

Crystal structure of dimethylammonium hydrogen oxalate hemi(oxalic acid)

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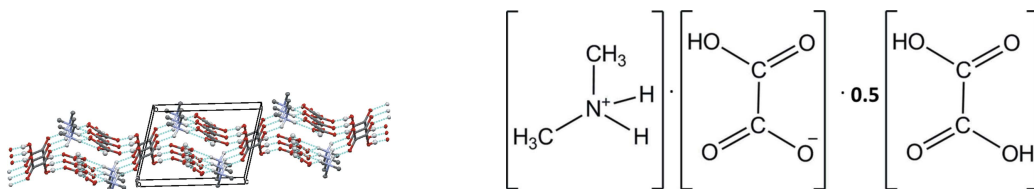
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Single crystals of the title salt, $\text{Me}_2\text{NH}_2^+\cdot\text{HC}_2\text{O}_4^-\cdot 0.5\text{H}_2\text{C}_2\text{O}_4$, were isolated as a side product from the reaction involving Me_2NH , $\text{H}_2\text{C}_2\text{O}_4$ and $\text{Sn}(n\text{-Bu})_3\text{Cl}$ in a 1:2 ratio in methanol or by the reaction of the $(\text{Me}_2\text{NH}_2)_2\text{C}_2\text{O}_4$ salt and $\text{Sn}(\text{CH}_3)_3\text{Cl}$ in a 2:1 ratio in ethanol. The asymmetric unit comprises a dimethylammonium cation (Me_2NH_2^+), an hydrogenoxalate anion (HC_2O_4^-), and half a molecule of oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) situated about an inversion center. From a supramolecular point of view, the three components interact together *via* hydrogen bonding. The Me_2NH_2^+ cations and the HC_2O_4^- anions are in close proximity through bifurcated $\text{N}-\text{H}\cdots(\text{O},\text{O})$ hydrogen bonds, while the HC_2O_4^- anions are organized into infinite chains *via* $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, propagating along the *a*-axis direction. In addition, the oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) molecules play the role of connectors between these chains. Both the carbonyl and hydroxyl groups of each diacid are involved in four intermolecular interactions with two Me_2NH_2^+ and two HC_2O_4^- ions of four distinct polymeric chains, *via* two $\text{N}-\text{H}\cdots\text{O}$ and two $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, respectively. The resulting molecular assembly can be viewed as a two-dimensional bilayer-like arrangement lying parallel to (010), and reinforced by a $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond.

1. Chemical context

Within the scope of our research on the crystal structure determination of new organotin compounds containing dialkylammonium, we recently reported the structures of bis(dimethylammonium) tetrachloridodimethylstannate(IV) [Diop *et al.*, 2011] and dimethylammonium dichloridotriphenylstannate(IV) [Sow *et al.*, 2012]. Continuing our quest in this field, we report herein on the crystal structure of the title salt, $\text{Me}_2\text{NH}_2^+\cdot\text{HC}_2\text{O}_4^-\cdot 0.5\text{H}_2\text{C}_2\text{O}_4$, isolated from two distinct reaction pathways, *viz.* mixing Me_2NH , $\text{H}_2\text{C}_2\text{O}_4$ and SnBu_3Cl in methanol or the reaction of the $(\text{Me}_2\text{NH}_2)_2\text{C}_2\text{O}_4$ salt and $\text{Sn}(\text{CH}_3)_3\text{Cl}$ in ethanol.



The title salt constitutes a new example of dialkylammonium hydrogenoxalates and thus supplements the number of crystal structures resolved to date for this type of salt (Birnbaum, 1972; Thomas & Pramatus, 1975; Thomas, 1977; G undisch *et al.*, 2001; Warden *et al.*, 2005). In addition,

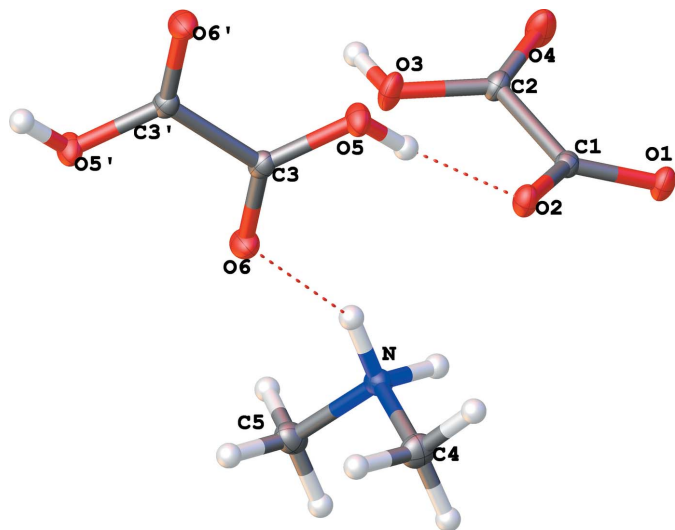


Figure 1
A view of the molecular structure of the title salt, with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

