Dichloridobis[2-(ethoxycarbonyl)ethyl-κ^2C,O]tin(IV)

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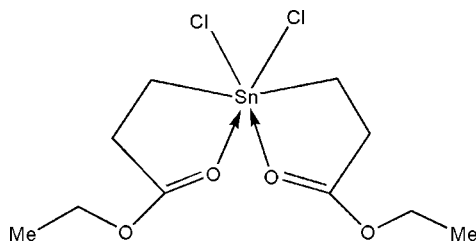
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The molecule of the title compound, $[SnCl_2(C_5H_9O_2)_2]$, has crystallographic twofold symmetry with the Sn atom lying on the rotation axis. The Sn atom is in a distorted $SnC_2Cl_2O_2$ octahedral geometry with two five-membered chelate rings formed by intramolecular Sn—O interactions [2.551 (2) Å]. The pendant ethyl group is disordered over two positions in a 0.695 (12):0.305 (12) ratio.

Related literature

For related structures, see: Balasubramanian *et al.* (1997); Harrison *et al.* (1979); Howie & Wardell (2002); Ng (1993). For background, see: Tian *et al.* (2005).

Experimental

Crystal data

$[SnCl_2(C_5H_9O_2)_2]$	V = 1544.9 (6) Å³
M_r = 391.83	Z = 4
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 20.251 (5) Å	μ = 2.00 mm⁻¹
b = 9.321 (2) Å	T = 295 (2) K
c = 9.053 (2) Å	0.22 × 0.20 × 0.09 mm
β = 115.304 (2)°	

Data collection

Bruker APEX CCD diffractometer	6017 measured reflections
Absorption correction: multi-scan	1597 independent reflections
(*SADABS*; Bruker, 2002)	1454 reflections with $I > 2\sigma(I)$
T_{min} = 0.667, T_{max} = 0.841	R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024	2 restraints
$wR(F^2)$ = 0.063	H-atom parameters constrained
S = 1.07	$\Delta\rho_{max}$ = 0.37 e Å⁻³
1597 reflections	$\Delta\rho_{min}$ = −0.30 e Å⁻³
85 parameters	

Table 1
Selected bond lengths (Å).

Sn1—C1	2.123 (3)	Sn1—O1	2.551 (2)
Sn1—Cl1	2.4062 (9)		

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2617).

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supplementary materials

Dichloridobis[2-(ethoxycarbonyl)ethyl-$\kappa^2 C,O$]tin(IV)

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Comment

Dichlorobis(3-alkoxy-3-oxopropyltins(IV), $Cl_2Sn(CH_2CH_2CO_2R)$, known as estertins, have received much attention because of their coordination chemistry and application in PVC stabilizers (Tian *et al.*, 2005). The structures of several estertin compounds, such as dichlorobis(3-methoxy-3-oxopropyltin (Harrison *et al.*, 1979; Ng, 1993), dihalobis(3-methoxy-3-oxopropyl)tin (Balasubramanian *et al.*, 1997) and diiodobis(3-ethoxy-3-oxopropyltin (Howie & Wardell, 2002), have been reported. We report herein the crystal structure of the title compound (Fig. 1).

The coordination geometry about the tin atom in (I) is a distorted octahedron (Fig. 1). The organic groups, $EtOCOCH_2CH_2$, act as C^4,O-chelating ligands, with the O atoms *trans* to the *cis*-chloride ions. The two carbon atoms of the ligands occupy *trans* positions. The chelate bite angles [72.36 (9)°] are slightly smaller than those [73.3 (2) and 74.0 (2)°] found in dichlorobis(3-methoxy-3-oxopropyltin (Harrison *et al.*, 1979). The Sn—O and Sn—Cl distances are similar to those [2.528 (2) and 2.4054 (9) Å, respectively] in dichlorobis(3-methoxy-3-oxopropyltin (Ng, 1993).

Experimental

The title compound was synthesized by the transesterification of $(MeOCOCH_2CH_2)_2SnCl_2$ (1.32 g, 5 mmol) with ethanol (30 ml) according to the reported procedure (Tian *et al.*, 2005). Colourless blocks of (I) were obtained from a solution of chloroform by slow evaporation at room temperature (yield 81%, m.p. 343–344 K).

Refinement

The ethyl group (C4 and C5) is disordered over two positions with site occupancy factors of 0.695 (12) and 0.305 (12). The C—C distances of the ethyl group were restrained to 1.53 (1) Å. H atoms were placed at calculated positions and were included in the refinement in the riding-model approximation, with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms, and C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene H atoms.
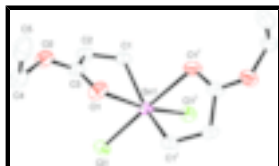
Figures

Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 30% probability level (H atoms omitted for clarity). For the ethyl group (C4 and C5), the minor disordered component has been omitted for clarity.

