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Crystal structures of diaquadi-μ-hydroxido-tris[trimethyltin(IV)] diformatotrimethylstannate(IV) and di-μ-hydroxido-tris[trimethyltin(IV)] chloride monohydrate

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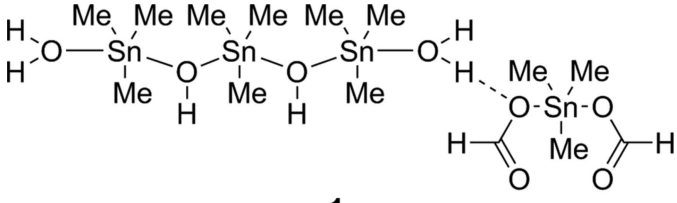
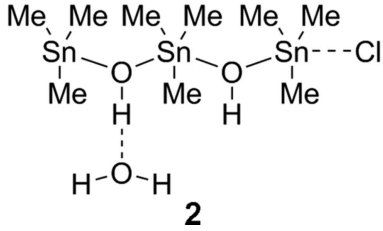
The title compounds, $[Sn_3(CH_3)_9(OH)_2(H_2O)_2][Sn(CH_3)_3(CHO_2)_2]$ (**1**) and $[Sn_3(CH_3)_9(OH)_2]Cl\cdot H_2O$ (**2**), are partially condensed products of hydrolysed trimethyltin chloride. In the structures of **1** and **2**, short cationic tristannatoxanes ($C_9H_{29}O_2Sn_3$) are bridged by a diformatotrimethyltin anion or a chloride anion, respectively. Hydrogen bridges are present and supposedly stabilize these structures against further polymerization to the known polymeric trimethyltin hydroxide. Especially noteworthy is that the formate present in this structure was formed from atmospheric CO_2.

1. Chemical context

Nowadays, there are many discussions about climate change and CO_2 emissions. Therefore, the activation of CO_2 plays an important role in today's research. It is already known that CO_2 is activated by electroreduction of different metals (Machunda *et al.*, 2011). A selective method to transform CO_2 into formate uses nanostructured tin catalysts (Zhang *et al.*, 2014). Compound **1** (Fig. 1) was formed from atmospheric CO_2 and thus can be regarded in the context of tin-mediated CO_2 activation. Compound **2** (Fig. 2) shows structural analogies and is also discussed herein. Structures **1** and **2** were obtained as byproducts from trapping reactions with trimethyltin chloride (Däschlein *et al.*, 2010; Unkelbach *et al.*, 2012; Koller *et al.*, 2015).



1



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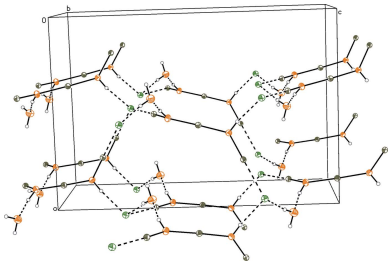

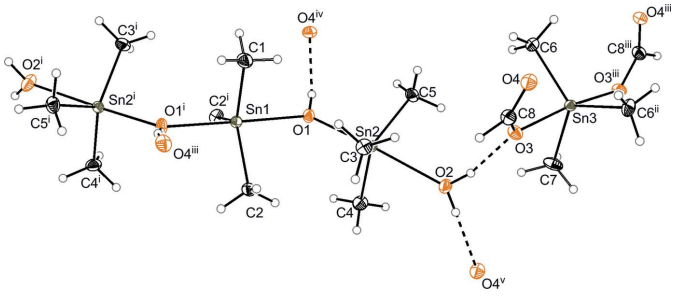

Figure 1
The molecular structure and atom numbering for compound **1**, with displacement ellipsoids drawn at the 30% probability level. [Symmetry codes: (i) $1-x, 2-y, z$; (ii) $1-x, 1-y, z$; (iii) $\frac{3}{2}-x, \frac{1}{2}+y, -z$; (iv) $-\frac{1}{2}+x, \frac{3}{2}-y, -z$; (v) $x, y, 1+z$.]

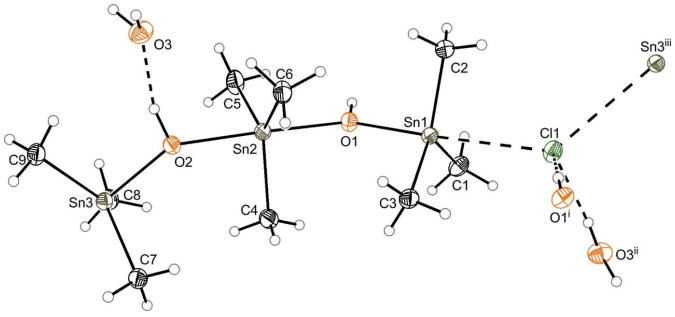

Figure 2
The molecular structure and atom numbering for compound **2**, with displacement ellipsoids drawn at the 30% probability level. [Symmetry codes: (i) $\frac{1}{2}+x, -y, z$; (ii) $\frac{3}{2}-x, y, \frac{1}{2}+z$; (iii) $\frac{3}{2}-x, -1+y, \frac{1}{2}+z$.]