and because of their capacity to easily develop hydrogen-bonding networks, carboxylic acids and their derivatives are of great interest in the field of crystal engineering, leading to a large diversity of supramolecular topologies (Ivasenko & Perepichka, 2011).

2. Structural comments

In the asymmetric unit of the title salt there are three components: one dimethylammonium cation (Me_2NH_2^+), one hydrogenoxalate anion (HC_2O_4^-), and half a molecule of oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) which possess inversion symmetry (Fig. 1). All three entities are linked by intermolecular interactions (Table 1 and Fig. 2). The Me_2NH_2^+ cation is in close proximity with the HC_2O_4^- anion through bifurcated $\text{N}-\text{H}\cdots(\text{O},\text{O})$ hydrogen bonds [$\text{N1}-\text{H1A}\cdots\text{O1} = 2.854(1) \text{ \AA}$ and $\text{N1}-\text{H1A}\cdots\text{O4} = 2.964(1) \text{ \AA}$]. The lengths of the $\text{N}-\text{C}$ bonds [$\text{N1}-\text{C4} = 1.4822(12)$ and $\text{N1}-\text{C5} = 1.4842(12) \text{ \AA}$] are nearly identical of those reported previously for $\text{Me}_2\text{NH}_2^+\cdot\text{HC}_2\text{O}_4^-$ (Thomas, 1977). The Me_2NH_2^+ cation is also involved in hydrogen bonding with one of the two carbonyl groups of the oxalic acid molecule [$\text{N1}-\text{H1B}\cdots\text{O6} = 2.846(1) \text{ \AA}$]. The HC_2O_4^- hydrogenoxalate anions form a

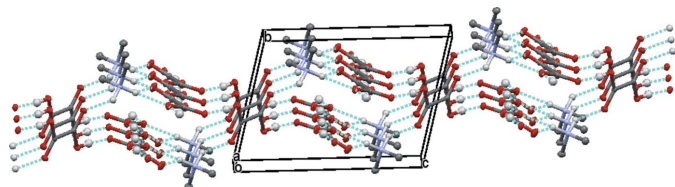


Figure 2
Crystal packing of the title salt, viewed along the *a* axis, showing the two-dimensional bilayer-like arrangement formed through $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (dashed lines; details are given in Table 1). H atoms not involved in hydrogen bonding have been omitted for clarity.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
$\text{O3}-\text{H3}\cdots\text{O1}^{\text{i}}$	0.84	1.73	2.564 (1)	174
$\text{O5}-\text{H5}\cdots\text{O2}$	0.84	1.73	2.565 (1)	170
$\text{N1}-\text{H1A}\cdots\text{O1}^{\text{ii}}$	0.91	2.08	2.854 (1)	143
$\text{N1}-\text{H1A}\cdots\text{O4}^{\text{ii}}$	0.91	2.23	2.964 (1)	137
$\text{N1}-\text{H1B}\cdots\text{O6}$	0.91	2.05	2.846 (1)	146
$\text{C5}-\text{H5C}\cdots\text{O4}^{\text{iii}}$	0.98	2.41	3.346 (1)	159

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

one-dimensional chain along the *a*-axis direction via the formation of $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds [$\text{O3}-\text{H3}\cdots\text{O1} = 2.564(1) \text{ \AA}$]. Furthermore, the HC_2O_4^- anion is also involved in hydrogen bonding with one of the two hydroxyl groups of the oxalic acid molecule [$\text{O5}-\text{H5}\cdots\text{O2} = 2.565(1) \text{ \AA}$].

