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## Structure Reports

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# Tetrakis(dipropylammonium) tetrakis(oxalato- $\kappa^2O^1, O^2$ )stannate(IV) monohydrate: a complex with an eight-coordinate $\text{Sn}^{\text{IV}}$ atom

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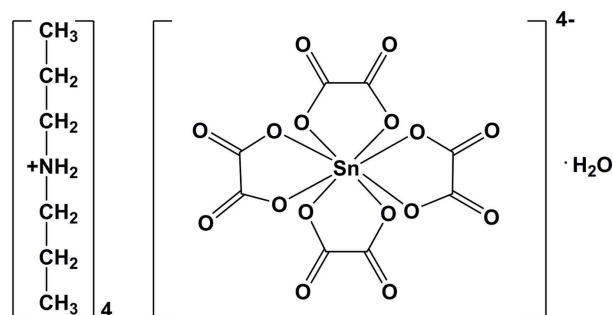
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Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å; disorder in solvent or counterion;  $R$  factor = 0.021;  $wR$  factor = 0.054; data-to-parameter ratio = 12.4.

In the title salt,  $[(\text{CH}_3\text{CH}_2\text{CH}_2)_2\text{NH}_2]_4[\text{Sn}(\text{C}_2\text{O}_4)_4]\cdot\text{H}_2\text{O}$ , the  $\text{Sn}^{\text{IV}}$  atom of the stannate anion is located on a special position with  $\bar{4}2m$  symmetry. It is eight-coordinated by four chelating oxalate anions. The dipropylammonium cation possesses mirror symmetry while the lattice water molecule is disordered about a position with  $\bar{4}2m$  symmetry and has an occupancy of 0.25. In the crystal, the anions and cations are linked by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds, forming a three-dimensional network. This network is further stabilized by weak  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds involving the water molecules and oxalate O atoms. The crystal studied was refined as an inversion twin.

## Related literature

For the chemistry of organotin complexes, see: Evans & Karpel (1985). For examples of zirconate anions with eight-coordinate  $\text{Zr}^{\text{IV}}$  atoms, see: Fu *et al.* (2005); Imaz *et al.* (2007). For an example of a related oxalatostannate(IV) complex, see: Gueye *et al.* (2010).



## Experimental

### Crystal data

$(\text{C}_6\text{H}_{16}\text{N})_4[\text{Sn}(\text{C}_2\text{O}_4)_4]\cdot\text{H}_2\text{O}$   
 $M_r = 897.57$   
 Tetragonal,  $\bar{4}2m$   
 $a = 14.5996$  (6) Å  
 $c = 9.5718$  (4) Å  
 $V = 2040.21$  (19) Å<sup>3</sup>

$Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.70$  mm<sup>-1</sup>  
 $T = 173$  K  
 $0.45 \times 0.30 \times 0.22$  mm

### Data collection

STOE IPDS2 diffractometer  
 Absorption correction: multi-scan  
 (*MULScan* in *PLATON*;  
 Spek, 2009)  
 $T_{\min} = 0.540$ ,  $T_{\max} = 1.000$

9636 measured reflections  
 1030 independent reflections  
 1027 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.071$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.021$   
 $wR(F^2) = 0.054$   
 $S = 1.08$   
 1030 reflections  
 83 parameters  
 3 restraints  
 H atoms treated by a mixture of  
 independent and constrained  
 refinement

$\Delta\rho_{\text{max}} = 0.39$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.62$  e Å<sup>-3</sup>  
 Absolute structure: Refined as an  
 inversion twin  
 Absolute structure parameter:  
 0.45 (4)

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1AN}\cdots\text{O1}^i$	0.88 (3)	2.50 (5)	3.091 (6)	125 (5)
$\text{N1}-\text{H1AN}\cdots\text{O2}^i$	0.88 (3)	2.12 (3)	2.997 (8)	179 (6)
$\text{N1}-\text{H1BN}\cdots\text{O2}$	0.88 (3)	2.02 (4)	2.821 (7)	151 (5)
$\text{N1}-\text{H1BN}\cdots\text{O4}$	0.88 (3)	2.44 (5)	3.105 (6)	133 (5)
$\text{O1W}-\text{H1WA}\cdots\text{O3}^{ii}$	0.85	2.27	3.125 (7)	179

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

Data collection: *X-Area* (Stoe & Cie, 2009); cell refinement: *X-Area* (Stoe & Cie, 2009); data reduction: *X-RED32* (Stoe & Cie, 2009); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL2013* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *pubCIF* (Westrip, 2010).

