Absorption correction:
  Gaussian
  T_{min} = 0.58, T_{max} = 0.87
2009 measured reflections
1917 independent reflections
1537 observed reflections
  [$I > 2\sigma(I)$]

$h = -9 \rightarrow 10$
$k = 0 \rightarrow 14$
$l = -9 \rightarrow 12$
3 standard reflections
  monitored every 200
  reflections
  intensity variation: 10%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0527$
$wR(F^2) = 0.1497$
$S = 1.068$
1917 reflections
123 parameters
Calculated weights
  $w = 1/[\sigma^2(F_o^2) + (0.0967P)^2 + 2.2710P]$
  where $P = (F_o^2 + 2F_c^2)/3$

$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −2.01 e Å^{-3}
Extinction correction: none
Atomic scattering factors
  from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8, 6.1.1.4)

Methyl H atoms were positioned from stereochemical considerations; a single orientation parameter was refined for each Me group as was an isotropic U [0.065 (7) Å^2] common to all methyl H atoms. Data collection: Siemens *R3m* system. Cell refinement: Siemens *R3m* system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS90* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Zr1	0.03818 (7)	0.05068 (5)	0.35310 (5)	0.0237 (2)
S1	−0.0959 (2)	−0.20149 (14)	0.2155 (2)	0.0338 (4)
S2	0.1519 (2)	0.31475 (13)	0.3198 (2)	0.0326 (4)
F1	−0.0185 (5)	−0.0903 (3)	0.4586 (4)	0.0410 (10)
F2	0.0812 (6)	0.1088 (3)	0.1940 (4)	0.0463 (11)
F3	−0.1949 (5)	0.0742 (4)	0.2786 (5)	0.0541 (12)
F4	0.2662 (5)	0.0166 (4)	0.4337 (5)	0.0536 (12)
O1	0.0143 (6)	−0.0988 (4)	0.2276 (5)	0.0385 (11)
O2	0.0766 (7)	0.2321 (4)	0.3933 (5)	0.0434 (12)
C11	0.0163 (9)	−0.3047 (6)	0.3232 (7)	0.041 (2)
C12	−0.0905 (10)	−0.2651 (7)	0.0682 (7)	0.051 (2)
C21	0.2215 (10)	0.4252 (6)	0.4344 (8)	0.047 (2)
C22	−0.0108 (10)	0.3831 (7)	0.2059 (8)	0.051 (2)

Table 2. *Selected geometric parameters* (Å, °)

Zr1—F4	1.973 (4)	S1—O1	1.546 (5)
Zr1—F3	1.983 (4)	S1—C11	1.774 (8)
Zr1—F2	2.001 (4)	S1—C12	1.781 (7)
Zr1—F1^i	2.153 (4)	S2—O2	1.544 (5)
Zr1—F1	2.180 (4)	S2—C22	1.776 (8)
Zr1—O1	2.216 (5)	S2—C21	1.785 (7)
Zr1—O2	2.217 (5)	F1—Zr1^i	2.153 (4)
F4—Zr1—F3	175.5 (2)	F4—Zr1—O2	92.4 (2)
F4—Zr1—F2	90.7 (2)	F3—Zr1—O2	91.0 (2)
F3—Zr1—F2	92.9 (2)	F2—Zr1—O2	76.8 (2)
F4—Zr1—F1^i	89.9 (2)	F1^i—Zr1—O2	69.6 (2)
F3—Zr1—F1^i	88.6 (2)	F1—Zr1—O2	134.3 (2)
F2—Zr1—F1^i	146.3 (2)	O1—Zr1—O2	152.8 (2)
F4—Zr1—F1	88.7 (2)	O1—S1—C11	106.7 (3)
F3—Zr1—F1	86.8 (2)	O1—S1—C12	102.9 (3)
F2—Zr1—F1	148.9 (2)	C11—S1—C12	97.3 (4)
F1^i—Zr1—F1	64.8 (2)	O2—S2—C22	105.5 (4)
F4—Zr1—O1	89.7 (2)	O2—S2—C21	103.0 (3)
F3—Zr1—O1	88.6 (2)	C22—S2—C21	99.9 (4)
F2—Zr1—O1	76.0 (2)	Zr1^i—F1—Zr1	115.2 (2)
F1^i—Zr1—O1	137.6 (2)	S1—O1—Zr1	129.0 (3)
F1—Zr1—O1	72.8 (2)	S2—O2—Zr1	126.1 (3)

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

The complex was prepared by refluxing ZrF_4 in dimethyl sulfoxide (Muetterties, 1960) and allowed to crystallize upon cooling. The complex decomposes slowly in air and therefore the data were collected with the crystal mounted in a Lindemann tube; intensities were corrected for crystal decomposition, indicated by a 10% reduction in the intensities of the standard reflections in the course of the experiment.