2. Structural commentary

In the crystal structures, no polymeric Sn–O structures were formed, as found in the trimethyltin hydroxide. The short trimethyltin hydroxide chain has a positive and the chloride or bisformatostannate a negative charge. In the structure of **1**, both the cation and the anion are located about a twofold rotation axis whereas in that of **2** all atoms are on general positions. Owing to the presence of hydrogen bonds, there is a change to a smaller Sn—O—Sn angle relative to the polymeric trimethyltin hydroxide (Sn—O—Sn = 140°; Anderson *et al.*, 2011). In **1**, the Sn1—O1—Sn2 angle is 135.44 (9)° while in **2** it is 135.30 (17)°. In the chloride structure **2**, a change in two further angles is noticed. The O1—Sn1—Cl1 angle [177.58 (10)°] and the O2—Sn3—Cl1′ angle [175.5 (12)°] decreases (compare Lerner *et al.*, 2005). The water molecules exist in different situations in the two structures. In the formate structure **1**, a water molecule coordinates directly to the Sn2 atom. In compound **2**, the water is embedded in a hydrogen-bonded network between the negatively charged hydroxyl unit (O3···H2–O2) and the chloride anion.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O3	0.87 (2)	1.92 (3)	2.770 (3)	164 (4)
O2—H2B···O4^{i}	0.86 (2)	1.93 (2)	2.791 (3)	178 (3)
O1—H1···O4^{ii}	0.79 (4)	2.14 (4)	2.917 (3)	167 (3)

Symmetry codes: (i) $x, y, z+1$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$.

Table 2
Hydrogen-bond geometry (Å, °) for **2**.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3	0.94 (3)	1.81 (3)	2.726 (5)	164 (6)
O1—H1···Cl1^{i}	0.95 (3)	2.32 (3)	3.251 (4)	168 (6)
O3—H3D···Cl1^{ii}	0.97 (3)	2.10 (3)	3.068 (5)	171 (8)

Symmetry codes: (i) $x-\frac{1}{2}, -y, z$; (ii) $-x+\frac{3}{2}, y, z-\frac{1}{2}$.

3. Supramolecular features

As described, both structures are intermolecularly linked *via* hydrogen bonds. In structure **1** (Fig. 3 and Table 1), the formate anion is sterically too demanding to coordinate directly to the outer tin atom of the cationic chain. Therefore, the formate bridges four cationic tristannoxanes *via* hydrogen-bonding interactions (O3···H2*A*–O2, O4···H2*B*–O2), thus forming a two-dimensional network. Additionally, hydrogen bonds between these sheets form a two-dimensional network along the *bc* plane (O4···H1—O1).

In the chloride structure **2** (Fig. 4 and Table 2), the chloride anion bridges three cationic tristannoxanes, two by Sn···Cl

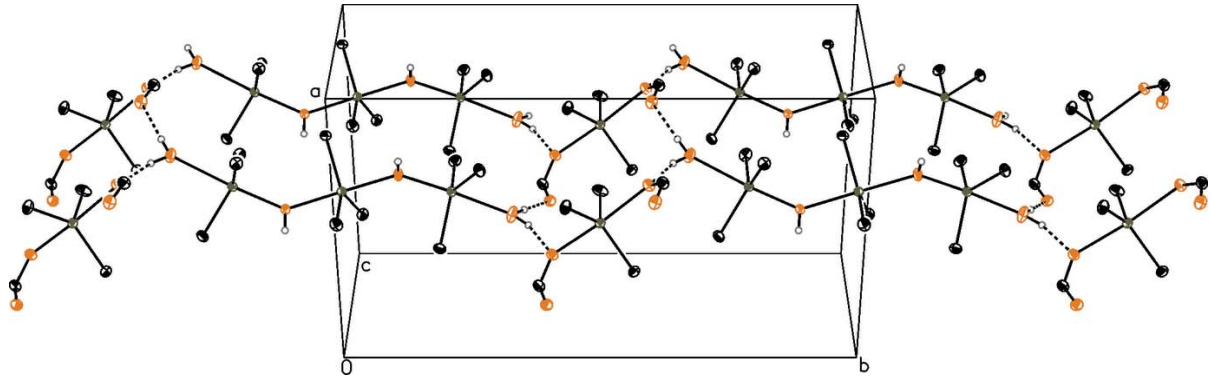

Figure 3
Crystal packing of compound **1**. H atoms not involved in hydrogen bonds have been omitted for clarity. Hydrogen bonds are drawn as black dashed lines (see Table 1).