HSE thanks the XRD Application Laboratory, CSEM, Neuchâtel for access to the X-ray diffraction equipment.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2795).

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## supporting information

*Acta Cryst.* (2014). E70, m49–m50 [doi:10.1107/S160053681303496X]

## Tetrakis(dipropylammonium) tetrakis(oxalato- $\kappa^2O^1, O^2$ )stannate(IV) monohydrate: a complex with an eight-coordinate Sn<sup>IV</sup> atom

Ndongo Gueye, Libasse Diop and Helen Stoeckli-Evans

### S1. Comment

The chemistry and applications of organotin(IV) complexes have been extensively discussed by Evans & Karpel (1985). Continuing our interest in Sn<sup>IV</sup> oxalate complexes (Gueye *et al.*, 2010), we studied the reaction of dipropylammonium oxalate with SnBr<sub>2</sub>, and report herein on the crystal structure of the title compound, ((CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>NH<sub>2</sub>)<sub>4</sub>[Sn(C<sub>2</sub>O<sub>4</sub>)<sub>4</sub>]·H<sub>2</sub>O.

The molecular structure of the title salt is illustrated in Fig. 1. The Sn<sup>IV</sup> atom of the stannate anion is located on a special position with  $\bar{4}2m$  symmetry. It is chelated by four bidentate oxalate ions, each lying in a mirror plane, and hence has a coordination number of eight. This coordination number has also been reported for some zirconium complexes, *viz.* bis-(4,4'-bipyridinium) tetrakis(oxalato- $\kappa^2O, O'$ )zirconate(IV) (Fu *et al.*, 2005), or several salts with general composition [(H<sub>2</sub>amine)<sub>2</sub>Zr(C<sub>2</sub>O<sub>4</sub>)<sub>4</sub>] (Imaz *et al.*, 2007).

The Sn1—O1 and Sn1—O3 bond lengths in the anion of the title compound are 2.142 (3) and 2.226 (3) Å, respectively. These values are similar to the bond lengths [2.189 (2) and 2.229 (2) Å] observed for another tin(IV)-oxalato complex, bis(dicyclohexylammonium) $\mu$ -oxalato- $\kappa^4O1, O2:O1', O2'$ -bis[aqua(oxalato- $\kappa^2O1, O2$ )diphenylstannate(IV)] (Gueye *et al.*, 2010).

In the crystal of the title salt, the stannate(IV) anions are linked *via* N—H···O hydrogen bonds to the [(CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>NH<sub>2</sub>]<sup>+</sup> cations (which have mirror symmetry), forming a three-dimensional network. The water molecule (disordered about a position with  $\bar{4}2m$  symmetry), is also involved in weak O—H···O hydrogen bonds with the stannate(IV) anions, hence further stabilizing the three-dimensional network (Fig. 2).

### S2. Experimental

The title compound was prepared by reacting in a 1:1 molar ratio of SnBr<sub>2</sub> and (Pr<sub>2</sub>NH<sub>2</sub>)<sub>2</sub>·C<sub>2</sub>O<sub>4</sub> in methanol. The solution was allowed to stand and yielded colourless block-like crystals of the title compound. The proof of the presence of the tin atom was confirmed by the structure analysis and by electron dispersive X-ray (EDX) analysis.

### S3. Refinement

The NH<sub>2</sub> and water H atoms were located in difference Fourier maps and refined with distance restraints: N—H = 0.88 (3) Å and O—H = 0.85 Å, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$  and  $= 1.5U_{\text{eq}}(\text{O})$ , respectively. The C-bond H-atoms were included in calculated positions and treated as riding atoms: C—H = 0.99 and 0.98 Å for CH<sub>2</sub> and CH<sub>3</sub> H atoms, respectively, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$  and  $= 1.2U_{\text{eq}}(\text{C})$  for other H-atoms.