We thank the SERC for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71530 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1073]

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Dimeric μ-Hydroxy-diphenyl-(trichloroacetato)tin

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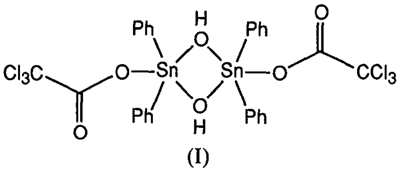
Abstract

The title compound, di-μ-hydroxy-bis[diphenyl(trichloroacetato-*O*)tin], $[Sn(C_2Cl_3O_2)(C_6H_5)_2(OH)]_2$, is a centrosymmetric dimer, with distorted trigonal-bipyramidally coordinated Sn atoms bridged by OH groups. The latter are hydrogen bonded to the unidentate carboxylates.

Comment

During an investigation of tin carboxylates (Alcock & Roe, 1989), the title compound (I) was obtained by a dearylation reaction coupled with a partial hydrolysis that occurred during recrystallization of the parent triphenyltin carboxylate in a non-polar solvent (*cf.* Ford & Sams, 1978).



(I)

The compound is a centrosymmetric dimer (Fig. 1), with hydroxy bridges forming a planar four-centre ring. The coordination geometry of the Sn atom is approximately trigonal bipyramidal; the principal distortion arises from the small O(11)—Sn(1)—O(11^i) angle [70.6 (2)°] in the four-membered ring, which is coupled with an increase in the C—Sn—C angle from 120 to 133.9 (2)° and a decrease in the O(1)—Sn(1)—O(11) axial angle to 158.3 (2)°. These distortions are significantly greater than in the tin trichloroacetates already reported (Alcock & Roe, 1989), though the bonds to Sn are normal in length. The hydroxy bridges are almost symmetrical, the difference in μ-Sn—O bond lengths being 0.138 Å. The acyl O atom of the carboxylate is hydrogen bonded to the μ-OH [O(2)···H(11) 1.95 (7) Å, O(11)···O(2) 2.64 (1) Å] to form a six-membered ring. The difference in the C—O bond lengths in the carboxylate is small, only 0.028 (9) Å, indicating delocalization of the double bond.

Several other $R_2Sn(OH)(X)$ structures have been determined, all of which crystallize as hydroxy-bridged dimers (Domingos & Sheldrick, 1974; Lecompte, Protas & Devaud, 1976; Barnes, Sampson & Weakly, 1980; Puff, Hevendehl, Hofer, Reuter & Schuh, 1985). $[Me_2Sn(OH)(NO_3)]_2$, the only one to contain a potentially bidentate ligand in addition to the hydroxide, does not form hydrogen bonds from the ligand to the hydroxide, unlike the title compound (Domingos & Sheldrick, 1974).

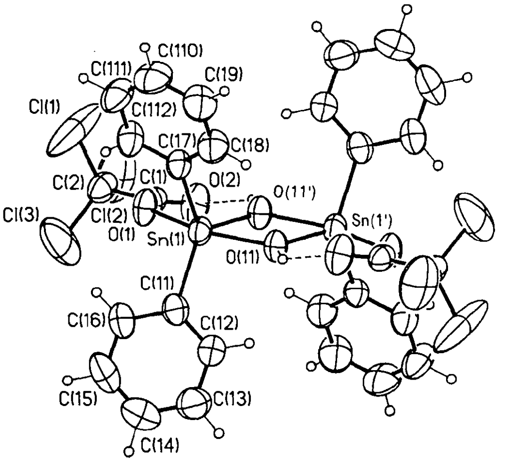


Fig. 1. View of the molecule showing the atomic numbering scheme; 50% probability ellipsoids are shown except for the H atoms which are represented by spheres of arbitrary size.