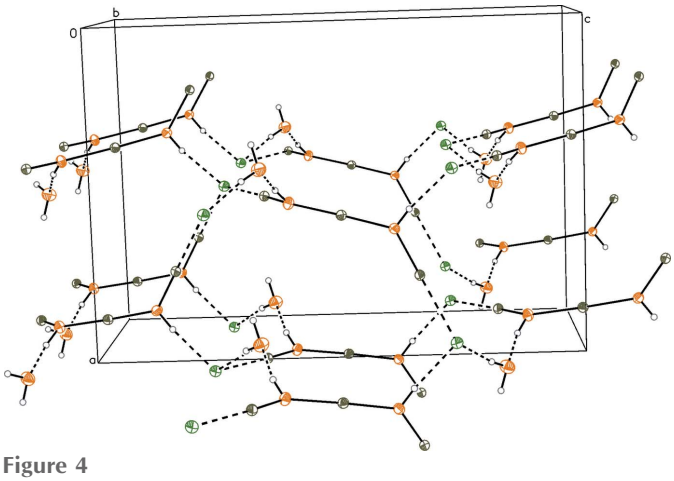

Figure 4
Crystal packing of compound **2**. H atoms not involved in hydrogen bonds have been omitted for clarity. Hydrogen bonds are drawn as black dashed lines (see Table 2).

interactions [Sn1···Cl1 = 3.024 (14); $Sn3^{iii}$···Cl1 = 3.166 (15) Å], one by a Cl1···$H1^{i}$—$O1^{i}$ hydrogen bond [3.251 (4) Å]. A fourth hydrogen bond, Cl1···$H3^{ii}$—$O3^{ii}$ [3.068 (5) Å], results in a distorted tetrahedral environment. Thus, a three-dimensional network of hydrogen bridges is formed. The interactions between Sn–Cl differ due to steric repulsion of the C2 and $C7^{iii}$ methyl groups. The van der Waals radius of a methyl group is 2 Å (Brown *et al.*, 2009) and the distance between the two units is *ca* 3.9 Å.

4. Database survey

The basic building block, trimethyltin hydroxide, has been known for a long time and has been completely characterized (Kraus & Bullard, 1929; Okawara & Yasuda, 1964). Since then, studies using single crystal X-ray analysis have been made for the exact structure. A polymeric structure with eight units has been found, which has an angle of *ca* 140° for the Sn—O—Sn bond (Anderson *et al.*, 2011). Tiekink (1986) succeeded in obtaining a bis(trimethyltin)carbonate, wherein the basic polymeric structure has been changed. Here, the trimethyltin units are linked *via* a carbonate. A dimeric structure including chloride as anion and water is also noted. The tin atoms are coordinated by the bridging Cl and HO substituents and angles of 133.2 (2)° for Sn1—Cl1—Sn2 and 179.2 (2)° for O1—Sn1—Cl1 were observed (Lerner *et al.*, 2005).

5. Synthesis and crystallization

The two structures were obtained as byproducts from trapping reactions with trimethyltin chloride (Strohmann *et al.*, 2006; Ott *et al.*, 2008). The samples were stored under atmospheric

Table 3
Experimental details.

	1	**2**
Crystal data		
Chemical formula	$[Sn_3(CH_3)_9(OH)_2(H_2O)_2][Sn(CH_3)_3(CHO_2)_2]$	$[Sn_3(CH_3)_9(OH)_2]Cl{\cdot}H_2O$
M_r	407.62	578.86
Crystal system, space group	Orthorhombic, $P2_12_12$	Orthorhombic, $Pca2_1$
Temperature (K)	154	100
a, b, c (Å)	11.0786 (8), 18.9529 (14), 6.6990 (5)	12.623 (3), 8.2675 (18), 18.421 (5)
V (Å^3)	1406.60 (18)	1922.4 (8)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	3.54	4.00
Crystal size (mm)	0.16 × 0.10 × 0.08	0.16 × 0.14 × 0.07
Data collection		
Diffractometer	Bruker D8 VENTURE area detector	Bruker D8 VENTURE area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.016, 0.038	0.010, 0.032
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	56576, 3966, 3811	16017, 5320, 5072
R_{int}	0.036	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.696	0.697
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.014, 0.027, 1.06	0.022, 0.050, 1.06
No. of reflections	3966	5320
No. of parameters	144	170
No. of restraints	2	5
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.33	1.01, −0.38
Absolute structure	Flack x determined using 1569 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)	Flack x determined using 2271 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.040 (19)	−0.026 (19)

Computer programs: *APEX3* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

conditions for a few months. By reaction with atmospheric moisture, partial hydrolysis occurred. In the case of compound **1**, CO_2 was also activated by a tin-mediated reaction.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms involved in hydrogen bonding were located in a difference Fourier synthesis map and freely refined. All other H atoms were positioned geometrically and refined using a riding model: C—H = 0.98 Å with U_{iso}(H) = 1.5U_{eq}(Cmethyl). The CH_3 hydrogen atoms were allowed to rotate but not to tip. Due to point group symmetry 2 of both the cation and anion in **1**, with the twofold rotation axis running through the respective central Sn atom and one of the methyl groups, the latter is equally disordered over two positions.

