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Diaquatetrachloridotin(IV)–diglyme (1/2)

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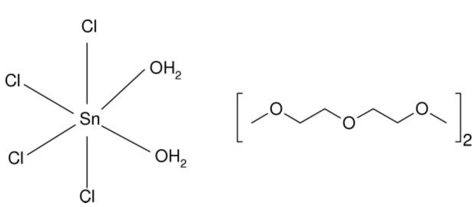
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.022; wR factor = 0.050; data-to-parameter ratio = 21.8.

In the title 1:2 adduct, $[SnCl_4(H_2O)_2]\cdot 2C_6H_{14}O_3$, the Sn^{IV} atom (site symmetry 2) adopts a *cis*-SnO_2Cl_4 octahedral geometry. In the crystal structure, O—H···O hydrogen bonds lead to associations of one metal complex and two diglyme molecules.

Related literature

For related structures, see: Valle *et al.* (1984); Hough *et al.* (1986); Azadmehr *et al.* (2001). For further synthetic details, see: Hutton & Oakes (1976). For reference structural data, see: Allen *et al.* (1987). For bond valence sum calculations, see: Brese & O'Keeffe (1991).



Experimental

Crystal data

$[SnCl_4(H_2O)_2]\cdot 2C_6H_{14}O_3$	V = 2300.38 (9) Å^3
M_r = 564.86	Z = 4
Orthorhombic, *Pbcn*	Mo $K\alpha$ radiation
a = 8.4023 (2) Å	μ = 1.61 mm^{-1}
b = 17.1528 (3) Å	T = 120 (2) K
c = 15.9612 (4) Å	0.55 × 0.43 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer	20197 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)	2643 independent reflections
T_{min} = 0.472, T_{max} = 0.795	2292 reflections with $I > 2\sigma(I)$
	R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.050	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.50 e Å^{-3}
2643 reflections	
121 parameters	

Table 1
Selected bond lengths (Å).

Sn1—O1	2.1343 (13)	Sn1—Cl2	2.3853 (5)
Sn1—Cl1	2.3772 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H2···O2	0.77 (2)	1.88 (2)	2.6503 (18)	175 (2)
O1—H1···O4	0.82 (2)	1.91 (2)	2.7296 (18)	175 (2)

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*, and *DENZO* (Otwinowski & Minor, 1997) and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2792).

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supplementary materials

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Comment

The title compound, (I), (Fig. 1) complements related adducts containing the same metal complex accompanied by various crown ethers (Valle *et al.*, 1984; Hough *et al.*, 1986; Azadmehr *et al.*, 2001).

In (I), the tin(IV) atom lies on a crystallographic twofold rotation axis, and bonds to two water molecules and four chloride ions, with the water O atoms in *cis* conformation {O1—Sn1—O1^{i} = 82.72 (8)°; i = -*x*, *y*, 3/2 - *z*]. Overall, a distorted octahedral coordination arises for the metal (Table 1). The bond valence sum (BVS) (Brese & O'Keeffe, 1991) for tin is 4.09 (expected value = 4.00).

In the crystal, the $Sn(H_2O)_2Cl_4$ moiety links to two adjacent $C_6H_{14}O_3$ (diglyme) molecules by way of O—H···O hydrogen bonds (Table 2), with each water molecule making two such bonds to the same diglyme species (Fig. 2). This hydrogen bonding pattern may correlate with the fact that the O—C—C—O torsion angles reflect *gauche* conformations about the C2—C3 and C4—C5 bonds [O2—C2—C3—O3 = 65.6 (2)°; O3—C4—C5—O4 = -64.9 (2)°], whereas the four C—C—O—C conformations are *trans*. Otherwise, the geometrical paramaters for (I) may be regarded as normal (Allen *et al.*, 1987).

Experimental

Air-stable, colourless slabs of (I) were isolated from the slow evaporation of a methanolic solution (20 ml) containing 0.1 mmol $Cl_3SnCH_2CH_2CO_2H$ (Hutton & Oakes, 1976) and 0.1 mmol diglyme. M.P. 353–355 K. IR (KBr): 3500–2500, 1363, 1471, 1454, 1354,1287, 1250, 1141, 1102, 1079, 1105, 860, 834, 701, 617 cm^{-1} Anal: Calc: C 25.52; H 5.71%. Found: C 25.23; H 5.85%.

Refinement

The water H atoms were located in a difference map and their positions were freely refined with the constraint U_{iso}(H) = 1.2U_{eq}(O). The C-bound H atoms were placed in calculated positions (C—H = 0.98–0.99 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate, but not to tip, to best fit the electron density.