Dichloridobis[2-(ethoxycarbonyl)ethyl-$\kappa^2 C,O$]tin(IV)

Crystal data

[SnCl$_2$(C$_5$H$_9$O$_2$)$_2$]	F_{000} = 776
M_r = 391.83	D_x = 1.685 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 3340 reflections
a = 20.251 (5) Å	θ = 2.2–27.0º
b = 9.321 (2) Å	μ = 2.00 mm^{-1}
c = 9.053 (2) Å	T = 295 (2) K
β = 115.304 (2)º	Block, colourless
V = 1544.9 (6) Å^3	0.22 × 0.20 × 0.09 mm
Z = 4	

Data collection

Bruker APEX CCD diffractometer	1597 independent reflections
Radiation source: fine-focus sealed tube	1454 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 295(2) K	θ_{max} = 26.5º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -24 \rightarrow 25$
T_{min} = 0.667, T_{max} = 0.841	$k = -11 \rightarrow 11$
6017 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.024	H-atom parameters constrained
$wR(F^2)$ = 0.063	$w = 1/[\sigma^2(F_o^2) + (0.0293P)^2 + 0.6231P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$
1597 reflections	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
85 parameters	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	$U_{iso}*/U_{eq}$	Occ. (<1)
Sn1	0.0000	0.31014 (3)	0.7500	0.06105 (12)	
Cl1	0.09960 (5)	0.47778 (9)	0.82861 (10)	0.0824 (2)	
O1	0.09330 (14)	0.1118 (3)	0.7936 (2)	0.0886 (7)	
O2	0.15884 (13)	0.0386 (3)	0.6668 (3)	0.0882 (7)	
C1	−0.00940 (17)	0.2465 (3)	0.5166 (3)	0.0689 (7)	
H1A	−0.0457	0.1711	0.4741	0.083*	
H1B	−0.0263	0.3275	0.4424	0.083*	
C2	0.06158 (18)	0.1935 (3)	0.5220 (3)	0.0701 (8)	
H2A	0.0516	0.1325	0.4280	0.084*	
H2B	0.0899	0.2749	0.5146	0.084*	
C3	0.10580 (17)	0.1111 (3)	0.6745 (3)	0.0659 (7)	
C4	0.2002 (5)	−0.0523 (10)	0.8138 (9)	0.094 (2)	0.695 (12)
H4A	0.2361	0.0047	0.9007	0.113*	0.695 (12)
H4B	0.1672	−0.0953	0.8535	0.113*	0.695 (12)
C5	0.2365 (5)	−0.1643 (9)	0.7589 (11)	0.127 (3)	0.695 (12)
H5A	0.2607	−0.2311	0.8461	0.191*	0.695 (12)
H5B	0.2717	−0.1204	0.7282	0.191*	0.695 (12)
H5C	0.2006	−0.2140	0.6666	0.191*	0.695 (12)
C4'	0.2254 (11)	−0.0095 (19)	0.801 (3)	0.094 (2)	0.305 (12)
H4C	0.2312	0.0303	0.9048	0.113*	0.305 (12)
H4D	0.2684	0.0115	0.7831	0.113*	0.305 (12)
C5'	0.2082 (11)	−0.1677 (19)	0.788 (3)	0.127 (3)	0.305 (12)
H5D	0.2465	−0.2178	0.8753	0.191*	0.305 (12)
H5E	0.2044	−0.2032	0.6850	0.191*	0.305 (12)
H5F	0.1628	−0.1827	0.7950	0.191*	0.305 (12)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0721 (2)	0.04839 (17)	0.05327 (16)	0.000	0.01786 (13)	0.000
Cl1	0.0798 (5)	0.0725 (5)	0.0833 (5)	−0.0154 (4)	0.0236 (4)	−0.0075 (4)
O1	0.1139 (18)	0.0912 (16)	0.0615 (12)	0.0366 (14)	0.0382 (12)	0.0192 (11)

O2	0.0941 (16)	0.0971 (16)	0.0656 (12)	0.0289 (13)	0.0266 (11)	0.0012 (11)
C1	0.0797 (19)	0.0659 (16)	0.0494 (14)	0.0011 (15)	0.0164 (13)	0.0037 (13)
C2	0.089 (2)	0.0623 (17)	0.0550 (15)	−0.0001 (14)	0.0270 (15)	0.0033 (12)
C3	0.0779 (19)	0.0535 (15)	0.0566 (15)	0.0016 (13)	0.0195 (14)	−0.0026 (12)
C4	0.096 (6)	0.113 (6)	0.074 (3)	0.038 (4)	0.037 (3)	0.022 (3)
C5	0.106 (7)	0.154 (5)	0.127 (5)	0.064 (5)	0.056 (4)	0.036 (4)
C4'	0.096 (6)	0.113 (6)	0.074 (3)	0.038 (4)	0.037 (3)	0.022 (3)
C5'	0.106 (7)	0.154 (5)	0.127 (5)	0.064 (5)	0.056 (4)	0.036 (4)