Experimental

Crystal data

$[Sn_2(C_2Cl_3O_2)_2(C_6H_5)_4(OH)_2]$
$M_r = 904.6$
Monoclinic
$P2_1/c$
$a = 10.429$ (2) Å
$b = 8.472$ (2) Å
$c = 19.228$ (5) Å
$\beta = 99.96$ (2)°
$V = 1673.3$ (7) Å^3
$Z = 2$

$D_x = 1.80$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 15 reflections
$\theta = 10$–11°
$\mu = 2.01$ mm^{-1}
$T = 293$ K
Blocks
0.43 × 0.35 × 0.27 mm
Colourless

Data collection

Siemens *R3m* diffractometer
ω-2θ scans
Absorption correction: Gaussian
$T_{min} = 0.59$, $T_{max} = 0.70$
3355 measured reflections
2959 independent reflections
2314 observed reflections [$I \geq 2\sigma(I)$]

$R_{int} = 0.028$
$\theta_{max} = 25°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 11$
$l = -23 \rightarrow 23$
3 standard reflections monitored every 200 reflections intensity variation: −2%

Refinement

Refinement on F
$R = 0.046$
$wR = 0.062$
$S = 1.21$
2314 reflections
193 parameters
$w = 1/[\sigma^2(F) + 0.0017F^2]$

$(\Delta/\sigma)_{max} = 0.02$
$\Delta\rho_{max} = 0.9$ e Å^{-3}
$\Delta\rho_{min} = -0.8$ e Å^{-3}
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1986)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sn(1)	0.43788 (4)	0.12624 (5)	0.05500 (2)	0.048 (1)
Cl(1)	−0.0219 (3)	0.1206 (4)	0.0935 (3)	0.186 (2)
Cl(2)	−0.0125 (3)	−0.1985 (4)	0.1300 (2)	0.129 (1)
Cl(3)	0.1337 (3)	0.0151 (6)	0.2221 (1)	0.146 (2)
O(11)	0.5943 (4)	0.0815 (5)	−0.0030 (2)	0.053 (2)
O(1)	0.2602 (5)	0.0855 (6)	0.0953 (3)	0.063 (2)
O(2)	0.2188 (5)	−0.1679 (6)	0.0714 (3)	0.077 (2)
C(1)	0.1966 (6)	−0.0395 (8)	0.0956 (3)	0.050 (2)
C(2)	0.0784 (6)	−0.0251 (8)	0.1347 (4)	0.059 (2)
C(11)	0.5515 (6)	0.1057 (7)	0.1564 (3)	0.053 (2)
C(12)	0.6656 (7)	0.0209 (8)	0.1675 (4)	0.059 (2)
C(13)	0.7380 (8)	0.0099 (10)	0.2350 (4)	0.077 (3)
C(14)	0.6962 (9)	0.0842 (11)	0.2899 (4)	0.079 (3)
C(15)	0.5842 (10)	0.1699 (11)	0.2791 (4)	0.085 (4)

C(16)	0.5107 (8)	0.1798 (9)	0.2126 (4)	0.067 (3)
C(17)	0.3561 (6)	0.3190 (7)	−0.0071 (3)	0.049 (2)
C(18)	0.4080 (8)	0.3676 (8)	−0.0646 (4)	0.067 (3)
C(19)	0.3531 (9)	0.4893 (10)	−0.1056 (4)	0.080 (3)
C(110)	0.2478 (9)	0.5659 (10)	−0.0903 (5)	0.083 (3)
C(111)	0.1950 (8)	0.5235 (9)	−0.0316 (5)	0.081 (3)
C(112)	0.2494 (8)	0.3986 (8)	0.0099 (4)	0.066 (3)

Table 2. *Selected geometric parameters* (Å, °)

