

(Johnson, Lewis, Raithby & Suss, 1979), (μ -H)-Ru₃(CO)₁₀(μ -COMe) (Johnson, Lewis, Orpen, Raithby & Suss, 1979; Churchill, Beanan, Wasserman, Bueno, Rahman & Keister, 1983) and (μ -AuPPh₃)Ru₃(CO)₁₀(μ -COMe) (Green, Mead, Mills, Salter & Stone, 1982; Bateman, Green, Mead, Mills, Slater, Stone & Woodward, 1983) and the iron cluster (μ -H)Fe₃(CO)₁₀(μ -COMe) (Shriver, Lehman & Strope, 1975) have structures similar to the Os system.

The feature of interest in (μ -H)Os₃(CO)₁₀(μ -COCH₂Cl) is the μ -halomethoxy ligand. It symmetrically spans the Os(2)—Os(3) edge along with the μ -hydride ligand. This type of multiple bridging is also observed in the clusters mentioned above. The metal—C bond distances and bond angles associated with the bridging —COCH₂Cl unit [Os(2)—C(231) = 1.986 (7), Os(3)—C(231) = 2.001 (7), C(231)—O(231) = 1.330 (7) Å, Os(2)—C(231)—Os(3) = 91.6 (3), Os(2)—C(231)—O(231) = 127.7 (5), Os(3)—C(231)—O(231) = 140.8 (6)°] are comparable to those found for the bridging —COMe unit found in (μ -H)Ru₃(CO)₁₀(μ -COMe) (Churchill *et al.*, 1983) [Ru—C = 1.978–1.991, C—O = 1.305 (5) Å, Ru—C—Ru = 90.62 (2), Ru—C—O = 128.6–140.3°] or (Johnson, Lewis, Orpen *et al.*, 1979) [Ru—C = 1.976–1.978, C—O = 1.299 (8) Å, Ru—C—Ru = 90.3 (3), Ru—C—O = 128.9–140.2°] and (μ -AuPPh₃)Ru₃(CO)₁₀(μ -COMe) (Green *et al.*, 1982) (Ru—C = 1.94–1.99 Å) or (μ -AuPPh₃)Os₃(CO)₁₀(μ -COMe) (Farrugia, 1986) (Os—C = 1.98–2.02, C—O = 1.28–1.30 Å, Os—C—Os = 92.0–92.2°). The title compound is also similar to the carbyne cluster (μ -H)Os₃(CO)₁₀(μ -CCH₂CHMe₂) (Green *et al.*, 1984) with respect to the —CCH₂CHMe₂ bridging moiety [Os—C range of 1.966–2.020 Å, Os—C—Os = 92.2 (8)°]. For the Os—H—Os bridge bond distances and angles are essentially equivalent in all these clusters.

We thank the National Science Foundation for support of this work through grant CHE 88-00515 and grant CHE 84-11630 for an X-ray diffractometer.

