H2W	0.87 (4)
C10—H10C	0.9800		

O1—C1—O2	126.7 (2)	H10A—C10—H10C	109.5
O1—C1—C2	116.77 (19)	H10B—C10—H10C	109.5
O2—C1—C2	116.50 (18)	C11—N2—C13	110.73 (16)
O4—C2—O3	126.7 (2)	C11—N2—C15	111.11 (16)
O4—C2—C1	116.34 (18)	C13—N2—C15	106.39 (15)
O3—C2—C1	116.95 (18)	C11—N2—C17	106.56 (15)
C9—N1—C7	111.62 (15)	C13—N2—C17	111.08 (16)
C9—N1—C5	106.21 (16)	C15—N2—C17	111.05 (16)
C7—N1—C5	111.38 (16)	N2—C11—C12	114.34 (16)
C9—N1—C3	111.07 (16)	N2—C11—H11A	108.7

C7—N1—C3	105.78 (16)	C12—C11—H11A	108.7
C5—N1—C3	110.86 (15)	N2—C11—H11B	108.7
C4—C3—N1	114.95 (18)	C12—C11—H11B	108.7
C4—C3—H3A	108.5	H11A—C11—H11B	107.6
N1—C3—H3A	108.5	C11—C12—H12A	109.5
C4—C3—H3B	108.5	C11—C12—H12B	109.5
N1—C3—H3B	108.5	H12A—C12—H12B	109.5
H3A—C3—H3B	107.5	C11—C12—H12C	109.5
C3—C4—H4A	109.5	H12A—C12—H12C	109.5
C3—C4—H4B	109.5	H12B—C12—H12C	109.5
H4A—C4—H4B	109.5	C14—C13—N2	114.72 (16)
C3—C4—H4C	109.5	C14—C13—H13A	108.6
H4A—C4—H4C	109.5	N2—C13—H13A	108.6
H4B—C4—H4C	109.5	C14—C13—H13B	108.6
C6—C5—N1	115.14 (17)	N2—C13—H13B	108.6
C6—C5—H5A	108.5	H13A—C13—H13B	107.6
N1—C5—H5A	108.5	C13—C14—H14A	109.5
C6—C5—H5B	108.5	C13—C14—H14B	109.5
N1—C5—H5B	108.5	H14A—C14—H14B	109.5
H5A—C5—H5B	107.5	C13—C14—H14C	109.5
C5—C6—H6A	109.5	H14A—C14—H14C	109.5
C5—C6—H6B	109.5	H14B—C14—H14C	109.5
H6A—C6—H6B	109.5	C16—C15—N2	114.97 (16)
C5—C6—H6C	109.5	C16—C15—H15A	108.5
H6A—C6—H6C	109.5	N2—C15—H15A	108.5
H6B—C6—H6C	109.5	C16—C15—H15B	108.5
N1—C7—C8	114.48 (17)	N2—C15—H15B	108.5
N1—C7—H7A	108.6	H15A—C15—H15B	107.5
C8—C7—H7A	108.6	C15—C16—H16A	109.5
N1—C7—H7B	108.6	C15—C16—H16B	109.5
C8—C7—H7B	108.6	H16A—C16—H16B	109.5
H7A—C7—H7B	107.6	C15—C16—H16C	109.5
C7—C8—H8A	109.5	H16A—C16—H16C	109.5
C7—C8—H8B	109.5	H16B—C16—H16C	109.5
H8A—C8—H8B	109.5	C18—C17—N2	114.90 (16)
C7—C8—H8C	109.5	C18—C17—H17A	108.5
H8A—C8—H8C	109.5	N2—C17—H17A	108.5
H8B—C8—H8C	109.5	C18—C17—H17B	108.5
N1—C9—C10	113.75 (17)	N2—C17—H17B	108.5
N1—C9—H9A	108.8	H17A—C17—H17B	107.5
C10—C9—H9A	108.8	C17—C18—H18A	109.5
N1—C9—H9B	108.8	C17—C18—H18B	109.5
C10—C9—H9B	108.8	H18A—C18—H18B	109.5
H9A—C9—H9B	107.7	C17—C18—H18C	109.5
C9—C10—H10A	109.5	H18A—C18—H18C	109.5
C9—C10—H10B	109.5	H18B—C18—H18C	109.5
H10A—C10—H10B	109.5	H3W—O5—H4W	100 (3)
C9—C10—H10C	109.5	H1W—O6—H2W	100 (3)

O1—C1—C2—O4	−67.8 (3)	C5—N1—C9—C10	179.55 (17)
O2—C1—C2—O4	111.9 (2)	C3—N1—C9—C10	58.9 (2)
O1—C1—C2—O3	112.5 (2)	C13—N2—C11—C12	−60.6 (2)
O2—C1—C2—O3	−67.8 (3)	C15—N2—C11—C12	57.4 (2)
C9—N1—C3—C4	54.2 (2)	C17—N2—C11—C12	178.52 (18)
C7—N1—C3—C4	175.46 (18)	C11—N2—C13—C14	−62.2 (2)
C5—N1—C3—C4	−63.7 (2)	C15—N2—C13—C14	176.99 (17)
C9—N1—C5—C6	−179.36 (19)	C17—N2—C13—C14	56.0 (2)
C7—N1—C5—C6	58.9 (2)	C11—N2—C15—C16	56.9 (2)
C3—N1—C5—C6	−58.6 (2)	C13—N2—C15—C16	177.47 (17)
C9—N1—C7—C8	−59.2 (2)	C17—N2—C15—C16	−61.6 (2)
C5—N1—C7—C8	59.3 (2)	C11—N2—C17—C18	177.74 (17)
C3—N1—C7—C8	179.88 (18)	C13—N2—C17—C18	57.1 (2)
C7—N1—C9—C10	−58.9 (2)	C15—N2—C17—C18	−61.1 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H1*W*···O1^{i}	0.82 (4)	1.96 (4)	2.749 (2)	161 (3)
O6—H2*W*···O3^{ii}	0.87 (4)	2.00 (4)	2.842 (2)	162 (3)
O5—H3*W*···O2^{iii}	0.86 (4)	1.88 (4)	2.720 (2)	166 (4)
O5—H4*W*···O4^{iv}	0.85 (4)	1.89 (4)	2.732 (2)	167 (3)
C3—H3*B*···O2^{i}	0.99	2.40	3.300 (3)	151
C5—H5*A*···O1	0.99	2.43	3.324 (3)	149
C5—H5*B*···O4^{iii}	0.99	2.34	3.270 (3)	157
C7—H7*A*···O3^{ii}	0.99	2.44	3.332 (3)	150
C8—H8*B*···O4^{iii}	0.98	2.57	3.497 (3)	158
C10—H10*A*···O2^{i}	0.98	2.50	3.450 (3)	162
C11—H11*B*···O5^{v}	0.99	2.41	3.384 (3)	167
C13—H13*A*···O1^{vi}	0.99	2.37	3.334 (3)	165
C15—H15*B*···O4^{vi}	0.99	2.51	3.166 (3)	124
C16—H16*C*···O6^{vii}	0.98	2.59	3.559 (3)	170
C18—H18*A*···O3^{i}	0.98	2.59	3.296 (2)	129

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