metal-organic compounds

Di-μ-hydroxido-bis[aquatrichlorido-tin(IV)] benzene hemisolvate mono-hydrate

Manuela Müller, Hans-Wolfram Lerner and Michael Bolte*

Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

In the title compound, $[Sn_2Cl_6(OH)_2(H_2O)_2]\cdot0.5C_6H_6\cdot H_2O$, the benzene molecule is located on a centre of inversion. The Sn atoms are in an octahedral environment, each coordinated by three Cl, one water and two briding hydroxide ligands. Two Cl atoms and the hydroxide groups are in the equatorial plane, whereas the third Cl atom and the water molecules are located in the axial positions.

Related literature

For related literature, see: Lerner *et al.* (2005); Ruth *et al.* (2005).
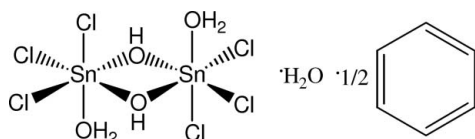
Experimental

Crystal data

$[Sn_2Cl_6(OH)_2(H_2O)_2]\cdot0.5C_6H_6\cdot H_2O$

M_r = 577.20

Triclinic, $P\bar{1}$

a = 6.3682 (7) Å

b = 10.8299 (12) Å

c = 11.8619 (13) Å

α = 74.180 (9)°

β = 78.048 (9)°

γ = 88.588 (9)°

V = 769.59 (15) Å^3

Z = 2

Mo $K\alpha$ radiation

μ = 4.29 mm^{-1}

T = 173 (2) K

$0.22 \times 0.21 \times 0.19$ mm

Data collection

Stoe IPDSII two-circle diffractometer

Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)

T_{min} = 0.452, T_{max} = 0.497 (expected range = 0.404–0.443)

11598 measured reflections

2887 independent reflections

2382 reflections with $I > 2\sigma(I)$

R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040

$wR(F^2)$ = 0.107

S = 1.04

2887 reflections

145 parameters

H-atom parameters constrained

$\Delta\rho_{max}$ = 1.07 e Å^{-3}

$\Delta\rho_{min}$ = −1.56 e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5A⋯O2	0.84	1.88	2.719 (6)	180
O5—H5B⋯O4^i	0.84	1.97	2.813 (6)	180

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2421).

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Lerner, H.-W., Haghiri Ilkechi, A., Bolte, M. & Wagner, M. (2005). *Z. Naturforsch. Teil B*, **60**, 413–415.

Ruth, K., Lerner, H.-W. & Bolte, M. (2005). *Acta Cryst.* E**61**, m1852–m1853.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXL97.* University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Stoe & Cie (2001). *X-AREA.* Stoe & Cie, Darmstadt, Germany.

supplementary materials

Di-*μ*-hydroxido-bis[aquatrichloridotin(IV)] benzene hemisolvate monohydrate

M. Müller, H.-W. Lerner and M. Bolte

Comment

Recently we have reported the synthesis and structure of the SnCl$_4$ complex with dimethylsulfide (Ruth *et al.*, 2005) and of the adduct of Me$_3$SnCl with Me$_3$SnOH and H$_2$O (Lerner *et al.*, 2005) (Me = CH$_3$). The structure of Me$_3$SnCl·Me$_3$SnOH·H$_2$O features an array of Me$_3$Sn units connected alternately by bridging Cl and OH ligands (Lerner *et al.*, 2005) whereas the structure of the dimethylsulfide complex with tin tetrachloride consists of discrete [(SnCl$_4$)·(SMe$_2$)$_2$] molecules (Ruth *et al.*, 2005). It is interesting to note that Me$_3$SnCl·Me$_3$SnOH·H$_2$O represents an intermediate in Me$_3$SnCl hydrolysis. Herein we report the X-ray crystal structure analysis of the HOSnCl$_3$ complex with water which features a dimer in solid state. The synthesis of [HOSnCl$_3$·H$_2$O]$_2$ was achieved by treatment of PhSnCl$_3$ with an aqueous HCl solution as indicated in the equation below.