Acknowledgements

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Crystal structures of diaquadi-μ-hydroxido-tris[trimethyltin(IV)] diformatotrimethylstannate(IV) and di-μ-hydroxido-tris[trimethyltin(IV)] chloride monohydrate

Felix Otte, Stephan G. Koller, Christopher Golz and Carsten Strohmann

Computing details

For both compounds, data collection: *APEX3* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

(1) Diaquadi-μ-hydroxido-tris[trimethyltin(IV)] diformatotrimethylstannate(IV)

Crystal data

$[Sn_3(CH_3)_9(OH)_2(H_2O)_2][Sn(CH_3)_3(CHO_2)_2]$
M_r = 407.62
Orthorhombic, $P2_12_12$
a = 11.0786 (8) Å
b = 18.9529 (14) Å
c = 6.6990 (5) Å
V = 1406.60 (18) Å^3
Z = 4
$F(000)$ = 784
D_x = 1.925 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9917 reflections
θ = 3–60°
μ = 3.54 mm^{-1}
T = 154 K
Block, colourless
0.16 × 0.10 × 0.08 mm

Data collection

Bruker D8 VENTURE area detector diffractometer
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs
HELIOS mirror optics monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.016, T_{max} = 0.038
56576 measured reflections
3966 independent reflections
3811 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 29.6°, θ_{min} = 2.8°
h = −15→15
k = −26→26
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.014
$wR(F^2)$ = 0.027
S = 1.06
3966 reflections
144 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0094P)^2 + 0.4247P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004

$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00294 (12)
Absolute structure: Flack *x* determined using 1569 quotients $[(I^{+})-(I^{-})]/[(I^{+})+(I^{-})]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.040 (19)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^{2})

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Sn1	0.5000	1.0000	0.39740 (3)	0.01976 (5)	
Sn2	0.51010 (2)	0.78803 (2)	0.42354 (2)	0.01984 (4)	
Sn3	0.5000	0.5000	0.09198 (3)	0.01949 (5)	
O1	0.43169 (16)	0.88858 (8)	0.3940 (3)	0.0275 (4)	
O2	0.6162 (2)	0.67229 (10)	0.4624 (3)	0.0395 (5)	
H2A	0.621 (4)	0.6398 (17)	0.370 (5)	0.070 (12)*	
H2B	0.639 (3)	0.6499 (15)	0.567 (4)	0.047 (9)*	
O3	0.63477 (16)	0.59175 (9)	0.1188 (3)	0.0275 (4)	
O4	0.69708 (18)	0.60086 (10)	−0.1986 (3)	0.0321 (4)	
C1	0.5000	1.0000	0.0790 (5)	0.0341 (7)	
H1A	0.5212	0.9529	0.0302	0.051*	0.5
H1B	0.5592	1.0343	0.0302	0.051*	0.5
H1C	0.4195	1.0128	0.0302	0.051*	0.5
C2	0.6556 (2)	0.96914 (13)	0.5607 (4)	0.0284 (5)	
H2C	0.6378	0.9263	0.6370	0.043*	
H2D	0.6787	1.0071	0.6524	0.043*	
H2E	0.7221	0.9598	0.4679	0.043*	
C3	0.6662 (2)	0.81086 (14)	0.2519 (4)	0.0306 (6)	
H3A	0.6517	0.8531	0.1709	0.046*	
H3B	0.6842	0.7708	0.1643	0.046*	
H3C	0.7347	0.8192	0.3414	0.046*	
C4	0.5122 (3)	0.78733 (14)	0.7404 (3)	0.0326 (5)	
H4A	0.5957	0.7907	0.7878	0.049*	
H4B	0.4762	0.7433	0.7891	0.049*	
H4C	0.4657	0.8276	0.7908	0.049*	
C5	0.3709 (2)	0.72972 (13)	0.2806 (4)	0.0308 (6)	
H5A	0.2923	0.7504	0.3140	0.046*	
H5B	0.3733	0.6806	0.3261	0.046*	
H5C	0.3827	0.7313	0.1357	0.046*	
C6	0.3758 (2)	0.56431 (13)	−0.0639 (4)	0.0318 (6)	
H6A	0.3182	0.5848	0.0307	0.048*	
H6B	0.3323	0.5357	−0.1622	0.048*	
H6C	0.4195	0.6022	−0.1324	0.048*	
C7	0.5000	0.5000	0.4082 (4)	0.0354 (8)	