Figures

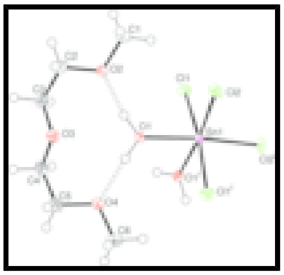

Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids. The H atoms are drawn as spheres of arbitrary radius and the hydrogen bonds are shown as double-dashed lines. Symmetry code: (i) -*x*, *y*, 3/2 - *z*.

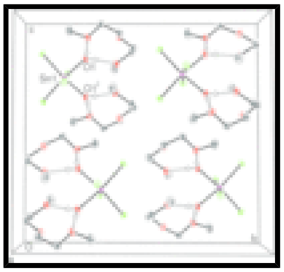

Fig. 2. Unit cell packing for (I) showing the isolated hydrogen bonded assembiles of one metal complex and two diglyme molecules. Symmetry code: (i) -*x*, *y*, 3/2 - *z*. The C-bound H atoms are omitted for clarity.

Diaquatetrachloridotin(IV)–diglyme (1/2)

Crystal data

$[SnCl_4(H_2O)_2]\cdot 2C_6H_{14}O_3$	F_{000} = 1144
M_r = 564.86	D_x = 1.631 Mg m^{-3}
Orthorhombic, *Pbcn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2n 2ab	Cell parameters from 10481 reflections
a = 8.4023 (2) Å	θ = 2.9–27.5º
b = 17.1528 (3) Å	μ = 1.61 mm^{-1}
c = 15.9612 (4) Å	*T* = 120 (2) K
V = 2300.38 (9) Å^3	Slab, colourless
Z = 4	0.55 × 0.43 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer	2643 independent reflections
Radiation source: fine-focus sealed tube	2292 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 120(2) K	θ_{max} = 27.6º
ω and φ scans	θ_{min} = 3.5º
Absorption correction: multi-scan (SADABS; Bruker, 2003)	*h* = −10→10
T_{min} = 0.472, T_{max} = 0.795	*k* = −19→22
20197 measured reflections	*l* = −17→20

Refinement

Refinement on F^2	Hydrogen site location: difmap and geom
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.022$	$w = 1/[\sigma^2(F_o^2) + (0.0206P)^2 + 1.2513P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.050$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.56$ e Å^{-3}
2643 reflections	$\Delta\rho_{min} = -0.50$ e Å^{-3}
121 parameters	Extinction correction: (SHELXL97; Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00095 (17)
Secondary atom site location: difference Fourier map	

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}
Sn1	0.0000	0.165876 (9)	0.7500	0.01280 (7)
Cl1	0.27717 (5)	0.17456 (3)	0.72161 (3)	0.02257 (11)
Cl2	0.04156 (6)	0.07342 (3)	0.85946 (3)	0.02251 (11)
O1	0.02883 (16)	0.25927 (8)	0.83704 (9)	0.0201 (3)
H1	−0.023 (2)	0.2999 (14)	0.8351 (14)	0.024*
H2	0.107 (3)	0.2655 (12)	0.8617 (14)	0.024*
C1	0.3671 (3)	0.19896 (12)	0.94703 (16)	0.0380 (6)
H1A	0.3444	0.1587	0.9051	0.057*
H1B	0.3199	0.1839	1.0008	0.057*
H1C	0.4826	0.2045	0.9535	0.057*
C2	0.3303 (2)	0.33246 (10)	0.97905 (12)	0.0211 (4)
H2A	0.4454	0.3351	0.9920	0.025*
H2B	0.2720	0.3220	1.0318	0.025*
C3	0.2759 (2)	0.40787 (10)	0.94202 (13)	0.0228 (4)
H3A	0.3058	0.4516	0.9793	0.027*
H3B	0.3273	0.4161	0.8869	0.027*