The asymmetric unit of the title compound contains one bis(μ-Hydroxo)-bis(aqua-trichloro-tin(iv)) molecule, a water molecule and half a benzene molecule. The latter is located on a centre of inversion. The Sn atoms are in an octahedral environment each coordinated by three Cl and one water and two briding hydroxyl ligands. Two Cl atom and the hydroxyl groups are in the equatorial plane, whereas the third Cl atom and the water molecules are located in the axial positions.

Experimental

A solution of aqueous HCl (38%) was added at ambient temperature to a solution PhSnCl$_3$ (50 mg) in 0.5 ml C$_6$D$_6$. Colourless crystals of the title compound were grown by storing this solution at ambient temperature for several days.

Refinement

H atoms bonded to C and those of the solvent water molecule were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2 U_{eq}(C,O)] using a riding model with C—H = 0.93Å and O—H = 0.88 Å. The H atoms of the hydroxyl O atoms and water O atoms bonded to Sn could not be located and were omitted from refinement.
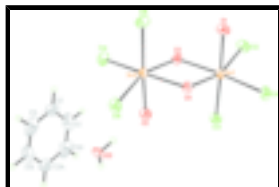
Figures

Fig. 1. Perspective view of the title compound with the atom numbering scheme. Displacement ellipsoids are at the 50% probability level. H atoms are drawn as small spheres of arbitrary radii. The atoms with suffix A have been generated by the symmetry operator −*x*, −*y* + 1, −*z* + 2.
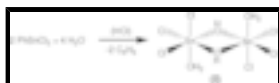

Fig. 2. The formation of the title compound.

Di-µ-hydroxido-bis[aquatrichloridotin(IV)] benzene hemisolvate monohydrate

Crystal data

$[Sn_2Cl_6(OH)_2(H_2O)_2]\cdot0.5C_6H_6\cdot H_2O$	$Z = 2$
$M_r = 577.20$	$F_{000} = 542$
Triclinic, $P\bar{1}$	$D_x = 2.491$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.3682$ (7) Å	Cell parameters from 10632 reflections
$b = 10.8299$ (12) Å	$\theta = 3.1–25.7°$
$c = 11.8619$ (13) Å	$\mu = 4.29$ mm^{-1}
$\alpha = 74.180$ (9)°	$T = 173$ (2) K
$\beta = 78.048$ (9)°	Block, colourless
$\gamma = 88.588$ (9)°	$0.22 \times 0.21 \times 0.19$ mm
$V = 769.59$ (15) Å^3	