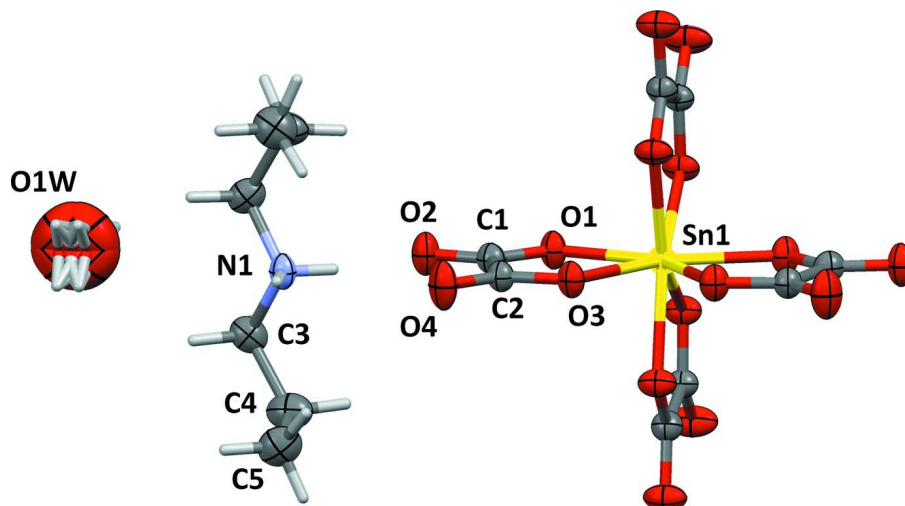


Figure 1

A view of the molecular components of the title salt. Displacement ellipsoids are drawn at the 50% probability level.

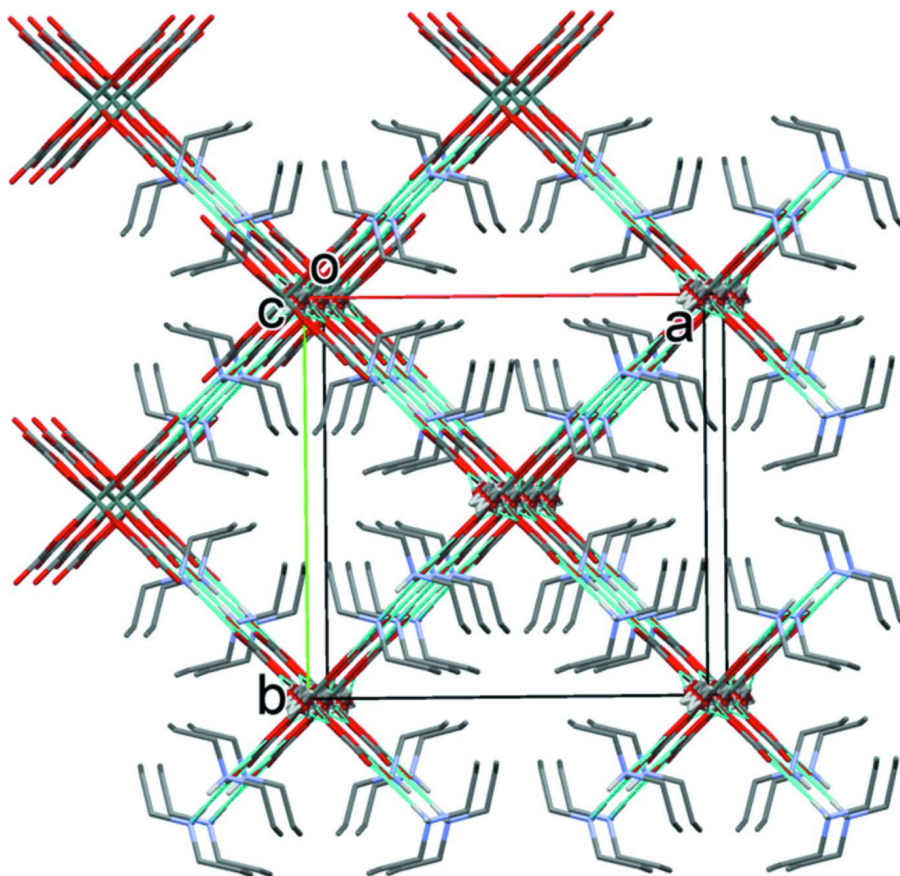


Figure 2

A view approximately along the *c* axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1 for details; C-bound H atoms have been omitted for clarity).