References

- B. A. FRENZ & ASSOCIATES INC. (1982). *SDP Structure Determination Package*. College Station, Texas, USA.
- BARRETO, R. D., FEHLNER, T. P., HSU, L.-Y., JAN, D.-Y. & SHORE, S. G. (1986). *Inorg. Chem.* **25**, 3572–3581.
- BATEMAN, L. W., GREEN, M., MEAD, K. A., MILLS, R. M., SLATER, I. D., STONE, F. G. A. & WOODWARD, P. (1983). *J. Chem. Soc. Dalton Trans.* pp. 2599–2608.
- CHURCHILL, M. R., BEANAN, L. R., WASSERMAN, H. J., BUENO, C., RAHMAN, Z. A. & KEISTER, J. B. (1983). *Organometallics*, **2**, 1179–1186.
- Enraf-Nonius (1985). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.
- FARRUGIA, L. J. (1986). *Acta Cryst.* **C42**, 680–682.
- GREEN, M., MEAD, K. A., MILLS, R. M., SALTER, I. D. & STONE, F. G. A. (1982). *J. Chem. Soc. Chem. Commun.* pp. 51–53.
- GREEN, M., ORPEN, A. G. & SCHAEVERIEN, C. J. (1984). *J. Chem. Soc. Chem. Commun.* pp. 37–39.
- JAN, D.-Y., HSU, L.-Y., WORKMAN, D. P. & SHORE, S. G. (1987). *Organometallics*, **6**, 1984–1985.
- JOHNSON, B. F. G., LEWIS, J., ORPEN, A. G., RAITHBY, P. R. & SUSS, G. (1979). *J. Organomet. Chem.* **173**, 187–197.
- JOHNSON, B. F. G., LEWIS, J., RAITHBY, P. R. & SUSS, G. (1979). *J. Chem. Soc. Dalton Trans.* pp. 1356–1361.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1982). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- SHAPLEY, J. R., STRICKLAND, D. S., ST GEORGE, G. M., CHURCHILL, M. R. & BUENO, C. (1983). *Organometallics*, **2**, 185–187.
- SHORE, S. G., JAN, D.-Y., HSU, L.-Y. & HSU, W.-L. (1983). *J. Am. Chem. Soc.* **105**, 5923–5924.
- SHRIVER, D. F., LEHMAN, D. & STROPE, D. (1975). *J. Am. Chem. Soc.* **97**, 1594–1596.
- SIEVERT, A. C., STRICKLAND, D. S., SHAPLEY, J. R., STEINMETZ, G. R. & GEOFFROY, G. L. (1982). *Organometallics*, **1**, 214–215.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1990). **C46**, 2088–2091

5,5-Dichloro-10,11-dihydrodibenzo[*b, f*]stannepine

BY HANS PREUT, JOST KOCH AND FRIEDO HUBER

Lehrstuhl für Anorganische Chemie II, Universität Dortmund, D-4600 Dortmund 50, Postfach 500 500, Federal Republic of Germany

(Received 22 January 1990; accepted 20 March 1990)

Abstract. C₁₄H₁₂Cl₂Sn, $M_r = 369.85$, monoclinic, $P2_1/n$, $a = 11.405$ (2), $b = 8.106$ (2), $c = 16.090$ (4) Å, $\beta = 105.72$ (2)°, $V = 1431.9$ (6) Å³, Z

$= 4$, $D_x = 1.716$ Mg m⁻³, $F(000) = 720$, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 2.14$ mm⁻¹, $T = 291$ (1) K, final $R = 0.023$ for 2390 unique reflexions [$F \geq 3.0\sigma(F)$].

0108-2701/90/112088-04\$03.00

© 1990 International Union of Crystallography

Two Cl atoms and two C(benzene) atoms are in slightly distorted tetrahedral positions around Sn, which is part of the seven-membered central stannepine ring. The tricyclic framework exhibits a folded boat conformation. The dihedral angle between the planes of the two benzene rings is $155.2(1)^\circ$. Intermolecular coordination *via* Sn—Cl interaction does not exist.

Introduction. Diorganotin compounds R_2SnX_2 are usually polymeric in the solid state when X is an electronegative ligand like a halide and when there are no sterical implications. Thus in Me_2SnCl_2 bridging Cl atoms form the equatorial plane of an octahedron around Sn, the two Me groups being in axial positions (Davies, Milledge, Puxley & Smith, 1970). In contrast, bis(biphenyl-2-yl)tin dichloride is a monomer with two Cl and two C(biphenyl) atoms in a heavily distorted tetrahedral environment around Sn (Baxter, Holt & Zuckerman, 1985). In the context of investigations on the acceptor properties of Sn in R_2SnX_2 (R_2Sn = stannacycle) we were interested to find out if Sn being a ring member retains its ability to take part in $X-Sn-X$ bridging forming a polymer in the solid state or if such R_2SnX_2 compounds are monomeric avoiding coordination to prevent additional constraints in the central ring. The first structure of a stannacycle is reported in the present paper; it shows that the title compound is monomeric in the solid state.

