

Fig. 1. $[(\text{CH}_3)_2\text{Sn}(\text{O}_2\text{CCH}_2\text{Cl})_2]_2$ 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 $2/m\bar{m}$ (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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References

- International Tables for X-ray Crystallography* (1962). Vol. III, pp. 202–216. Birmingham: Kynoch Press.
LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.

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

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Abstract. $\{[(\text{CH}_3)_2\text{Sn}(\text{O}_2\text{CCF}_3)_2\text{O}]_2, (\text{C}_8\text{H}_{12}\text{F}_6\text{O}_5\text{Sn}_2)_2, M_r = 2 \times 539.4$, monoclinic space group $C2/m$, $a = 16.887$ (5), $b = 8.649$ (3), $c = 11.502$ (2) Å, $\beta = 103.23$ (2)°, $D_x = 2.19$ g cm $^{-3}$, $Z = 4$. The structure was refined from X-ray diffractometer measurements to give $R_w = 0.026$. The dimeric unit lies in the mirror plane and has a structure virtually identical to that of $\{[(\text{C}_4\text{H}_9)_2\text{Sn}(\text{O}_2\text{CCl}_3)_2\text{O}]_2$ [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. $\{[(\text{CH}_3)_2\text{Sn}(\text{O}_2\text{CCF}_3)_2\text{O}]_2$ was obtained from an attempted recrystallization of $(\text{CH}_3)_4\text{Sn}_2(\text{O}_2\text{CCF}_3)_2$ from chloroform. Heating a chloroform

solution of $(\text{CH}_3)_4\text{Sn}_2(\text{O}_2\text{CCF}_3)_2$ 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 $7^\circ \leq 2\theta \leq 34^\circ$ using Mo $K\alpha$ radiation ($\lambda = 0.71069$ 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 $2\theta \leq 48^\circ$ 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 $\mu = 31.5$ 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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unique reflections out of a possible 1284. Of these, 150 were 'unobserved', *i.e.* had intensities less than 3σ where σ is the error based on counting statistics. The non-hydrogen atoms were found from Patterson and difference electron density maps, and their coordinates including anisotropic temperature factors were refined by least-squares calculations using the program *CUDLS* to give $R_1 = 0.028$ and $R_w = \{[\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2}\}$ of 0.033 where $w = [\sigma^2 + (kF)^2]^{-1}$; k was adjusted to make $\langle w|\Delta F|^2 \rangle$ independent of $|F|$ and this resulted in $k = 0.0025$. Unobserved reflections were given zero weight if $|F_c| < |F_o|$. The F atoms were

Table 1. Atomic positions ($\times 10^4$, for H $\times 10^3$)

	x	y	z
Sn(1)	952 (1)	0	-72 (1)
Sn(2)	455 (1)	0	2980 (1)
O(1)	2151 (3)	0	1472 (5)
O(2)	1792 (3)	0	3193 (5)
O(3)	971 (4)	0	5651 (5)
O(4)	915 (3)	0	7545 (4)
O(5)	256 (3)	0	1162 (4)
C(1)	2271 (5)	0	2553 (7)
C(2)	3181 (5)	0	3217 (8)
C(3)	1272 (5)	0	6693 (7)
C(4)	2208 (4)	0	7083 (9)
C(5)	8749 (5)	2336 (12)	252 (7)
C(6)	9492 (5)	2347 (10)	6517 (7)
F(1)	3311 (7)	0	4327 (11)
F(2)	3577 (5)	1127 (11)	2879 (9)
F(3)	3683 (10)	0	2496 (17)
F(4)	3370 (6)	1136 (14)	3914 (14)
F(5)	2585 (10)	0	6247 (15)
F(6)	2460 (6)	1136 (13)	7821 (12)
F(7)	2530 (7)	1131 (15)	6697 (16)
F(8)	2483 (13)	0	8262 (22)
H(1)	828 (5)	211 (10)	54 (7)
H(2)	902 (7)	274 (15)	79 (10)
H(3)	886 (10)	251 (22)	-61 (15)
H(4)	961 (10)	283 (21)	708 (14)
H(5)	963 (10)	263 (21)	574 (14)
H(6)	903 (7)	238 (15)	627 (58)