Tetrakis(dipropylammonium) tetrakis(oxalato- $\kappa^2O^1,O^2$ )stannate(IV)

## Crystal data

 $(C_6H_{16}N)_4[Sn(C_2O_4)_4] \cdot H_2O$  $M_r = 897.57$ Tetragonal,  $I\bar{4}2m$  $a = 14.5996$  (6) Å $c = 9.5718$  (4) Å $V = 2040.21$  (19) Å<sup>3</sup> $Z = 2$  $F(000) = 944$  $D_x = 1.461$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 16495 reflections

 $\theta = 2.0$ – $26.1^\circ$  $\mu = 0.70$  mm<sup>-1</sup> $T = 173$  K

Rod, colourless

 $0.45 \times 0.30 \times 0.22$  mm

## Data collection

STOE IPDS2

diffractometer

Radiation source: fine-focus sealed tube

Plane graphite monochromator

 $\varphi + \omega$  scans

Absorption correction: multi-scan

(MULScanABS in PLATON; Spek, 2009)

 $T_{\min} = 0.540$ ,  $T_{\max} = 1.000$ 

9636 measured reflections

1030 independent reflections

1027 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.071$  $\theta_{\max} = 25.6^\circ$ ,  $\theta_{\min} = 2.0^\circ$  $h = -17 \rightarrow 17$  $k = -17 \rightarrow 17$  $l = -10 \rightarrow 11$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.021$  $wR(F^2) = 0.054$  $S = 1.08$ 

1030 reflections

83 parameters

3 restraints

Primary atom site location: difference Fourier map

Secondary atom site location: inferred from neighbouring sites

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.030P)^2 + 1.5457P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.39$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.62$  e Å<sup>-3</sup>

Absolute structure: Refined as an inversion twin

Absolute structure parameter: 0.45 (4)

## 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.** Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Sn1	0.5000	0.5000	0.5000	0.01816 (16)	
O1	0.40111 (16)	0.40111 (16)	0.5675 (4)	0.0298 (8)	
O2	0.29743 (14)	0.29743 (14)	0.5079 (8)	0.0356 (7)	
O3	0.43891 (16)	0.43891 (16)	0.3084 (3)	0.0281 (7)	
O4	0.3366 (2)	0.3366 (2)	0.2305 (4)	0.0436 (10)	
C1	0.3559 (2)	0.3559 (2)	0.4778 (6)	0.0276 (13)	
C2	0.3776 (2)	0.3776 (2)	0.3231 (5)	0.0249 (9)	

N1	0.1921 (2)	0.1921 (2)	0.3202 (5)	0.0289 (9)	
H1AN	0.195 (3)	0.195 (3)	0.228 (3)	0.035*	
H1BN	0.2322 (15)	0.2322 (15)	0.350 (6)	0.035*	
C3	0.0985 (3)	0.2174 (3)	0.3686 (4)	0.0369 (8)	
H3A	0.0947	0.2087	0.4711	0.044*	
H3B	0.0532	0.1760	0.3245	0.044*	
C4	0.0741 (3)	0.3159 (3)	0.3335 (4)	0.0475 (10)	
H4A	0.0160	0.3315	0.3811	0.057*	
H4B	0.1223	0.3564	0.3720	0.057*	
C5	0.0637 (3)	0.3361 (3)	0.1801 (4)	0.0497 (10)	
H5A	0.1232	0.3296	0.1337	0.075*	
H5B	0.0413	0.3989	0.1679	0.075*	
H5C	0.0199	0.2931	0.1388	0.075*	
O1W	0.0000	-0.026 (3)	0.5000	0.091 (15)	0.25
H1WA	-0.0167	-0.0355	0.5840	0.136*	0.25