Experimental. The title compound was prepared from 2,2'-dilithiobibenzyl and $SnCl_4$ in Et_2O , similar to a procedure given by Kuivila & Beumel (1958). Single crystals were obtained from a solution of 1 g in 10 ml Et_2O and 40 ml *n*-pentane after 2 weeks at 248 K. Crystal size $\sim 0.50 \times 0.44 \times 0.16$ mm, D_m not determined, $\omega/2\theta$ scan, scan speed $1.50-14.65^\circ \text{ min}^{-1}$ in θ , scan width $(1.2 + \text{dispersion})^\circ$; Nicolet $R3m/V$ diffractometer, graphite-monochromated $Mo K\alpha$; lattice parameters from least-squares fit with 25 reflexions up to $2\theta = 30.02^\circ$; ω scans of low order reflexions along the three crystal axes showed acceptable mosaicity; six standard reflexions (611, 040, 004, $\bar{6}1\bar{1}$, 040, 004) recorded every 2.5 h, only random deviations over 31.4 h of X-ray exposure; 4065 reflexions measured, $3.0 \leq 2\theta \leq 50.0^\circ$, $-14 \leq h \leq 14$, $-10 \leq k \leq 10$, $-20 \leq l \leq 20$; after averaging ($R_{int} = 0.029$): 2496 unique reflexions, 2390 with $F \geq 3.0\sigma(F)$; Lorentz-polarization correction and absorption correction *via* ψ scans, max./min. transmission 0.96/0.68; systematic absences ($h0l$) $h+1 = 2n+1$, ($0k0$) $k = 2n+1$ conform to space group $P2_1/n$; structure solution by direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H

atoms, which were placed in geometrically calculated positions (C—H 0.96 Å); refinement on F with 2390 reflexions and 155 refined parameters; $w = 1.0/[\sigma^2(F) + (0.000413 F^2)]$ which led to featureless analysis of variance in terms of $\sin\theta$ and F_o ; $S = 1.51$, $R = 0.023$, $wR = 0.029$, $(\Delta/\sigma)_{max} = 0.035$; largest peak in final ΔF map $\pm 0.4(1) e \text{ \AA}^{-3}$; atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B, 2.3.1); programs: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987).

Discussion. The structure of the title compound together with the numbering scheme is shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths, bond angles, and least-squares planes and a dihedral angle are given in Table 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53150 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

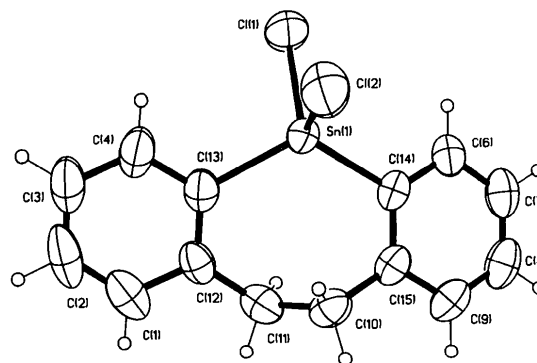


Fig. 1. General view (*SHELXTL-Plus* graphic) of the molecule, showing the atom-numbering scheme.

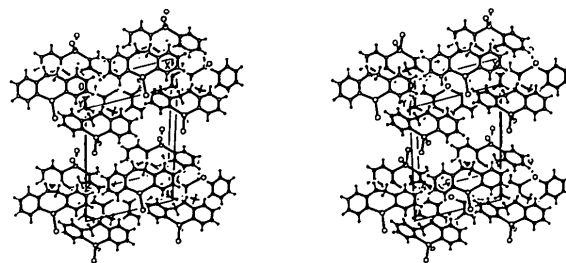


Fig. 2. Stereoscopic view (*SHELXTL Plus* graphic) of the unit cell (a nearly horizontal, c vertical).