Data collection

Stoe IPDSII two-circle diffractometer	2887 independent reflections
Radiation source: fine-focus sealed tube	2382 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.079$
$T = 173(2)$ K	$\theta_{max} = 25.6°$
ω scans	$\theta_{min} = 3.0°$
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	$h = -7 \rightarrow 7$
$T_{min} = 0.452$, $T_{max} = 0.497$	$k = -13 \rightarrow 13$
11598 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2 + 0.5227P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
2887 reflections	$\Delta\rho_{max} = 1.07$ e Å^{-3}
145 parameters	$\Delta\rho_{min} = -1.56$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.18222 (6)	0.78127 (4)	0.63209 (3)	0.01735 (15)
Sn2	0.01184 (6)	0.47034 (4)	0.75109 (3)	0.01833 (15)
Cl1	0.0470 (3)	0.96136 (15)	0.69735 (14)	0.0272 (4)
Cl2	0.3898 (3)	0.87397 (17)	0.44209 (14)	0.0298 (4)
Cl3	0.4706 (2)	0.75927 (16)	0.73589 (14)	0.0248 (3)
Cl4	0.1403 (3)	0.28184 (17)	0.70417 (16)	0.0337 (4)
Cl5	−0.2737 (3)	0.48558 (17)	0.64389 (14)	0.0270 (4)
Cl6	−0.2008 (3)	0.38714 (17)	0.94412 (14)	0.0302 (4)
O1	−0.0872 (7)	0.7947 (5)	0.5432 (4)	0.0260 (10)
O2	0.2802 (7)	0.4640 (5)	0.8379 (4)	0.0257 (10)
O3	0.2191 (7)	0.5961 (4)	0.6082 (4)	0.0208 (9)
O4	−0.0274 (7)	0.6559 (4)	0.7707 (4)	0.0190 (9)
O5	0.2830 (8)	0.2681 (5)	1.0377 (4)	0.0278 (11)
H5A	0.2825	0.3284	0.9758	0.033*
H5B	0.2068	0.2905	1.0950	0.033*
C1	0.6070 (17)	0.0702 (8)	0.8914 (8)	0.053 (3)
H1	0.6824	0.1211	0.8159	0.063*
C2	0.4107 (18)	0.0216 (8)	0.9002 (9)	0.050 (2)
H2	0.3496	0.0348	0.8315	0.061*
C3	0.2988 (14)	−0.0484 (9)	1.0120 (11)	0.054 (3)
H3	0.1573	−0.0809	1.0210	0.065*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0166 (2)	0.0156 (2)	0.0180 (2)	0.00007 (16)	−0.00268 (16)	−0.00222 (17)
Sn2	0.0197 (2)	0.0152 (2)	0.0189 (2)	−0.00060 (16)	−0.00191 (17)	−0.00422 (17)
Cl1	0.0309 (8)	0.0194 (8)	0.0307 (8)	0.0043 (6)	−0.0049 (7)	−0.0075 (6)
Cl2	0.0268 (8)	0.0334 (9)	0.0215 (7)	−0.0035 (7)	0.0020 (6)	0.0001 (7)
Cl3	0.0207 (8)	0.0288 (8)	0.0256 (7)	0.0002 (6)	−0.0085 (6)	−0.0061 (6)
Cl4	0.0428 (10)	0.0222 (9)	0.0397 (9)	0.0073 (7)	−0.0092 (8)	−0.0145 (7)
Cl5	0.0219 (8)	0.0339 (9)	0.0287 (8)	0.0006 (6)	−0.0057 (6)	−0.0139 (7)

Cl6	0.0320 (9)	0.0292 (9)	0.0229 (7)	−0.0082 (7)	0.0043 (6)	−0.0033 (7)
O1	0.024 (2)	0.031 (3)	0.024 (2)	0.0016 (19)	−0.0092 (19)	−0.005 (2)
O2	0.024 (2)	0.028 (3)	0.025 (2)	0.0018 (19)	−0.0066 (19)	−0.006 (2)
O3	0.022 (2)	0.019 (2)	0.021 (2)	−0.0006 (17)	0.0006 (17)	−0.0085 (17)
O4	0.019 (2)	0.016 (2)	0.021 (2)	0.0000 (17)	−0.0017 (17)	−0.0039 (17)
O5	0.034 (3)	0.029 (3)	0.017 (2)	0.007 (2)	−0.0055 (19)	−0.0013 (19)
C1	0.074 (7)	0.029 (4)	0.041 (5)	0.000 (4)	0.021 (5)	−0.011 (4)
C2	0.080 (7)	0.031 (5)	0.056 (5)	0.018 (4)	−0.045 (5)	−0.016 (4)
C3	0.030 (4)	0.039 (5)	0.100 (8)	0.004 (4)	−0.021 (5)	−0.025 (5)

Geometric parameters (Å, °)