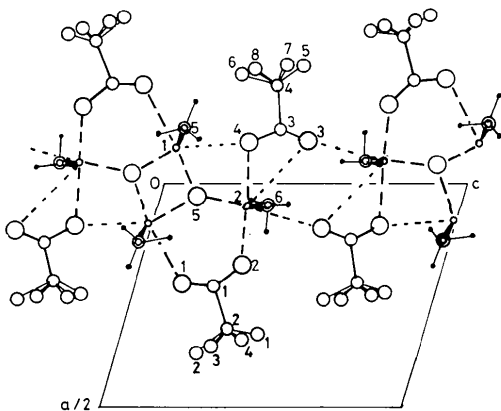


Fig. 1. Structure of $\{[(\text{CH}_3)_2\text{Sn}(\text{O}_2\text{CCF}_3)]_2\text{O}\}_2$. The circles represent in decreasing order of size O, F, C and Sn. H atoms are shown as dots.

found to be disordered, so two sets of $\frac{1}{2}$ occupancy sites rotated 60° to each other about the C—C axes were selected.

An extinction correction given by $F^* = F[1 + 0.68 \times 10^{-7} \times \beta(2\theta)F^2]^{1/2}$ (Larson, 1967) was applied. The average value of the shift/error in the final round was 0.04. Atomic scattering factors corrected for

Table 2. Bond distances (Å) and angles ($^\circ$)

Tin coordination

Sn(1)—C(5)	2.104 (10) $\times 2$	Sn(2)—C(6)	2.107 (9) $\times 2$
Sn(1)—O(5)	2.039 (5)	Sn(2)—O(5)	2.040 (4)
Sn(1)—O(5)'	2.137 (4)	Sn(2)—O(4)	2.253 (5)
Sn(1)—O(1)	2.367 (5)	Sn(2)—O(2)	2.215 (5)
Sn(1)—O(4)	2.727 (5)	Sn(2)—O(3)*	2.996 (6)
		Sn(2)—O(3)	3.164 (7)

C(5)—Sn(1)—C(5)'	147.5 (3)	C(6)—Sn(2)—C(6)'	148.9 (2)
C(5)—Sn(1)—O(5)	105.0 (2) $\times 2$	C(6)—Sn(2)—O(5)	105.5 (2)
O(5)′—Sn(1)—C(5)	99.3 (2) $\times 2$	O(4)—Sn(2)—C(6)	92.9 (2) $\times 2$
O(5)′—Sn(1)—O(5)	77.5 (2)	O(4)—Sn(2)—O(5)	78.9 (2)
O(1)—Sn(1)—C(5)	83.7 (2) $\times 2$	O(2)—Sn(2)—C(6)	89.5 (2) $\times 2$
O(1)—Sn(1)—O(5)	90.5 (2)	O(2)—Sn(2)—O(5)	92.2 (2)
O(1)—Sn(1)—O(5)′	168.0 (3)	O(4)—Sn(2)—O(2)	171.2 (4)
O(4)—Sn(1)—O(5)′	67.1 (2)	O(3)*—Sn(2)—O(2)	80.6 (2)
O(4)—Sn(1)—C(5)	81.5 (2) $\times 2$	O(3)*—Sn(2)—C(6)	74.6 (2) $\times 2$
		O(3)—Sn(2)—O(4)	44.1 (2)
		O(3)—Sn(2)—C(6)	81.5 (2) $\times 2$

Trifluoroacetate (F atoms are disordered with each site occupied by half an atom)