Table 1. Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^4$)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Sn(1)	0.38072 (1)	0.07508 (2)	0.12497 (1)	472
Cl(1)	0.34696 (9)	-0.1229 (1)	0.01597 (6)	841
Cl(2)	0.40089 (7)	0.3185 (1)	0.05167 (5)	785
C(1)	0.1033 (3)	0.1147 (4)	0.2654 (3)	709
C(2)	-0.0022 (3)	0.1345 (4)	0.2000 (3)	832
C(3)	-0.0004 (3)	0.1385 (4)	0.1156 (3)	796
C(4)	0.1094 (3)	0.1200 (4)	0.0966 (2)	685
C(6)	0.6405 (2)	-0.0544 (3)	0.2032 (2)	561
C(7)	0.7450 (3)	-0.0838 (3)	0.2701 (3)	673
C(8)	0.7511 (3)	-0.0307 (4)	0.3515 (2)	722
C(9)	0.6545 (3)	-0.0527 (4)	0.3689 (2)	657
C(10)	0.4417 (3)	0.1728 (3)	0.3239 (2)	573
C(11)	0.3294 (3)	0.0638 (3)	0.3212 (2)	620
C(12)	0.2163 (3)	0.0942 (3)	0.2478 (2)	538
C(13)	0.2173 (2)	0.0982 (3)	0.1610 (2)	524
C(14)	0.5433 (2)	0.0282 (3)	0.2208 (2)	459
C(15)	0.5479 (3)	0.0838 (3)	0.3038 (2)	494

The atoms Cl(1), Cl(2), C(13) and C(14) form a slightly distorted tetrahedron around Sn. Considering this arrangement and the non-existence of short intermolecular Sn—Cl contacts even weak intermolecular coordination is safely excluded. The Sn—Cl distances of 2.331 (1) and 2.342 (1) Å are in the range which appears to be characteristic for non-bridging Sn—Cl bonds. They are significantly shorter than in organotin chlorides in which Cl participates in bridging as in pentacoordinated Me_3SnCl [2.431 (2) Å; Lefferts, Molloy, Hossain, van der Helm & Zuckerman, 1982], and are comparable to Sn—Cl distances in organotin chlorides with discrete molecules in the solid state, like Ph_3SnCl [2.32 (1) Å; Bokii, Zakharova & Struchkov, 1970], bis(biphenyl-2-yl)tin(IV) dichloride [mean: 2.386 (2) Å; Baxter, Holt & Zuckerman, 1985], or in diphenyltin(IV) dichloride. This compound was originally described as being monomeric (Greene & Bryan, 1971), but a reinterpretation of the data established four-membered chains with tetracoordinated Sn in the terminal molecules and hexacoordinated Sn in the two central molecules of each chain; the mean Sn—Cl bond length for Cl atoms participating in bridging is 2.355 (1) Å and for non-bridging Cl atoms 2.336 (1) Å (Bokii, Struchkov & Prokof'iev, 1972).

The bond angles C(11)—C(10)—C(15) and C(10)—C(11)—C(12) as well as the angle C(13)—Sn(1)—C(14) in the title compound are significantly larger than the ideal tetrahedral angle, probably as a consequence of internal strain in the seven-membered central ring. The angles at the — CH_2 — CH_2 — bridge of 115.2 (2) and 116.9 (2)° are only slightly different. Larger differences are observed at the analogous — CH_2 — CH_2 — bridges in the compounds 5,5-diphenyl-10,11-dihydro-5*H*-dibenzo[*b,f*]germepine [114.0 and 120.4°; Corey, Corey, Glick & Dueber, 1972], 5-methyl-5-phenyl-

Table 2. Bond distances (Å), bond angles (°), least-squares planes and dihedral angle (°)

