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Bis(μ-monochloroacetato-*O*,*O*′)-bis[dimethyltin(IV)]

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Abstract. $[(CH_3)_2Sn(O_2CCH_2Cl)]_2$, $C_8H_{16}Cl_2O_4Sn_2$, M_r = 488·40, monoclinic, space group $C2/m$, a = 14·661 (5), b = 7·425 (5), c = 7·963 (3) Å, β = 118·19 (2)°, D_m = 2·05 (3), D_x = 2·05 g cm^{-3}, Z = 2. The structure was refined from 960 diffractometer measured X-ray reflections to R_w = 0·063. The molecules are dimers with C_{2h} symmetry, Sn—Sn = 2·692 (3) Å and slightly asymmetrically bridging monochloroacetate groups [Sn—O = 2·241 (7) and 2·349 (7) Å]. Each Sn completes a trigonal bipyramidal coordination with two equatorially arranged methyl groups [Sn—C = 2·11 (1) Å].

Introduction. Bis(μ-monochloroacetato-*O*,*O*′)-bis[dimethyltin(IV)] was prepared by the solvolysis of hexamethylditin by a tenfold excess of monochloroacetic acid in chloroform solution in a bath held at 35 °C over a 72 h period. When all gas evolution had ceased distilled water was added, the mixture allowed to warm to room temperature and the white solid filtered, washed with distilled water and dried in vacuum. Crystals for X-ray analysis were obtained by slow crystallization from $CHCl_3$ solution. A cylindrically shaped crystal 0·1 mm in diameter and 0·2 mm long, elongated along [101], was mounted on a Syntex $P\bar{1}$ diffractometer. Lattice parameters were calculated from the settings of 15 well centered reflections with $20 \leq 2\theta \leq 36°$ using Mo $K\alpha$ radiation (λ = 0·71069 Å). The only systematic absences were hkl, $h + k = 2n + 1$, indicating one of the space groups $C2$, Cm or $C2/m$. The latter was assumed and found to give satisfactory refinement. Intensities of 988 reflections with $0 \leq h \leq 19$, $-1 \leq k \leq 9$ and $-10 \leq l \leq 9$ and $2\theta < 54°$ were measured using a $\theta/2\theta$ scan. No correction was made for absorption ($\mu r \sim 0·1$). Equivalent reflections were averaged to give 960 unique reflections out of a possible 1057. Of these, 223 had intensities less than three times the standard counting error and a further seven that were misset during measurement were not used. The structure was solved from the Patterson function and the positional and anisotropic temperature factors of the non-hydrogen atoms were refined by least-squares calculations using the locally written program *CUDLS* to give R_1 = 0·059 and R_w $\{=[\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}\}$ equal to 0·063 where $w = [\sigma^2 + (0·025F_o)^2]^{-1}$, σ being the standard error in F_o derived from counting statistics. An extinction correction given by $F^* = F[1 + 0·4 \times 10^{-6}\beta(2\theta)F^2]^{1/2}$ (Larson, 1967) was applied. Atomic scattering factors corrected for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1962). The final coordinates are given in Table 1.‡ The hydrogen-atom position in Table 1 was deduced from chemical considerations but it corresponds to a peak of 0·5 e Å^{-3} in the final difference synthesis. No clear positions for the methyl H atoms could be seen in this synthesis whose largest feature was a peak of 1·4 e Å^{-3} at the center of symmetry in the molecule.

Table 1. *Atomic coordinates* (×10^4, *for* H ×10^3)

	x	*y*	*z*
Sn	485 (1)	0	3926 (1)
Cl	3168 (3)	0	2205 (5)
O(1)	1964 (6)	0	6682 (13)
O(2)	1222 (6)	0	8574 (14)
C(1)	1991 (9)	0	8299 (19)
C(2)	3086 (10)	0	9930 (19)
C(3)	833 (8)	2400 (18)	2923 (16)
H(1)	344	120	975

Table 2. *Bond lengths* (Å) *and angles* (°)