H7A	0.5631	0.4681	0.4570	0.053*	0.5
H7B	0.4212	0.4840	0.4570	0.053*	0.5
H7C	0.5156	0.5479	0.4570	0.053*	0.5
C8	0.6991 (3)	0.61657 (14)	−0.0214 (4)	0.0293 (6)	
H8	0.754 (3)	0.6574 (18)	0.034 (5)	0.059 (11)*	
H1	0.365 (3)	0.8863 (17)	0.352 (5)	0.051 (11)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01811 (9)	0.01947 (9)	0.02172 (9)	0.00005 (9)	0.000	0.000
Sn2	0.02197 (7)	0.01935 (7)	0.01820 (7)	−0.00208 (7)	−0.00006 (8)	0.00073 (5)
Sn3	0.02102 (9)	0.02158 (9)	0.01588 (9)	0.00227 (9)	0.000	0.000
O1	0.0229 (8)	0.0186 (8)	0.0409 (11)	−0.0023 (6)	−0.0077 (9)	0.0001 (8)
O2	0.0672 (15)	0.0270 (10)	0.0243 (10)	0.0151 (10)	−0.0095 (10)	−0.0033 (8)
O3	0.0312 (9)	0.0298 (9)	0.0215 (9)	−0.0063 (7)	0.0027 (7)	−0.0014 (7)
O4	0.0402 (11)	0.0324 (10)	0.0237 (9)	0.0027 (8)	0.0069 (8)	0.0040 (8)
C1	0.0422 (19)	0.0356 (17)	0.0246 (15)	−0.0051 (19)	0.000	0.000
C2	0.0232 (11)	0.0292 (12)	0.0328 (14)	0.0009 (9)	−0.0063 (11)	−0.0031 (11)
C3	0.0274 (13)	0.0346 (14)	0.0298 (14)	−0.0020 (10)	0.0062 (11)	0.0034 (11)
C4	0.0401 (14)	0.0367 (12)	0.0209 (10)	0.0067 (16)	0.0047 (13)	−0.0009 (9)
C5	0.0332 (14)	0.0272 (12)	0.0320 (14)	−0.0078 (10)	−0.0052 (11)	−0.0016 (10)
C6	0.0341 (13)	0.0263 (12)	0.0350 (15)	0.0070 (10)	−0.0090 (13)	−0.0015 (11)
C7	0.0332 (17)	0.056 (2)	0.0170 (14)	−0.014 (2)	0.000	0.000
C8	0.0310 (14)	0.0290 (14)	0.0280 (14)	−0.0054 (11)	0.0015 (10)	0.0024 (10)

Geometric parameters (Å, º)

Sn1—O1	2.2433 (16)	C1—H1B	0.9800
Sn1—O1^{i}	2.2433 (16)	C1—H1C	0.9800
Sn1—C1	2.133 (3)	C2—H2C	0.9800
Sn1—C2	2.124 (2)	C2—H2D	0.9800
Sn1—C2^{i}	2.124 (2)	C2—H2E	0.9800
Sn2—O1	2.1036 (16)	C3—H3A	0.9800
Sn2—O2	2.5023 (19)	C3—H3B	0.9800
Sn2—C3	2.121 (2)	C3—H3C	0.9800
Sn2—C4	2.123 (2)	C4—H4A	0.9800
Sn2—C5	2.126 (2)	C4—H4B	0.9800
Sn3—O3^{ii}	2.2991 (17)	C4—H4C	0.9800
Sn3—O3	2.2990 (17)	C5—H5A	0.9800
Sn3—C6	2.114 (2)	C5—H5B	0.9800
Sn3—C6^{ii}	2.114 (2)	C5—H5C	0.9800
Sn3—C7	2.119 (3)	C6—H6A	0.9800
O1—H1	0.79 (4)	C6—H6B	0.9800
O2—H2A	0.87 (2)	C6—H6C	0.9800
O2—H2B	0.86 (2)	C7—H7A	0.9800
O3—C8	1.269 (3)	C7—H7B	0.9800
O4—C8	1.224 (3)	C7—H7C	0.9800