Sn(1)—Cl(1)	2.331 (1)	C(6)—C(14)	1.389 (4)
Sn(1)—Cl(2)	2.342 (1)	C(7)—C(8)	1.363 (6)
Sn(1)—C(13)	2.105 (3)	C(8)—C(9)	1.384 (5)
Sn(1)—C(14)	2.100 (2)	C(9)—C(15)	1.396 (4)
C(1)—C(2)	1.377 (5)	C(10)—C(11)	1.547 (4)
C(1)—C(12)	1.402 (5)	C(10)—C(15)	1.518 (4)
C(2)—C(3)	1.364 (7)	C(11)—C(12)	1.514 (4)
C(3)—C(4)	1.374 (5)	C(12)—C(13)	1.400 (5)
C(4)—C(13)	1.389 (4)	C(14)—C(15)	1.397 (4)
C(6)—C(7)	1.392 (4)		
C(13)—Sn(1)—C(14)	119.0 (1)	C(11)—C(10)—C(15)	115.2 (2)
Cl(2)—Sn(1)—C(14)	109.54 (8)	C(10)—C(11)—C(12)	116.9 (2)
Cl(2)—Sn(1)—C(13)	106.21 (8)	C(1)—C(12)—C(11)	119.8 (3)
Cl(1)—Sn(1)—C(14)	111.16 (8)	C(11)—C(12)—C(13)	123.1 (3)
Cl(1)—Sn(1)—C(13)	106.88 (8)	C(1)—C(12)—C(13)	117.1 (3)
Cl(1)—Sn(1)—Cl(2)	102.70 (4)	C(4)—C(13)—C(12)	120.1 (3)
C(2)—C(1)—C(12)	121.3 (4)	Sn(1)—C(13)—C(12)	121.3 (2)
C(1)—Sn(1)—C(3)	121.2 (3)	Sn(1)—C(13)—C(4)	118.5 (2)
C(2)—C(3)—C(4)	118.6 (4)	Sn(1)—C(14)—C(6)	122.2 (2)
C(3)—C(4)—C(13)	121.6 (3)	C(6)—C(14)—C(15)	121.6 (3)
C(7)—C(6)—C(14)	119.3 (3)	Sn(1)—C(14)—C(15)	116.2 (2)
C(6)—C(7)—C(8)	119.9 (3)	C(10)—C(15)—C(14)	122.0 (3)
C(7)—C(8)—C(9)	120.8 (3)	C(9)—C(15)—C(14)	117.4 (3)
C(8)—C(9)—C(15)	121.0 (3)	C(9)—C(15)—C(10)	120.6 (3)

No. Plane through atoms
1 C(1), C(2), C(3), C(4), C(12), C(13)

2 C(6), C(7), C(8), C(9), C(14), C(15)

Equation of the plane (x along a;
y in the plane ab; z along c)
-0.124x - 0.991y - 0.057z =
-1.150 (4) Å
0.451x + 0.871y - 0.196z =
1.891 (8) Å

Dihedral angle: 1,2 155.2 (1)

10,11-dihydro-5*H*-dibenzo[*b,f*]silepine [111.7 (5) and 117.1 (5)°; Corey, Corey & Glick, 1977], and in the all-carbon compound 10,11-dihydro-5*H*-dibenzo[*a,d*]cycloheptene [113.3 (2) and 120.3 (2)°; Reboul, Cristau & Pèpe, 1981]. The C—M—C angles in these compounds decrease from 119.0 (1)° ($M = \text{Sn}$) to 114.8 (5)° ($M = \text{Ge}$) and 111.4 (3)° ($M = \text{Si}$) presumably as a consequence of the decrease of the mean M—C distances: 2.103 (3) Å ($M = \text{Sn}$), 1.95 (1) Å ($M = \text{Ge}$) and 1.884 (7) Å ($M = \text{Si}$). A comparison of the dihedral angles between the planes of the two benzene rings of the silepine compound (137.2°) and of the title compound [155.2 (1)°] indicates that the distortion of the molecule increases with decreasing covalent radius of the respective heteroatom in the central ring.