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn1	0.02109 (19)	0.02109 (19)	0.0123 (2)	0.000	0.000	0.000
O1	0.0352 (12)	0.0352 (12)	0.0190 (15)	-0.0117 (16)	-0.0020 (10)	-0.0020 (10)
O2	0.0417 (10)	0.0417 (10)	0.0234 (17)	-0.0187 (13)	-0.0001 (18)	-0.0001 (18)
O3	0.0346 (11)	0.0346 (11)	0.0151 (14)	-0.0089 (14)	-0.0026 (9)	-0.0026 (9)
O4	0.0548 (17)	0.0548 (17)	0.0211 (19)	-0.026 (2)	-0.0044 (12)	-0.0044 (12)
C1	0.0298 (13)	0.0298 (13)	0.023 (4)	-0.0010 (17)	-0.0020 (15)	-0.0020 (15)
C2	0.0273 (14)	0.0273 (14)	0.020 (2)	-0.0032 (18)	-0.0006 (13)	-0.0006 (13)
N1	0.0323 (14)	0.0323 (14)	0.022 (2)	-0.0083 (18)	-0.0029 (13)	-0.0029 (13)
C3	0.0356 (19)	0.049 (2)	0.0262 (17)	0.0009 (16)	0.0026 (14)	0.0014 (16)
C4	0.056 (3)	0.054 (3)	0.033 (2)	0.015 (2)	-0.0008 (19)	-0.0046 (18)
C5	0.064 (3)	0.051 (3)	0.035 (2)	0.002 (2)	0.000 (2)	0.0054 (18)
O1W	0.15 (5)	0.065 (15)	0.057 (7)	0.000	0.01 (3)	0.000

*Geometric parameters (Å, °)*

Sn1—O1 <sup>i</sup>	2.142 (3)	N1—H1AN	0.88 (3)
Sn1—O1 <sup>ii</sup>	2.142 (3)	N1—H1BN	0.88 (3)
Sn1—O1 <sup>iii</sup>	2.142 (3)	C3—C4	1.519 (6)
Sn1—O1	2.142 (3)	C3—H3A	0.9900
Sn1—O3	2.226 (3)	C3—H3B	0.9900
Sn1—O3 <sup>ii</sup>	2.226 (3)	C4—C5	1.506 (5)
Sn1—O3 <sup>iii</sup>	2.226 (3)	C4—H4A	0.9900
Sn1—O3 <sup>i</sup>	2.226 (3)	C4—H4B	0.9900
O1—C1	1.269 (6)	C5—H5A	0.9800
O2—C1	1.240 (6)	C5—H5B	0.9800
O3—C2	1.274 (6)	C5—H5C	0.9800
O4—C2	1.225 (6)	O1W—O1W <sup>v</sup>	0.55 (6)
C1—C2	1.547 (7)	O1W—O1W <sup>vi</sup>	0.55 (6)
N1—C3	1.490 (4)	O1W—O1W <sup>vii</sup>	0.77 (8)