C1—H1A	0.9800	C8—H8	1.05 (3)
O1—Sn1—O1^{i}	178.83 (11)	H1A—C1—H1C	109.5
C1—Sn1—O1^{i}	89.42 (5)	H1B—C1—H1C	109.5
C1—Sn1—O1	89.42 (5)	Sn1—C2—H2C	109.5
C2—Sn1—O1	91.14 (8)	Sn1—C2—H2D	109.5
C2—Sn1—O1^{i}	89.46 (8)	Sn1—C2—H2E	109.5
C2^{i}—Sn1—O1	89.46 (8)	H2C—C2—H2D	109.5
C2^{i}—Sn1—O1^{i}	91.14 (8)	H2C—C2—H2E	109.5
C2^{i}—Sn1—C1	121.00 (7)	H2D—C2—H2E	109.5
C2—Sn1—C1	121.00 (7)	Sn2—C3—H3A	109.5
C2—Sn1—C2^{i}	118.01 (15)	Sn2—C3—H3B	109.5
O1—Sn2—O2	176.30 (8)	Sn2—C3—H3C	109.5
O1—Sn2—C3	95.79 (9)	H3A—C3—H3B	109.5
O1—Sn2—C4	95.99 (9)	H3A—C3—H3C	109.5
O1—Sn2—C5	97.41 (9)	H3B—C3—H3C	109.5
C3—Sn2—O2	81.51 (9)	Sn2—C4—H4A	109.5
C3—Sn2—C4	122.30 (12)	Sn2—C4—H4B	109.5
C3—Sn2—C5	116.97 (11)	Sn2—C4—H4C	109.5
C4—Sn2—O2	83.41 (9)	H4A—C4—H4B	109.5
C4—Sn2—C5	117.08 (11)	H4A—C4—H4C	109.5
C5—Sn2—O2	86.09 (9)	H4B—C4—H4C	109.5
O3—Sn3—O3^{ii}	171.04 (9)	Sn2—C5—H5A	109.5
C6—Sn3—O3^{ii}	92.99 (9)	Sn2—C5—H5B	109.5
C6^{ii}—Sn3—O3^{ii}	91.44 (9)	Sn2—C5—H5C	109.5
C6^{ii}—Sn3—O3	92.99 (9)	H5A—C5—H5B	109.5
C6—Sn3—O3	91.43 (9)	H5A—C5—H5C	109.5
C6^{ii}—Sn3—C6	120.80 (16)	H5B—C5—H5C	109.5
C6—Sn3—C7	119.60 (8)	Sn3—C6—H6A	109.5
C6^{ii}—Sn3—C7	119.60 (8)	Sn3—C6—H6B	109.5
C7—Sn3—O3^{ii}	85.52 (4)	Sn3—C6—H6C	109.5
C7—Sn3—O3	85.52 (4)	H6A—C6—H6B	109.5
Sn1—O1—H1	112 (2)	H6A—C6—H6C	109.5
Sn2—O1—Sn1	135.44 (9)	H6B—C6—H6C	109.5
Sn2—O1—H1	112 (2)	Sn3—C7—H7A	109.5
Sn2—O2—H2A	125 (3)	Sn3—C7—H7B	109.5
Sn2—O2—H2B	131 (2)	Sn3—C7—H7C	109.5
H2A—O2—H2B	102 (3)	H7A—C7—H7B	109.5
C8—O3—Sn3	125.94 (17)	H7A—C7—H7C	109.5
Sn1—C1—H1A	109.5	H7B—C7—H7C	109.5
Sn1—C1—H1B	109.5	O3—C8—H8	110 (2)
Sn1—C1—H1C	109.5	O4—C8—O3	128.1 (3)
H1A—C1—H1B	109.5	O4—C8—H8	122 (2)
Sn3—O3—C8—O4	5.5 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O3	0.87 (2)	1.92 (3)	2.770 (3)	164 (4)
O2—H2*B*···O4^{iii}	0.86 (2)	1.93 (2)	2.791 (3)	178 (3)
O1—H1···O4^{iv}	0.79 (4)	2.14 (4)	2.917 (3)	167 (3)

Symmetry codes: (iii) *x*, *y*, *z*+1; (iv) *x*−1/2, −*y*+3/2, −*z*.

(2) Di-*μ*-hydroxido-tris[trimethyltin(IV)] chloride monohydrate

Crystal data

$[Sn_3(CH_3)_9(OH)_2]Cl·H_2O$
M_r = 578.86
Orthorhombic, $Pca2_1$
a = 12.623 (3) Å
b = 8.2675 (18) Å
c = 18.421 (5) Å
V = 1922.4 (8) Å^3
Z = 4
F(000) = 1104
D_x = 2.000 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 9903 reflections
θ = 2.9–29.6°
μ = 4.00 mm^{-1}
T = 100 K
Block, colourless
0.16 × 0.14 × 0.07 mm

Data collection

Bruker D8 VENTURE area detector diffractometer
Detector resolution: 10.4167 pixels mm^{-1}
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.010, T_{max} = 0.032
16017 measured reflections
5320 independent reflections
5072 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 29.7°, θ_{min} = 2.2°
h = −15→17
k = −11→11
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.050
S = 1.06
5320 reflections
170 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0269P)^2 + 0.6817P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.01 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack *x* determined using 2271 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.026 (19)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7513 (5)	0.1853 (7)	0.7655 (3)	0.0397 (11)