C(1)—O(1)	1.212 (11)	C(3)—O(3)	1.190 (9)
C(1)—O(2)	1.213 (10)	C(3)—O(4)	1.263 (10)
C(1)—C(2)	1.552 (11)	C(3)—C(4)	1.541 (10)
C(2)—F(1)	1.245 (15)	C(4)—F(5)	1.269 (21)
C(2)—F(2)	1.291 (11) $\times 2$	C(4)—F(6)	1.304 (13) $\times 2$
C(2)—F(3)	1.315 (22)	C(4)—F(8)	1.329 (27)
C(2)—F(4)	1.262 (14) $\times 2$	C(4)—F(7)	1.249 (16) $\times 2$

O(1)—C(1)—O(2)	130.1 (7)	O(3)—C(3)—O(4)	127.7 (8)
O(1)—C(1)—C(2)	114.8 (8)	O(3)—C(3)—C(4)	117.8 (8)
O(2)—C(1)—C(2)	115.1 (7)	O(4)—C(3)—C(4)	114.5 (7)
C(1)—C(2)—F(1)	115.3 (9)	C(3)—C(4)—F(5)	116.0 (10)
C(1)—C(2)—F(2)	112.0 (6) $\times 2$	C(3)—C(4)—F(6)	110.5 (7) $\times 2$
C(1)—C(2)—F(3)	113.5 (10)	C(3)—C(4)—F(8)	113.1 (12)
C(1)—C(2)—F(4)	113.0 (7) $\times 2$	C(3)—C(4)—F(7)	113.4 (7) $\times 2$
F(1)—C(2)—F(2)	109.1 (7) $\times 2$	F(5)—C(4)—F(6)	110.3 (8) $\times 2$
F(2)—C(2)—F(2)′	98.0 (9)	F(6)—C(4)—F(6)′	97.7 (10)
F(3)—C(2)—F(4)	107.1 (9) $\times 2$	F(8)—C(4)—F(7)	106.5 (10) $\times 2$
F(4)—C(2)—F(4)′	102.2 (11)	F(7)—C(4)—F(7)′	103.1 (12)

Angles at oxygen

Sn(1)—O(5)—Sn(1)′	102.5 (2)	C(1)—O(1)—Sn(1)	133.0 (5)
Sn(1)—O(5)—Sn(2)	136.6 (2)	C(1)—O(2)—Sn(2)	137.6 (5)
Sn(1)′—O(5)—Sn(2)	120.8 (3)	C(3)—O(4)—Sn(2)	115.8 (4)
[The bond Sn(1)—O(5) is shorter than Sn(1)′—O(5).]		C(3)—O(4)—Sn(1)	151.0 (4)
		C(3)—O(3)—Sn(2)†	171.9 (6)
		C(3)—O(3)—Sn(2)	72.4 (5)

Methyl groups

	Sn—C—H	C—H	H—F	C—H—F	C—F
C(5)H(1)F(6)	94 (7)	0.95 (8)	2.62 (8)	152 (7)	3.50 (2)
F(8)			2.77 (8)	146 (7)	3.60 (2)
C(5)H(2)F(2)	114 (10)	0.77 (12)	2.86 (12)	127 (10)	3.37 (1)
F(3)			2.92 (12)	132 (10)	3.48 (2)
C(5)H(3)F(6)	87 (11)	1.06 (18)	2.88 (16)	110 (10)	3.39 (1)
C(6)H(4)	108 (15)	0.76 (18)			
C(6)H(5)F(4)	118 (10)	1.00 (18)	2.84 (15)	118 (10)	} 3.42 (2)
C(6)H(6)F(4)	96 (12)	0.77 (29)	2.97 (57)	120 (45)	
F(7)			2.98 (21)	141 (7)	

* Intermolecular bond.