3. Supramolecular features

From a supramolecular point of view, the combination of these intermolecular interactions leads to the formation of a molecular assembly which can be described as a two-dimensional bilayer-like arrangement, parallel to (010), consisting of anti-parallel infinite chains of $\text{Me}_2\text{NH}_2^+\cdot\text{HC}_2\text{O}_4^-$ (Table 1 and Fig. 3), with an inter-chain distance of *ca* 3.0 \AA . The oxalic acid molecules are organized in a parallel offset fashion, and act as hydrogen-bond connectors between the chains, involving both the carbonyl and hydroxyl groups (Table 1 and Figs. 2 and 3).

4. Database survey

The crystal structure of $\text{Me}_2\text{NH}_2^+\cdot\text{HC}_2\text{O}_4^-$, first reported by Thomas & Pramatus (1975) and then completed in 1977

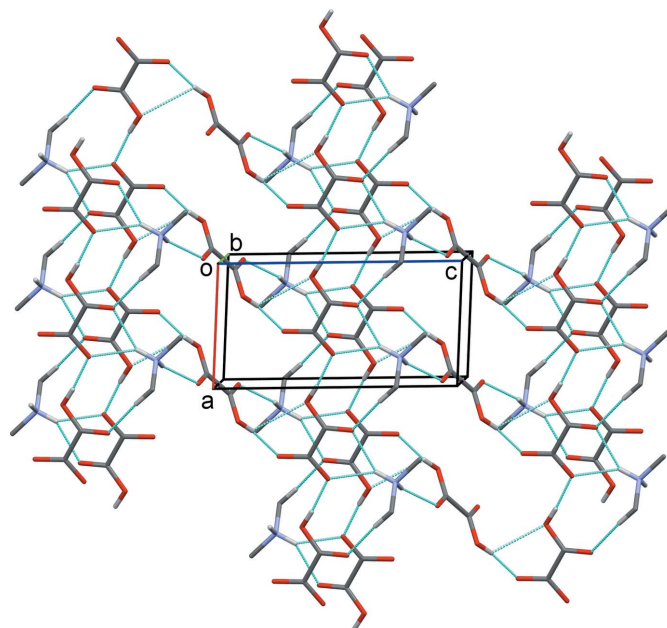


Figure 3
Crystal packing of the title salt viewed along the *b* axis. The hydrogen bonds are shown as dashed lines (see Table 1 for details) and H atoms not involved in hydrogen bonding have been omitted for clarity.

(Thomas, 1977), shows a supramolecular structure qualified as a puckered layer. In particular, the HC_2O_4^- ions are linked *via* $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds [2.533 (1) Å], leading to an infinite chain along [100]. In the title salt, the HC_2O_4^- ions interact in the same manner but through slightly longer $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds [2.564 (1) Å]. In addition, the oxalic acid molecules that co-crystallize with $\text{Me}_2\text{NH}_2^+\cdot\text{HC}_2\text{O}_4^-$ act both as donors and acceptors of hydrogen bonds through $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ bonds with the Me_2NH_2^+ cation and HC_2O_4^- anion, respectively. Consequently, the degree of supramolecularity is increased here, resulting in a two-dimensional architecture parallel to (010), which is reinforced by a $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond (Table 1 and Figs. 2 and 3).

5. Synthesis and crystallization

Crystals of the title compound were obtained by mixing in 20 ml methanol (98% purity) Me_2NH (0.30 g, 6.67 mmol), $\text{H}_2\text{C}_2\text{O}_4$ (0.60 g, 6.67 mmol) and $\text{Sn}(n\text{-Bu})_3\text{Cl}$ (4.39 g, 13.33 mmol). Another experimental method is the reaction between the $(\text{Me}_2\text{NH}_2)_2\text{C}_2\text{O}_4$ salt (0.50 g, 2.77 mmol), previously synthesized from oxalic acid and dimethylamine, and $\text{Sn}(\text{CH}_3)_3\text{Cl}$ (0.28 g, 1.39 mmol) in 15 ml of ethanol (98% purity). In both cases, the reaction mixture was stirred for *ca* 2 h at room temperature. Colourless crystals were obtained after one week by slow evaporation of the solvent.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All the H atoms were placed in calculated positions and refined as riding: $\text{O}-\text{H} = 0.84$ Å, $\text{N}-\text{H} = 0.91$ Å, and $\text{C}-\text{H} = 0.98$ Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}, \text{O})$ and $1.2U_{\text{eq}}(\text{N})$.

Acknowledgements

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Table 2
Experimental details.