H1A	0.7352	0.2995	0.7566	0.059*
H1B	0.8181	0.1767	0.7924	0.059*
H1C	0.6941	0.1366	0.7941	0.059*
C2	0.7054 (4)	−0.1756 (6)	0.6542 (3)	0.0345 (10)
H2A	0.6448	−0.1902	0.6868	0.052*
H2B	0.7609	−0.2537	0.6667	0.052*
H2C	0.6828	−0.1931	0.6039	0.052*
C3	0.8814 (4)	0.1523 (7)	0.5927 (3)	0.0358 (11)
H3A	0.8661	0.1136	0.5435	0.054*
H3B	0.9515	0.1141	0.6077	0.054*
H3C	0.8802	0.2709	0.5933	0.054*
C4	0.6887 (5)	0.4752 (7)	0.5271 (3)	0.0436 (13)
H4A	0.7587	0.4640	0.5046	0.065*
H4B	0.6969	0.4882	0.5797	0.065*
H4C	0.6527	0.5702	0.5071	0.065*
C5	0.4333 (5)	0.2706 (8)	0.5290 (3)	0.0490 (15)
H5A	0.4053	0.3790	0.5191	0.073*
H5B	0.4221	0.2438	0.5802	0.073*
H5C	0.3965	0.1915	0.4984	0.073*
C6	0.6638 (5)	0.0545 (6)	0.4578 (3)	0.0381 (11)
H6A	0.6173	0.0158	0.4189	0.057*
H6B	0.6712	−0.0298	0.4948	0.057*
H6C	0.7336	0.0803	0.4376	0.057*
C7	0.7215 (4)	0.6488 (6)	0.3435 (3)	0.0367 (10)
H7A	0.7630	0.5597	0.3640	0.055*
H7B	0.7275	0.7439	0.3750	0.055*
H7C	0.7485	0.6753	0.2950	0.055*
C8	0.4542 (4)	0.7036 (6)	0.4051 (3)	0.0359 (11)
H8A	0.3839	0.6535	0.4026	0.054*
H8B	0.4493	0.8169	0.3898	0.054*
H8C	0.4806	0.6985	0.4551	0.054*
C9	0.5063 (5)	0.4823 (7)	0.2352 (3)	0.0390 (11)
H9A	0.5524	0.5207	0.1960	0.059*
H9B	0.4335	0.5179	0.2260	0.059*
H9C	0.5085	0.3639	0.2371	0.059*
O1	0.6340 (3)	0.1729 (4)	0.61594 (19)	0.0327 (7)
H1	0.575 (4)	0.168 (9)	0.648 (3)	0.06 (2)*
O2	0.5652 (3)	0.3638 (4)	0.39350 (19)	0.0333 (7)
H2	0.519 (4)	0.293 (6)	0.369 (3)	0.040 (16)*
Sn1	0.76540 (2)	0.06256 (3)	0.66540 (2)	0.02692 (7)
Sn2	0.59742 (2)	0.26536 (3)	0.50553 (2)	0.02755 (7)
Sn3	0.55935 (2)	0.57842 (4)	0.33566 (2)	0.02786 (7)
O3	0.4394 (3)	0.1219 (5)	0.3417 (3)	0.0488 (10)
H3D	0.467 (7)	0.043 (8)	0.308 (4)	0.08 (3)*
H3E	0.365 (4)	0.127 (15)	0.328 (10)	0.18 (6)*
Cl1	0.94986 (10)	−0.10984 (17)	0.73601 (7)	0.0379 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.037 (3)	0.049 (3)	0.033 (3)	0.000 (3)	−0.002 (2)	−0.011 (2)
C2	0.033 (2)	0.034 (2)	0.037 (3)	−0.0031 (19)	−0.002 (2)	0.0016 (19)
C3	0.029 (2)	0.042 (3)	0.036 (3)	−0.003 (2)	0.002 (2)	0.004 (2)
C4	0.058 (4)	0.037 (3)	0.035 (3)	−0.009 (3)	−0.009 (2)	0.003 (2)
C5	0.035 (3)	0.068 (4)	0.044 (3)	0.007 (3)	0.004 (2)	0.019 (3)
C6	0.048 (3)	0.034 (2)	0.032 (2)	0.007 (2)	−0.006 (2)	−0.0010 (19)
C7	0.032 (2)	0.044 (3)	0.034 (2)	−0.002 (2)	0.000 (2)	0.006 (2)
C8	0.036 (3)	0.041 (3)	0.031 (2)	0.003 (2)	0.003 (2)	0.002 (2)
C9	0.046 (3)	0.039 (3)	0.032 (2)	0.000 (2)	−0.008 (2)	0.001 (2)
O1	0.0276 (17)	0.0410 (18)	0.0295 (17)	0.0025 (15)	0.0020 (14)	0.0056 (14)
O2	0.041 (2)	0.0288 (17)	0.0306 (17)	−0.0010 (15)	−0.0037 (15)	0.0010 (13)
Sn1	0.02689 (14)	0.02985 (14)	0.02403 (13)	−0.00150 (11)	0.00091 (13)	0.00026 (11)
Sn2	0.02642 (14)	0.02809 (14)	0.02815 (14)	0.00031 (11)	−0.00014 (13)	0.00004 (12)
Sn3	0.02768 (15)	0.02928 (14)	0.02664 (14)	0.00066 (11)	−0.00002 (13)	0.00054 (12)
O3	0.047 (2)	0.045 (2)	0.054 (3)	0.0023 (18)	−0.005 (2)	−0.011 (2)
Cl1	0.0311 (6)	0.0487 (7)	0.0339 (6)	0.0014 (5)	−0.0004 (5)	0.0028 (5)

Geometric parameters (Å, º)