N1—C3 <sup>iv</sup>	1.490 (4)	O1W—H1WA	0.8505
O1 <sup>i</sup> —Sn1—O1 <sup>ii</sup>	95.23 (5)	O4—C2—O3	127.3 (4)
O1 <sup>i</sup> —Sn1—O1 <sup>iii</sup>	95.23 (5)	O4—C2—C1	119.5 (4)
O1 <sup>ii</sup> —Sn1—O1 <sup>iii</sup>	144.86 (18)	O3—C2—C1	113.2 (4)
O1 <sup>i</sup> —Sn1—O1	144.86 (18)	C3—N1—C3 <sup>iv</sup>	111.0 (4)
O1 <sup>ii</sup> —Sn1—O1	95.23 (5)	C3—N1—H1AN	110 (2)
O1 <sup>iii</sup> —Sn1—O1	95.23 (5)	C3 <sup>iv</sup> —N1—H1AN	110 (2)
O1 <sup>i</sup> —Sn1—O3	142.09 (12)	C3—N1—H1BN	110.1 (19)
O1 <sup>ii</sup> —Sn1—O3	75.60 (8)	C3 <sup>iv</sup> —N1—H1BN	110.1 (19)
O1 <sup>iii</sup> —Sn1—O3	75.60 (8)	H1AN—N1—H1BN	106 (6)
O1—Sn1—O3	73.05 (13)	N1—C3—C4	112.4 (3)
O1 <sup>i</sup> —Sn1—O3 <sup>ii</sup>	75.60 (8)	N1—C3—H3A	109.1
O1 <sup>ii</sup> —Sn1—O3 <sup>ii</sup>	73.05 (13)	C4—C3—H3A	109.1
O1 <sup>iii</sup> —Sn1—O3 <sup>ii</sup>	142.09 (12)	N1—C3—H3B	109.1
O1—Sn1—O3 <sup>ii</sup>	75.60 (8)	C4—C3—H3B	109.1
O3—Sn1—O3 <sup>ii</sup>	132.75 (11)	H3A—C3—H3B	107.9
O1 <sup>i</sup> —Sn1—O3 <sup>iii</sup>	75.60 (8)	C5—C4—C3	115.2 (4)
O1 <sup>ii</sup> —Sn1—O3 <sup>iii</sup>	142.09 (12)	C5—C4—H4A	108.5
O1 <sup>iii</sup> —Sn1—O3 <sup>iii</sup>	73.05 (13)	C3—C4—H4A	108.5
O1—Sn1—O3 <sup>iii</sup>	75.60 (8)	C5—C4—H4B	108.5
O3—Sn1—O3 <sup>iii</sup>	132.75 (11)	C3—C4—H4B	108.5
O3 <sup>ii</sup> —Sn1—O3 <sup>iii</sup>	69.05 (17)	H4A—C4—H4B	107.5
O1 <sup>i</sup> —Sn1—O3 <sup>i</sup>	73.05 (13)	C4—C5—H5A	109.5
O1 <sup>ii</sup> —Sn1—O3 <sup>i</sup>	75.60 (8)	C4—C5—H5B	109.5
O1 <sup>iii</sup> —Sn1—O3 <sup>i</sup>	75.60 (8)	H5A—C5—H5B	109.5
O1—Sn1—O3 <sup>i</sup>	142.09 (12)	C4—C5—H5C	109.5
O3—Sn1—O3 <sup>i</sup>	69.05 (17)	H5A—C5—H5C	109.5
O3 <sup>ii</sup> —Sn1—O3 <sup>i</sup>	132.75 (11)	H5B—C5—H5C	109.5
O3 <sup>iii</sup> —Sn1—O3 <sup>i</sup>	132.75 (11)	O1W <sup>v</sup> —O1W—O1W <sup>vi</sup>	90.004 (7)
C1—O1—Sn1	119.8 (3)	O1W <sup>v</sup> —O1W—O1W <sup>vii</sup>	45.002 (4)
C2—O3—Sn1	118.2 (3)	O1W <sup>vi</sup> —O1W—O1W <sup>vii</sup>	45.002 (4)
O2—C1—O1	124.0 (6)	O1W <sup>v</sup> —O1W—H1WA	108.2
O2—C1—C2	120.3 (5)	O1W <sup>vi</sup> —O1W—H1WA	84.7
O1—C1—C2	115.7 (4)	O1W <sup>vii</sup> —O1W—H1WA	98.9
Sn1—O1—C1—O2	180.000 (1)	O1—C1—C2—O4	180.000 (1)
Sn1—O1—C1—C2	0.000 (1)	O2—C1—C2—O3	180.000 (1)
Sn1—O3—C2—O4	180.000 (1)	O1—C1—C2—O3	0.000 (1)
Sn1—O3—C2—C1	0.000 (1)	C3 <sup>iv</sup> —N1—C3—C4	174.3 (3)
O2—C1—C2—O4	0.000 (1)	N1—C3—C4—C5	67.2 (5)

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

#### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1AN $\cdots$ O1 <sup>viii</sup>	0.88 (3)	2.50 (5)	3.091 (6)	125 (5)

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N1—H1AN···O2 <sup>viii</sup>	0.88 (3)	2.12 (3)	2.997 (8)	179 (6)
N1—H1BN···O2	0.88 (3)	2.02 (4)	2.821 (7)	151 (5)
N1—H1BN···O4	0.88 (3)	2.44 (5)	3.105 (6)	133 (5)
O1W—H1WA···O3 <sup>ix</sup>	0.85	2.27	3.125 (7)	179

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Symmetry codes: (viii)  $-x+1/2, -y+1/2, z-1/2$ ; (ix)  $x-1/2, y-1/2, z+1/2$ .