Crystal data	
Chemical formula	$\text{C}_2\text{H}_8\text{N}^+\cdot\text{C}_2\text{HO}_4^-\cdot 0.5\text{C}_2\text{H}_2\text{O}_4$
M_r	180.14
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
a, b, c (Å)	5.6519 (3), 7.5809 (4), 10.3100 (6)
α, β, γ (°)	75.467 (2), 88.120 (2), 69.487 (2)
V (Å ³)	399.76 (4)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.14
Crystal size (mm)	0.5 × 0.3 × 0.1
Data collection	
Diffractometer	Bruker D8 Venture triumph Mo
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2014)
$T_{\text{min}}, T_{\text{max}}$	0.693, 0.746
No. of measured, independent and observed [$I \geq 2\sigma(I)$] reflections	10413, 1840, 1655
R_{int}	0.023
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.651
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.028, 0.075, 1.07
No. of reflections	1840
No. of parameters	113
H-atom treatment	H-atom parameters not refined
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.38, -0.26

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SUPERFLIP* (Palatinus & Chapuis, 2007), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008).

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supporting information

Acta Cryst. (2015). E71, 473–475 [https://doi.org/10.1107/S2056989015005964]

Crystal structure of dimethylammonium hydrogen oxalate hemi(oxalic acid)

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Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Olex2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *Olex2* (Dolomanov *et al.*, 2009).

Dimethylammonium hydrogen oxalate hemi(oxalic acid)

Crystal data

$C_2H_8N^+ \cdot C_2HO_4^- \cdot 0.5C_2H_2O_4$

$M_r = 180.14$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.6519$ (3) Å

$b = 7.5809$ (4) Å

$c = 10.3100$ (6) Å

$\alpha = 75.467$ (2)°

$\beta = 88.120$ (2)°

$\gamma = 69.487$ (2)°

$V = 399.76$ (4) Å³

$Z = 2$

$F(000) = 190.1598$

$D_x = 1.497$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7049 reflections

$\theta = 3.0$ – 27.6 °

$\mu = 0.14$ mm⁻¹

$T = 100$ K

Prism, colourless

$0.5 \times 0.3 \times 0.1$ mm

Data collection

Bruker D8 Venture triumph Mo diffractometer

Radiation source: X-ray tube, Siemens KFF Mo 2K-90C

TRIUMPH curved crystal monochromator

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2014)

$T_{\min} = 0.693$, $T_{\max} = 0.746$

10413 measured reflections

1840 independent reflections

1655 reflections with $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 27.6$ °, $\theta_{\min} = 3.0$ °

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.075$

$S = 1.07$

1840 reflections

113 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 0.1325P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.38$ e Å⁻³

$\Delta\rho_{\min} = -0.26$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.75407 (12)	0.32103 (10)	0.47166 (7)	0.01407 (16)
O2	0.57685 (12)	0.40754 (10)	0.26360 (7)	0.01329 (16)
O3	0.14369 (12)	0.38767 (11)	0.36954 (7)	0.01625 (17)
H3	0.0215	0.3632	0.4083	0.024*
O4	0.36051 (14)	0.20987 (11)	0.56626 (7)	0.02010 (17)
O5	0.29036 (13)	0.37192 (10)	0.09144 (7)	0.01471 (16)
H5	0.3683	0.3944	0.1497	0.022*
O6	0.01961 (13)	0.67729 (10)	0.07499 (7)	0.01568 (16)
C1	0.57857 (16)	0.34999 (13)	0.38821 (9)	0.01061 (18)
C2	0.34558 (17)	0.30728 (13)	0.45205 (9)	0.01216 (19)
C3	0.08336 (17)	0.52128 (13)	0.04793 (9)	0.01171 (19)
N1	0.22327 (15)	0.83866 (11)	0.24521 (8)	0.01223 (17)
H1A	0.3034	0.7852	0.3284	0.015*
H1B	0.1697	0.7490	0.2236	0.015*
C4	0.40571 (19)	0.88277 (15)	0.14735 (10)	0.0182 (2)
H4A	0.3224	0.9359	0.0569	0.027*
H4B	0.5500	0.7630	0.1500	0.027*
H4C	0.4653	0.9785	0.1705	0.027*
C5	-0.00007 (19)	1.01362 (14)	0.24892 (11)	0.0177 (2)
H5A	-0.0886	1.0711	0.1598	0.027*
H5B	0.0557	1.1091	0.2751	0.027*
H5C	-0.1149	0.9761	0.3143	0.027*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0100 (3)	0.0212 (4)	0.0119 (3)	-0.0072 (3)	-0.0008 (2)	-0.0031 (3)
O2	0.0113 (3)	0.0178 (3)	0.0107 (3)	-0.0060 (3)	-0.0004 (2)	-0.0024 (3)
O3	0.0089 (3)	0.0274 (4)	0.0131 (3)	-0.0087 (3)	0.0002 (3)	-0.0030 (3)
O4	0.0162 (4)	0.0286 (4)	0.0148 (3)	-0.0123 (3)	-0.0008 (3)	0.0022 (3)
O5	0.0134 (3)	0.0149 (3)	0.0150 (3)	-0.0021 (3)	-0.0044 (3)	-0.0059 (3)
O6	0.0178 (3)	0.0130 (3)	0.0166 (3)	-0.0046 (3)	-0.0040 (3)	-0.0051 (3)
C1	0.0087 (4)	0.0105 (4)	0.0127 (4)	-0.0028 (3)	0.0003 (3)	-0.0039 (3)
C2	0.0103 (4)	0.0154 (4)	0.0126 (4)	-0.0057 (3)	0.0004 (3)	-0.0050 (3)
C3	0.0127 (4)	0.0138 (4)	0.0091 (4)	-0.0059 (3)	-0.0003 (3)	-0.0018 (3)
N1	0.0145 (4)	0.0118 (4)	0.0107 (4)	-0.0050 (3)	-0.0005 (3)	-0.0028 (3)
C4	0.0166 (5)	0.0202 (5)	0.0186 (5)	-0.0074 (4)	0.0050 (4)	-0.0055 (4)
C5	0.0152 (5)	0.0148 (4)	0.0211 (5)	-0.0037 (4)	0.0033 (4)	-0.0034 (4)