C1—H1A	0.9800	C6—Sn2	2.125 (5)
C1—H1B	0.9800	C7—H7A	0.9800
C1—H1C	0.9800	C7—H7B	0.9800
C1—Sn1	2.113 (5)	C7—H7C	0.9800
C2—H2A	0.9800	C7—Sn3	2.133 (5)
C2—H2B	0.9800	C8—H8A	0.9800
C2—H2C	0.9800	C8—H8B	0.9800
C2—Sn1	2.120 (5)	C8—H8C	0.9800
C3—H3A	0.9800	C8—Sn3	2.114 (5)
C3—H3B	0.9800	C9—H9A	0.9800
C3—H3C	0.9800	C9—H9B	0.9800
C3—Sn1	2.118 (5)	C9—H9C	0.9800
C4—H4A	0.9800	C9—Sn3	2.123 (5)
C4—H4B	0.9800	O1—H1	0.95 (3)
C4—H4C	0.9800	O1—Sn1	2.100 (3)
C4—Sn2	2.120 (5)	O1—Sn2	2.222 (3)
C5—H5A	0.9800	O2—H2	0.94 (3)
C5—H5B	0.9800	O2—Sn2	2.255 (4)
C5—H5C	0.9800	O2—Sn3	2.071 (3)
C5—Sn2	2.117 (6)	Sn1—Cl1	3.0240 (14)
C6—H6A	0.9800	O3—H3D	0.97 (3)
C6—H6B	0.9800	O3—H3E	0.98 (3)
C6—H6C	0.9800	Cl1—Sn3^{i}	3.1663 (15)
H1A—C1—H1B	109.5	H8B—C8—H8C	109.5
H1A—C1—H1C	109.5	Sn3—C8—H8A	109.5

H1B—C1—H1C	109.5	Sn3—C8—H8B	109.5
Sn1—C1—H1A	109.5	Sn3—C8—H8C	109.5
Sn1—C1—H1B	109.5	H9A—C9—H9B	109.5
Sn1—C1—H1C	109.5	H9A—C9—H9C	109.5
H2A—C2—H2B	109.5	H9B—C9—H9C	109.5
H2A—C2—H2C	109.5	Sn3—C9—H9A	109.5
H2B—C2—H2C	109.5	Sn3—C9—H9B	109.5
Sn1—C2—H2A	109.5	Sn3—C9—H9C	109.5
Sn1—C2—H2B	109.5	Sn1—O1—H1	109 (4)
Sn1—C2—H2C	109.5	Sn1—O1—Sn2	135.30 (17)
H3A—C3—H3B	109.5	Sn2—O1—H1	115 (4)
H3A—C3—H3C	109.5	Sn2—O2—H2	109 (4)
H3B—C3—H3C	109.5	Sn3—O2—H2	105 (4)
Sn1—C3—H3A	109.5	Sn3—O2—Sn2	141.84 (17)
Sn1—C3—H3B	109.5	C1—Sn1—C2	120.1 (2)
Sn1—C3—H3C	109.5	C1—Sn1—C3	116.2 (2)
H4A—C4—H4B	109.5	C1—Sn1—Cl1	85.16 (17)
H4A—C4—H4C	109.5	C2—Sn1—Cl1	83.06 (14)
H4B—C4—H4C	109.5	C3—Sn1—C2	120.7 (2)
Sn2—C4—H4A	109.5	C3—Sn1—Cl1	84.55 (15)
Sn2—C4—H4B	109.5	O1—Sn1—C1	95.96 (19)
Sn2—C4—H4C	109.5	O1—Sn1—C2	94.52 (17)
H5A—C5—H5B	109.5	O1—Sn1—C3	96.85 (17)
H5A—C5—H5C	109.5	O1—Sn1—Cl1	177.58 (10)
H5B—C5—H5C	109.5	C4—Sn2—C6	122.3 (3)
Sn2—C5—H5A	109.5	C4—Sn2—O1	89.81 (18)
Sn2—C5—H5B	109.5	C4—Sn2—O2	88.55 (17)
Sn2—C5—H5C	109.5	C5—Sn2—C4	118.5 (3)
H6A—C6—H6B	109.5	C5—Sn2—C6	119.2 (3)
H6A—C6—H6C	109.5	C5—Sn2—O1	91.36 (18)
H6B—C6—H6C	109.5	C5—Sn2—O2	90.15 (19)
Sn2—C6—H6A	109.5	C6—Sn2—O1	90.83 (17)
Sn2—C6—H6B	109.5	C6—Sn2—O2	89.35 (17)
Sn2—C6—H6C	109.5	O1—Sn2—O2	178.17 (14)
H7A—C7—H7B	109.5	C8—Sn3—C7	115.3 (2)
H7A—C7—H7C	109.5	C8—Sn3—C9	120.9 (2)
H7B—C7—H7C	109.5	C9—Sn3—C7	117.6 (2)
Sn3—C7—H7A	109.5	O2—Sn3—C7	99.50 (18)
Sn3—C7—H7B	109.5	O2—Sn3—C8	97.50 (17)
Sn3—C7—H7C	109.5	O2—Sn3—C9	97.99 (18)
H8A—C8—H8B	109.5	H3D—O3—H3E	102 (10)
H8A—C8—H8C	109.5	Sn1—Cl1—Sn3^{i}	127.21 (4)

Symmetry code: (i) $-x+3/2, y-1, z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3	0.94 (3)	1.81 (3)	2.726 (5)	164 (6)

O1—H1···Cl1^{ii}	0.95 (3)	2.32 (3)	3.251 (4)	168 (6)
O3—H3*D*···Cl1^{iii}	0.97 (3)	2.10 (3)	3.068 (5)	171 (8)

Symmetry codes: (ii) $x-1/2, -y, z$; (iii) $-x+3/2, y, z-1/2$.