C4	0.0482 (2)	0.46917 (10)	0.88487 (13)	0.0254 (4)
H4A	0.1021	0.4714	0.8298	0.030*
H4B	0.0690	0.5187	0.9148	0.030*
C5	−0.1273 (2)	0.45811 (11)	0.87301 (13)	0.0257 (4)
H5A	−0.1798	0.4520	0.9281	0.031*
H5B	−0.1733	0.5044	0.8449	0.031*
C6	−0.3192 (2)	0.38022 (12)	0.80483 (15)	0.0327 (5)
H6A	−0.3336	0.3337	0.7700	0.049*
H6B	−0.3587	0.4261	0.7747	0.049*
H6C	−0.3786	0.3740	0.8573	0.049*
O2	0.30068 (16)	0.27173 (7)	0.92018 (9)	0.0260 (3)
O3	0.10705 (14)	0.40525 (7)	0.93230 (8)	0.0210 (3)
O4	−0.15401 (14)	0.39017 (7)	0.82313 (8)	0.0226 (3)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01400 (9)	0.00920 (10)	0.01519 (11)	0.000	−0.00062 (6)	0.000
Cl1	0.0149 (2)	0.0241 (2)	0.0287 (3)	0.00062 (16)	0.00141 (18)	−0.00129 (19)
Cl2	0.0303 (2)	0.0155 (2)	0.0217 (3)	0.00401 (17)	−0.00013 (18)	0.00507 (18)
O1	0.0210 (7)	0.0128 (6)	0.0265 (8)	0.0060 (5)	−0.0101 (6)	−0.0061 (6)
C1	0.0429 (13)	0.0233 (11)	0.0477 (15)	0.0116 (9)	−0.0239 (11)	−0.0025 (10)
C2	0.0193 (8)	0.0252 (10)	0.0187 (11)	−0.0034 (7)	−0.0042 (7)	−0.0030 (7)
C3	0.0215 (9)	0.0206 (9)	0.0263 (12)	−0.0066 (7)	−0.0017 (8)	−0.0031 (8)
C4	0.0341 (10)	0.0129 (9)	0.0291 (12)	−0.0003 (7)	−0.0054 (9)	0.0018 (8)
C5	0.0334 (11)	0.0165 (9)	0.0273 (12)	0.0086 (7)	−0.0029 (8)	−0.0030 (8)
C6	0.0214 (10)	0.0325 (11)	0.0441 (14)	0.0059 (8)	−0.0066 (9)	−0.0025 (10)
O2	0.0300 (7)	0.0179 (6)	0.0300 (8)	0.0067 (5)	−0.0164 (6)	−0.0047 (6)
O3	0.0199 (6)	0.0170 (6)	0.0260 (8)	−0.0009 (5)	−0.0016 (5)	0.0034 (5)
O4	0.0196 (6)	0.0183 (6)	0.0299 (8)	0.0052 (5)	−0.0034 (5)	−0.0035 (6)

Geometric parameters (Å, °)

Sn1—O1	2.1343 (13)	C2—H2B	0.9900
Sn1—O1^{i}	2.1343 (13)	C3—O3	1.428 (2)
Sn1—Cl1	2.3772 (4)	C3—H3A	0.9900
Sn1—Cl1^{i}	2.3772 (4)	C3—H3B	0.9900
Sn1—Cl2^{i}	2.3853 (5)	C4—O3	1.421 (2)
Sn1—Cl2	2.3853 (5)	C4—C5	1.499 (3)
O1—H1	0.82 (2)	C4—H4A	0.9900
O1—H2	0.77 (2)	C4—H4B	0.9900
C1—O2	1.433 (2)	C5—O4	1.429 (2)
C1—H1A	0.9800	C5—H5A	0.9900
C1—H1B	0.9800	C5—H5B	0.9900
C1—H1C	0.9800	C6—O4	1.429 (2)
C2—O2	1.425 (2)	C6—H6A	0.9800
C2—C3	1.494 (2)	C6—H6B	0.9800
C2—H2A	0.9900	C6—H6C	0.9800