Geometric parameters (\AA , $^\circ$)

O1—C1	1.2573 (11)	N1—H1A	0.9100
O2—C1	1.2480 (11)	N1—H1B	0.9100
O3—H3	0.8400	N1—C4	1.4822 (12)
O3—C2	1.3089 (11)	N1—C5	1.4842 (12)
O4—C2	1.2105 (12)	C4—H4A	0.9800
O5—H5	0.8400	C4—H4B	0.9800
O5—C3	1.3051 (11)	C4—H4C	0.9800
O6—C3	1.2111 (12)	C5—H5A	0.9800
C1—C2	1.5515 (13)	C5—H5B	0.9800
C3—C3 ⁱ	1.5501 (17)	C5—H5C	0.9800
C2—O3—H3	109.5	C5—N1—H1A	109.0
C3—O5—H5	109.5	C5—N1—H1B	109.0
O1—C1—C2	114.27 (8)	N1—C4—H4A	109.5
O2—C1—O1	126.60 (8)	N1—C4—H4B	109.5
O2—C1—C2	119.13 (8)	N1—C4—H4C	109.5
O3—C2—C1	112.45 (8)	H4A—C4—H4B	109.5
O4—C2—O3	126.54 (9)	H4A—C4—H4C	109.5
O4—C2—C1	121.01 (8)	H4B—C4—H4C	109.5
O5—C3—C3 ⁱ	111.64 (10)	N1—C5—H5A	109.5
O6—C3—O5	126.87 (8)	N1—C5—H5B	109.5
O6—C3—C3 ⁱ	121.48 (10)	N1—C5—H5C	109.5
H1A—N1—H1B	107.8	H5A—C5—H5B	109.5
C4—N1—H1A	109.0	H5A—C5—H5C	109.5
C4—N1—H1B	109.0	H5B—C5—H5C	109.5
C4—N1—C5	112.87 (8)		
O1—C1—C2—O3	162.31 (8)	O2—C1—C2—O3	-17.79 (12)
O1—C1—C2—O4	-17.45 (13)	O2—C1—C2—O4	162.45 (9)

Symmetry code: (i) $-x, -y+1, -z$.Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 \cdots O1 ⁱⁱ	0.84	1.73	2.564 (1)	174
O5—H5 \cdots O2	0.84	1.73	2.565 (1)	170
N1—H1A \cdots O1 ⁱⁱⁱ	0.91	2.08	2.854 (1)	143
N1—H1A \cdots O4 ⁱⁱⁱ	0.91	2.23	2.964 (1)	137
N1—H1B \cdots O6	0.91	2.05	2.846 (1)	146
C5—H5C \cdots O4 ^{iv}	0.98	2.41	3.346 (1)	159

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