Geometric parameters (Å, °)

Sn1—C1^i	2.123 (3)	C2—H2A	0.9700
Sn1—C1	2.123 (3)	C2—H2B	0.9700
Sn1—Cl1	2.4062 (9)	C4—C5	1.479 (7)
Sn1—Cl1^i	2.4062 (9)	C4—H4A	0.9700
Sn1—O1^i	2.551 (2)	C4—H4B	0.9700
Sn1—O1	2.551 (2)	C5—H5A	0.9600
O1—C3	1.208 (3)	C5—H5B	0.9600
O2—C3	1.296 (4)	C5—H5C	0.9600
O2—C4'	1.45 (2)	C4'—C5'	1.508 (10)
O2—C4	1.496 (8)	C4'—H4C	0.9700
C1—C2	1.501 (5)	C4'—H4D	0.9700
C1—H1A	0.9700	C5'—H5D	0.9600
C1—H1B	0.9700	C5'—H5E	0.9600
C2—C3	1.498 (4)	C5'—H5F	0.9600
C1^i—Sn1—C1	147.58 (17)	H2A—C2—H2B	107.8
C1^i—Sn1—Cl1	100.36 (9)	O1—C3—O2	123.1 (3)
C1—Sn1—Cl1	100.54 (9)	O1—C3—C2	123.3 (3)
C1^i—Sn1—Cl1^i	100.54 (9)	O2—C3—C2	113.6 (3)
C1—Sn1—Cl1^i	100.36 (8)	C5—C4—O2	105.7 (6)
Cl1—Sn1—Cl1^i	99.01 (5)	C5—C4—H4A	110.6
C1^i—Sn1—O1^i	72.36 (9)	O2—C4—H4A	110.6
C1—Sn1—O1^i	84.17 (10)	C5—C4—H4B	110.6
Cl1—Sn1—O1^i	171.24 (6)	O2—C4—H4B	110.6
Cl1^i—Sn1—O1^i	87.31 (7)	H4A—C4—H4B	108.7
C1^i—Sn1—O1	84.17 (10)	C4—C5—H5A	109.5
C1—Sn1—O1	72.36 (9)	C4—C5—H5B	109.5
Cl1—Sn1—O1	87.31 (7)	H5A—C5—H5B	109.5
Cl1^i—Sn1—O1	171.24 (6)	C4—C5—H5C	109.5
O1^i—Sn1—O1	87.10 (12)	H5A—C5—H5C	109.5
C3—O1—Sn1	106.85 (18)	H5B—C5—H5C	109.5
C3—O2—C4'	127.8 (8)	O2—C4'—C5'	97.8 (14)
C3—O2—C4	114.1 (3)	O2—C4'—H4C	112.2
C2—C1—Sn1	112.48 (19)	C5'—C4'—H4C	112.2
C2—C1—H1A	109.1	O2—C4'—H4D	112.2
Sn1—C1—H1A	109.1	C5'—C4'—H4D	112.2

C2—C1—H1B	109.1	H4C—C4′—H4D	109.8
Sn1—C1—H1B	109.1	C4′—C5′—H5D	109.5
H1A—C1—H1B	107.8	C4′—C5′—H5E	109.5
C3—C2—C1	112.7 (3)	H5D—C5′—H5E	109.5
C3—C2—H2A	109.1	C4′—C5′—H5F	109.5
C1—C2—H2A	109.1	H5D—C5′—H5F	109.5
C3—C2—H2B	109.1	H5E—C5′—H5F	109.5
C1—C2—H2B	109.1		
C1ⁱ—Sn1—O1—C3	−179.4 (2)	Sn1—O1—C3—C2	−10.9 (4)
C1—Sn1—O1—C3	23.3 (2)	C4′—O2—C3—O1	−23.8 (10)
Cl1—Sn1—O1—C3	−78.7 (2)	C4—O2—C3—O1	4.0 (6)
O1ⁱ—Sn1—O1—C3	108.1 (2)	C4′—O2—C3—C2	156.3 (9)
C1ⁱ—Sn1—C1—C2	−75.5 (2)	C4—O2—C3—C2	−175.9 (5)
Cl1—Sn1—C1—C2	53.8 (2)	C1—C2—C3—O1	−14.1 (4)
Cl1ⁱ—Sn1—C1—C2	155.14 (19)	C1—C2—C3—O2	165.9 (3)
O1ⁱ—Sn1—C1—C2	−118.7 (2)	C3—O2—C4—C5	158.5 (8)
O1—Sn1—C1—C2	−29.9 (2)	C4′—O2—C4—C5	−75 (2)
Sn1—C1—C2—C3	36.4 (3)	C3—O2—C4′—C5′	109.5 (14)
Sn1—O1—C3—O2	169.1 (2)	C4—O2—C4′—C5′	41.5 (16)

Symmetry codes: (i) −*x*, *y*, −*z*+3/2.

Fig. 1