O1—Sn1—O1^{i}	82.72 (8)	H2A—C2—H2B	108.4
O1—Sn1—Cl1	88.04 (4)	O3—C3—C2	108.66 (14)
O1^{i}—Sn1—Cl1	86.57 (4)	O3—C3—H3A	110.0
O1—Sn1—Cl1^{i}	86.57 (4)	C2—C3—H3A	110.0
O1^{i}—Sn1—Cl1^{i}	88.04 (4)	O3—C3—H3B	110.0
Cl1—Sn1—Cl1^{i}	172.81 (2)	C2—C3—H3B	110.0
O1—Sn1—Cl2^{i}	172.96 (4)	H3A—C3—H3B	108.3
O1^{i}—Sn1—Cl2^{i}	90.32 (4)	O3—C4—C5	108.18 (15)
Cl1—Sn1—Cl2^{i}	92.608 (16)	O3—C4—H4A	110.1
Cl1^{i}—Sn1—Cl2^{i}	92.169 (17)	C5—C4—H4A	110.1
O1—Sn1—Cl2	90.32 (4)	O3—C4—H4B	110.1
O1^{i}—Sn1—Cl2	172.96 (4)	C5—C4—H4B	110.1
Cl1—Sn1—Cl2	92.169 (16)	H4A—C4—H4B	108.4
Cl1^{i}—Sn1—Cl2	92.608 (16)	O4—C5—C4	109.14 (14)
Cl2^{i}—Sn1—Cl2	96.65 (2)	O4—C5—H5A	109.9
Sn1—O1—H1	123.4 (16)	C4—C5—H5A	109.9
Sn1—O1—H2	122.1 (16)	O4—C5—H5B	109.9
H1—O1—H2	111 (2)	C4—C5—H5B	109.9
O2—C1—H1A	109.5	H5A—C5—H5B	108.3
O2—C1—H1B	109.5	O4—C6—H6A	109.5
H1A—C1—H1B	109.5	O4—C6—H6B	109.5
O2—C1—H1C	109.5	H6A—C6—H6B	109.5
H1A—C1—H1C	109.5	O4—C6—H6C	109.5
H1B—C1—H1C	109.5	H6A—C6—H6C	109.5
O2—C2—C3	108.58 (15)	H6B—C6—H6C	109.5
O2—C2—H2A	110.0	C2—O2—C1	111.81 (14)
C3—C2—H2A	110.0	C4—O3—C3	112.30 (13)
O2—C2—H2B	110.0	C6—O4—C5	111.33 (14)
C3—C2—H2B	110.0		
O2—C2—C3—O3	65.6 (2)	C5—C4—O3—C3	176.00 (15)
O3—C4—C5—O4	−64.9 (2)	C2—C3—O3—C4	−170.23 (16)
C3—C2—O2—C1	172.98 (17)	C4—C5—O4—C6	−175.75 (16)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H2···O2	0.77 (2)	1.88 (2)	2.6503 (18)	175 (2)
O1—H1···O4	0.82 (2)	1.91 (2)	2.7296 (18)	175 (2)

Fig. 1

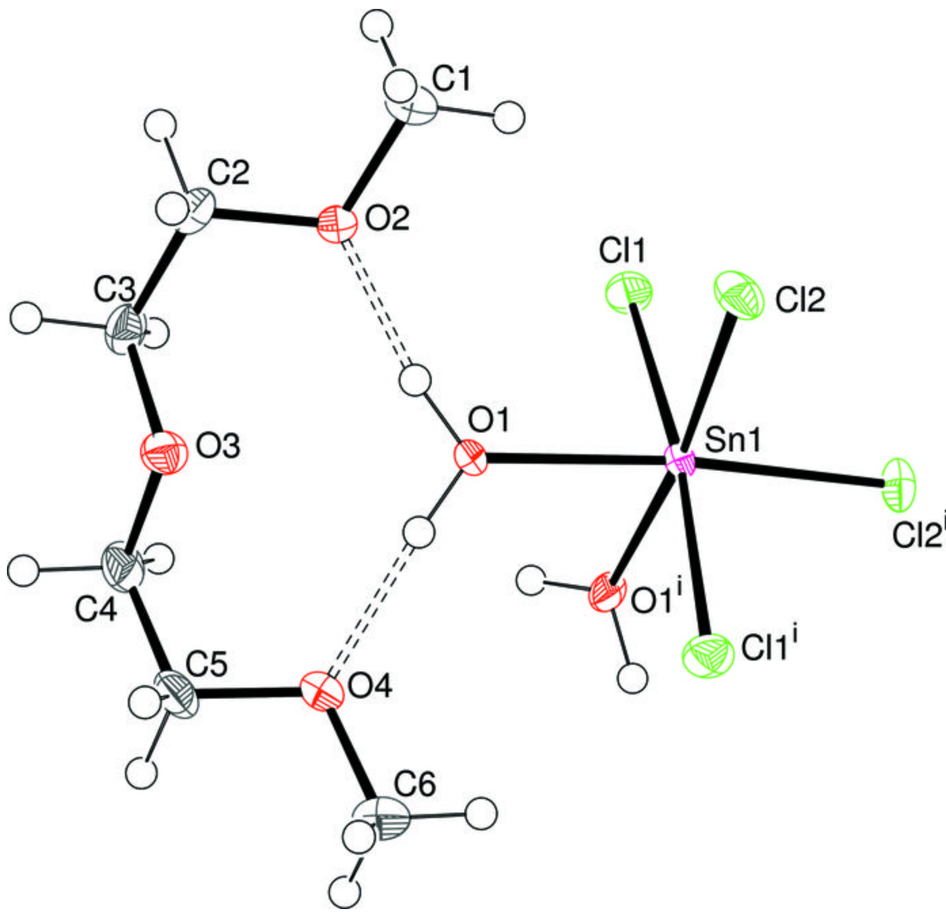

Fig. 2