† Sn in neighbouring dimer.

anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1962). A difference synthesis calculated at this stage showed a large negative peak in the neighbourhood of the F atoms and slightly smaller positive peaks in the positions expected for the methyl H atoms. The H atoms were therefore included in a further calculation in which the positional and temperature coordinates of the H and methyl C atoms were varied. The agreement index dropped to $R_1 = 0.026$ and $R_w = 0.026$. The average shift/error for this refinement was 0.4 [max. = 1.5 for U of H(3)]. The final value of $[\sum w(|F_c| - |F_o|)^2]/(\text{number of reflections} - \text{number of variables}) = 2.2$. The final positional coordinates are given in Table 1.*

Discussion. Bond lengths and angles are given in Table 2 and the structure is illustrated in Fig. 1. The dimer is essentially identical to that of $\{[(\text{C}_4\text{H}_9)_2\text{Sn}(\text{O}_2\text{CCl}_3)]_2\text{O}\}_2$ (Graziani *et al.* 1977) although the packing of the molecules, and consequently the crystal cell and space group, are quite different. $\{[(\text{CH}_3)_2\text{Sn}(\text{O}_2\text{CCH}_3)]_2\text{O}\}_2$ crystallizes with a cell similar to the trifluoroacetate and presumably has the same structural topology (Zvonkova, Povet'eva, Vozžennikov, Gluškova, Jakovenko & Khvatkina, 1966) although the space group is different ($P2_1/a$ in the setting used here). In the trifluoroacetate all atoms, except the methyl groups and two thirds of the F atoms, lie in the mirror plane. Each dimer contains four Sn atoms of which two are crystallographically distinct. Both have trigonal bipyramidal coordination with the two methyl

groups (Sn—C = 2.11 Å) and the bridging oxygen (Sn—O = 2.040 Å) in the equatorial plane. The axial Sn—O bonds are slightly longer (2.24 Å average). In addition each Sn has one or two further O neighbours (Sn—O ~ 2.8 Å) lying between the two methyl groups.

Although the Sn atoms have similar environments, the trifluoroacetate (tfa) ions are quite different. One forms a symmetrical bridge between Sn(1) and Sn(2). The other is strongly bonded only to Sn(2) making the ion very asymmetric. The other oxygen atom, O(3), forms a weak bond to Sn(2) in the adjacent dimer linking the molecules into chains running along the c axis. The chains are bonded to each other only through weak C—H...F interactions between the F atoms of one chain and the methyl groups of the chains related by the C -centring operation.

The model successfully refined with each $-\text{CF}_3$ group replaced by two half $-\text{CF}_3$ groups rotated by 60° about the tfa C—C axis. The temperature factors of the F atoms are elongated in such a way as to suggest that the CF_3 groups might be rotating but a hindered rotation or even static disorder cannot be ruled out. All other atoms show their largest thermal vibrations perpendicular to the molecular plane.

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References

- GRAZIANI, R., BOMBIERI, G., FORSELLINI, E., FURLAN, P., PERUZZO, V. & TAGLIAVINI, G. (1977). *J. Organomet. Chem.* **125**, 43–55.
International Tables for X-ray Crystallography (1962). Vol. III, pp. 202–216. Birmingham: Kynoch Press.
LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.
ZVONKOVA, Z. V., POVET'eva, Z. P., VOZŽENNIKOV, V. M., GLUŠKOVA, V. P., JAKOVENKO, V. I. & KHVATKINA, A. N. (1966). *Acta Cryst.* **21**, A155.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33801 (8 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trichloro(η -*N*-methyl-4-pentenylammonium)platinum(II)

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Abstract. $\text{C}_6\text{H}_{14}\text{Cl}_3\text{NPt}$, orthorhombic, $Pna2_1$, $a = 18.623$ (7), $b = 8.385$ (2), $c = 7.261$ (2) Å, $D_m = 2.31$ (1), $Z = 4$, $D_c = 2.338$ gcm $^{-3}$. Cell parameters were determined by a least-squares refinement of the

setting angles of 15 reflections. The zwitterionic complex was prepared by method (1) of Denning & Venanzi [*J. Chem. Soc.* (1963), pp. 3241–3247]. *N*-Methyl-4-pentenylamine was provided by J. Ambühl [Dis-