Sn(1)—O(11)	2.162 (5)	Sn(1)—O(1)	2.156 (5)
Sn(1)—C(11)	2.107 (6)	Sn(1)—C(17)	2.113 (6)
Sn(1)—O(11$^{\rm i}$)	2.024 (4)	Cl(1)—C(2)	1.720 (8)
Cl(2)—C(2)	1.742 (8)	Cl(3)—C(2)	1.714 (7)
O(1)—C(1)	1.250 (8)	O(2)—C(1)	1.222 (9)
C(1)—C(2)	1.556 (10)		
O(11)—Sn(1)—O(1)	158.3 (2)	O(11)—Sn(1)—C(11)	96.2 (2)
O(1)—Sn(1)—C(11)	92.0 (2)	O(11)—Sn(1)—C(17)	96.4 (2)
O(1)—Sn(1)—C(17)	92.2 (2)	C(11)—Sn(1)—C(17)	133.9 (2)
O(11)—Sn(1)—O(11$^{\rm i}$)	70.6 (2)	O(1)—Sn(1)—O(11$^{\rm i}$)	87.7 (2)
C(11)—Sn(1)—O(11$^{\rm i}$)	113.8 (2)	C(17)—Sn(1)—O(11$^{\rm i}$)	112.1 (2)
Sn(1)—O(11)—Sn(1$^{\rm i}$)	109.4 (2)	Sn(1)—O(1)—C(1)	128.9 (5)
O(1)—C(1)—O(2)	128.1 (6)	O(1)—C(1)—C(2)	113.7 (6)
O(2)—C(1)—C(2)	118.2 (6)	Cl(1)—C(2)—Cl(2)	107.4 (4)
Cl(1)—C(2)—Cl(3)	112.6 (5)	Cl(2)—C(2)—Cl(3)	107.8 (4)
Cl(1)—C(2)—C(1)	107.6 (5)	Cl(2)—C(2)—C(1)	112.3 (5)
Cl(3)—C(2)—C(1)	109.2 (4)		

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

Dimeric μ-hydroxy-(trichloroacetato)diphenyltin, $[SnPh_2(O_2CCCl_3)OH]_2$, was prepared from $Ph_3SnO_2CCCl_3.MeOH$ (Alcock & Roe, 1989) by recrystallization from CCl_4/hexane by the liquid diffusion method. Crystals formed at the interface after 12 h and were used directly for structure determination. The formation of this dimer, rather than the tetrameric bis-μ-oxo complex, $\{[SnPh_2(O_2CCCl_3)]_2\}_2$ [Alcock & Roe, 1989, compound (2)], is apparently favoured by the presence of traces of water in the (nominally dry) solvents interacting with the relatively strong acid.

Scan speed was 2-15°(ω) min^{-1}, depending on the intensity of a 2 s prescan; backgrounds were measured at each end of the scan for 0.25 of the scan time. The data were rescaled to correct for the slight crystal decay. Reflections were processed using profile analysis. Systematic reflection conditions $h0l$, $l = 2n$ and $0k0$, $k = 2n$, indicated space group $P2_1/c$. Heavy atoms were located by the Patterson interpretation section of *SHELXTL-Plus* (Sheldrick, 1986) and the light atoms were then found by successive Fourier syntheses. Anisotropic displacement parameters were used for all non-H atoms. The hydroxy H atom was refined, with an O—H constraint of 0.85 (1) Å. Other H atoms were inserted at calculated positions and not refined; all were given fixed isotropic temperature factors, $U = 0.07$ Å^2. Computations were performed with *SHELXTL-Plus* on a DEC MicroVAX II.

We thank the SERC for support.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71518 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1059]

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Tris(2-methoxyphenyl)tin Iodide

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Abstract

The Sn atom in the title compound, $[SnI(C_7H_7O)_3]$, has a distorted tetrahedral geometry. There are indications of only weak oxygen–tin intramolecular interactions.

Comment

Crystal structures have been reported for a number of intramolecular complexes of triorganotin halides possessing suitably-sited donor groups. These groups include alcohol and ether groups, *e.g.* as in $ClPh_2SnCH_2CH_2CH_2OEt$ (Forrester, Howie, Ross, Low & Wardell, 1991), $IPh_2SnCH_2CH_2CH_2OH$ (Forrester, Garden, Howie & Wardell, 1992), *cis*-1-$ClMe_2Sn$-3-$PhCH_2O$(cyclohexane) (Ochiai *et al.*, 1988) and 3-*C*-(IBu_2SnCH_2)-1,2;5,6-di-*O*-isopropylidene-α-D-allofuranose (1) (Cox *et al.*, 1989). The most frequently reported chelate-ring sizes are generally five- and six-membered but four-membered rings, as in (1), can also be obtained.

In contrast to the compounds listed above, the geometry about Sn in $(2\text{-}MeOC_6H_4)_3SnI$ (2) is essentially tetrahedral with I—Sn—C angles ranging from