Sn—O(1)	2·241 (7)	Sn—Sn—C(3)	122·3 (4)
Sn—O(2)	2·349 (7)	Sn—Sn—O(1)	86·3 (3)
Sn—Sn	2·692 (3)	Sn—Sn—O(2)	82·4 (3)
Sn—C(3)	2·112 (14) ×2	C(3)—Sn—C(3)	115·1 (5)
C(1)—O(1)	1·27 (2)	Sn—O(1)—C(1)	123·0 (8)
C(1)—O(2)	1·25 (2)	Sn—O(2)—C(1)	122·8 (9)
C(1)—C(2)	1·51 (2)	O(1)—C(1)—O(2)	125·5 (10)
C(2)—Cl	1·76 (2)	O(1)—C(1)—C(2)	112·5 (13)
		O(2)—C(1)—C(2)	122·0 (14)
		C(1)—C(2)—Cl	114·3 (12)

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‡ Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33800 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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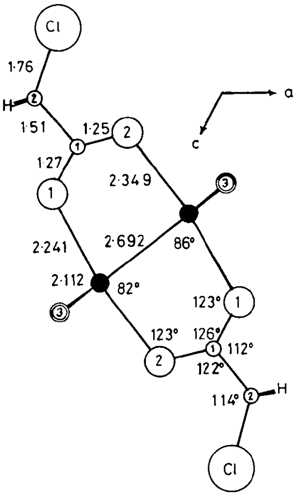

Fig. 1. $[(CH_3)_2Sn(O_2CCH_2Cl)]_2$ viewed down the twofold axis. The large numbered circles are O, the small numbered circles are C, the shaded circles are Sn. All atoms except C(3) and H lie on the mirror plane.

Discussion. Bond lengths and angles are given in Table 2 and a diagram of the molecule is shown in Fig. 1. The molecule has $2/m$ (C_{2h}) crystallographic symmetry but, apart from the Cl and H atoms, differs only slightly, although significantly, from $2/mmm$ (D_{2h}) symmetry. The molecules are isolated from each other; no intermolecular distance less than 3·6 Å occurs between non-hydrogen atoms.

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Di-μ_3-oxo-bis(μ-trifluoroacetato-O,O')-bis(trifluoroacetato)tetrakis[dimethyltin(IV)]

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Abstract. $\{[(CH_3)_2Sn(O_2CCF_3)]_2O\}_2$, $(C_8H_{12}F_6O_5Sn_2)_2$, $M_r = 2 \times 539{\cdot}4$, monoclinic space group $C2/m$, $a = 16{\cdot}887\,(5)$, $b = 8{\cdot}649\,(3)$, $c = 11{\cdot}502\,(2)$ Å, $\beta = 103{\cdot}23\,(2)°$, $D_x = 2{\cdot}19$ g cm^{-3}, $Z = 4$. The structure was refined from X-ray diffractomer measurements to give $R_w = 0{\cdot}026$. The dimeric unit lies in the mirror plane and has a structure virtually identical to that of $\{[(C_4H_9)_2Sn(O_2CCCl_3)]_2O\}_2$ [Graziani, Bombieri, Forsellini, Furlan, Peruzzo & Tagliavini (1977), *J. Organomet. Chem.* **125**, 43–55], with one symmetrically bridging trifluoroacetate group and one very asymmetric group. Both Sn atoms have trigonal bipyramidal coordination (the methyl groups in the equatorial plane) with one or two further O neighbours.

Introduction. $\{[(CH_3)_2Sn(O_2CCF_3)]_2O\}_2$ was obtained from an attempted recrystallization of $(CH_3)_4Sn_2(O_2CCF_3)_2$ from chloroform. Heating a chloroform solution of $(CH_3)_4Sn_2(O_2CCF_3)_2$ results in tin–tin cleavage and formation of the title compound as colourless crystals. The density could not readily be measured because of impurities in the bulk sample but a crystal cylindrical in shape (radius = 0·1 mm, cylinder axis = [100]) was selected and mounted on a Syntex $P2_1$ diffractometer. Lattice parameters were calculated from the settings of 15 well centred reflections with $7° \leq 2\theta \leq 34°$ using Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å). The only systematic absences were hkl, $h + k = 2n + 1$, indicating space groups $C2/m$, Cm or $C2$. The former was assumed and found to give a satisfactory structure. Intensities of 1543 reflections with $0 \leq h \leq 18$, $-1 \leq k \leq 9$, $-12 \leq l \leq 12$ and $2\theta \leq 48°$ were measured using a θ–2θ scan on a Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation.

No correction for absorption was necessary since $\mu = 31{\cdot}5$ cm^{-1}. Standard reflections were measured at regular intervals and showed no significant variation. Equivalent reflections were averaged to give 1243

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