Sn1—O4	2.072 (4)	Sn2—Cl5	2.4038 (17)
Sn1—O3	2.102 (4)	O5—H5A	0.8398
Sn1—O1	2.175 (5)	O5—H5B	0.8391
Sn1—Cl2	2.3307 (16)	C1—C2	1.341 (14)
Sn1—Cl1	2.3685 (16)	C1—C3^i	1.364 (15)
Sn1—Cl3	2.3876 (16)	C1—H1	0.9500
Sn2—O4	2.088 (4)	C2—C3	1.388 (15)
Sn2—O3	2.091 (4)	C2—H2	0.9500
Sn2—O2	2.161 (5)	C3—C1^i	1.364 (15)
Sn2—Cl4	2.3441 (17)	C3—H3	0.9500
Sn2—Cl6	2.3537 (16)		
O4—Sn1—O3	71.71 (16)	O4—Sn2—Cl6	89.85 (12)
O4—Sn1—O1	82.67 (17)	O3—Sn2—Cl6	159.25 (13)
O3—Sn1—O1	86.18 (18)	O2—Sn2—Cl6	86.68 (13)
O4—Sn1—Cl2	160.56 (13)	Cl4—Sn2—Cl6	101.36 (7)
O3—Sn1—Cl2	91.84 (12)	O4—Sn2—Cl5	93.53 (13)
O1—Sn1—Cl2	86.04 (13)	O3—Sn2—Cl5	93.81 (13)
O4—Sn1—Cl1	92.10 (12)	O2—Sn2—Cl5	176.57 (13)
O3—Sn1—Cl1	163.20 (12)	Cl4—Sn2—Cl5	93.52 (6)
O1—Sn1—Cl1	87.29 (14)	Cl6—Sn2—Cl5	96.63 (6)
Cl2—Sn1—Cl1	103.15 (6)	Sn2—O3—Sn1	107.71 (18)
O4—Sn1—Cl3	95.04 (13)	Sn1—O4—Sn2	108.94 (18)
O3—Sn1—Cl3	92.68 (13)	H5A—O5—H5B	106.6
O1—Sn1—Cl3	177.66 (13)	C2—C1—C3^i	121.6 (9)
Cl2—Sn1—Cl3	96.05 (6)	C2—C1—H1	119.2
Cl1—Sn1—Cl3	93.25 (6)	C3^i—C1—H1	119.2
O4—Sn2—O3	71.60 (16)	C1—C2—C3	118.6 (8)
O4—Sn2—O2	85.57 (18)	C1—C2—H2	120.7
O3—Sn2—O2	82.77 (18)	C3—C2—H2	120.7
O4—Sn2—Cl4	165.97 (13)	C1^i—C3—C2	119.7 (8)
O3—Sn2—Cl4	95.83 (13)	C1^i—C3—H3	120.2
O2—Sn2—Cl4	86.68 (14)	C2—C3—H3	120.2
O4—Sn2—O3—Sn1	1.34 (18)	O1—Sn1—O4—Sn2	89.7 (2)
O2—Sn2—O3—Sn1	−86.4 (2)	Cl2—Sn1—O4—Sn2	34.8 (5)
Cl4—Sn2—O3—Sn1	−172.27 (16)	Cl1—Sn1—O4—Sn2	176.74 (17)

Cl6—Sn2—O3—Sn1	−26.3 (5)	Cl3—Sn1—O4—Sn2	−89.80 (17)
Cl5—Sn2—O3—Sn1	93.79 (17)	O3—Sn2—O4—Sn1	−1.36 (18)
O4—Sn1—O3—Sn2	−1.35 (18)	O2—Sn2—O4—Sn1	82.5 (2)
O1—Sn1—O3—Sn2	−84.9 (2)	Cl4—Sn2—O4—Sn1	25.8 (7)
Cl2—Sn1—O3—Sn2	−170.78 (17)	Cl6—Sn2—O4—Sn1	169.16 (17)
Cl1—Sn1—O3—Sn2	−17.5 (6)	Cl5—Sn2—O4—Sn1	−94.21 (17)
Cl3—Sn1—O3—Sn2	93.08 (18)	C3^{i}—C1—C2—C3	2.5 (15)
O3—Sn1—O4—Sn2	1.36 (18)	C1—C2—C3—C1^{i}	−2.5 (15)

Symmetry codes: (i) −x+1, −y, −z+2.

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O5—H5A⋯O2	0.84	1.88	2.719 (6)	180
O5—H5B⋯O4^{ii}	0.84	1.97	2.813 (6)	180

Symmetry codes: (ii) −x, −y+1, −z+2.

Fig. 1

Fig. 2

$$2\ PhSnCl_3 + 4\ H_2O \xrightarrow[-2\ C_6H_6]{(HCl)}$$

(I)