Financial support from Deutsche Forschungsgemeinschaft and Fonds der Chemischen Industrie is gratefully acknowledged.

References

- BAXTER, J. L., HOLT, E. M. & ZUCKERMAN, J. J. (1985). *Organometallics*, **4**, 255–259.
BOKII, N. G., STRUCHKOV, YU. T. & PROKOF'IEV, A. K. (1972). *J. Struct. Chem. (USSR)*, **13**, 619–623.
BOKII, N. G., ZAKHAROVA, G. N. & STRUCHKOV, YU. T. (1970). *J. Struct. Chem. (USSR)*, **11**, 828–835.
COREY, E. R., COREY, J. Y. & GLICK, M. D. (1977). *J. Organomet. Chem.* **129**, 17–25.

- COREY, J. Y., COREY, E. R., GLICK, M. D. & DUEBER, J. S. (1972). *J. Heterocycl. Chem.* **9**, 1379–1383.
- DAVIES, A. G., MILLEDGE, H. J., PUXLEY, D. C. & SMITH, P. J. (1970). *J. Chem. Soc. A*, pp. 2862–2866.
- GREENE, P. T. & BRYAN, R. F. (1971). *J. Chem. Soc. A*, pp. 2549–2554.
- KUIVILA, H. G. & BEUMEL, O. F. JR (1958). *J. Am. Chem. Soc.* **80**, 3250–3253.
- LEFFERTS, J. L., MOLLOY, K. C., HOSSAIN, M. B., VAN DER HELM, D. & ZUCKERMAN, J. J. (1982). *J. Organomet. Chem.* **240**, 349–361.
- LE PAGE, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.
- NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
- REBOUL, J. P., CRISTAU, B. & PÈPE, G. (1981). *Acta Cryst.* **B37**, 394–398.
- SHELDRIK, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet R3m/V crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.
- SPEK, A. L. (1982). The *EUCLID* Package. In *Computational Crystallography*, edited by D. SAYRE. Oxford: Clarendon Press.
- WILLIAMS, D. E. (1984). *PCK83*. Quantum Chem. Program Exchange Program No. 481.

Acta Cryst. (1990). **C46**, 2091–2093

Structure of Bis(formato)dimethyltin(IV)

BY FRED MISTRY, STEVEN J. RETTIG, JAMES TROTTER AND FELIX AUBKE

Department of Chemistry, University of British Columbia, 2036 Main Mall, Vancouver, BC, Canada, V6T 1Y6

(Received 3 November 1989; accepted 3 April 1990)

Abstract. $[\text{Sn}(\text{CH}_3)_2(\text{CHO}_2)_2]_n$, $(\text{C}_4\text{H}_8\text{O}_4\text{Sn})_m$, $M_r = 238.79$, orthorhombic, $Pnma$, $a = 12.693$ (2), $b = 9.128$ (2), $c = 6.013$ (2) Å, $V = 696.7$ (2) Å³, $Z = 4$, $D_x = 2.28$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 36.25$ cm⁻¹, $F(000) = 456$, $T = 294$ K, $R = 0.030$ ($wR = 0.037$) for 2683 reflections with $I \geq 3\sigma(I)$. The structure is that of a sheet polymer with linear Me_2Sn moieties nearly symmetrically bridged by formate anions. The coordination about the Sn atom is octahedral with $\text{Sn—O} = 2.246$ (2) and 2.249 (1) Å and $\text{Sn—C} = 2.097$ (3) and 2.116 (2) Å.

Introduction. Structural information on many dimethyltin(IV) salts of various acids has been reported over the years. Invariably, associated structures are encountered in which the dimethyltin(IV) groups are linked by bridging polydentate anions. A survey of important structural features allows classification into two general groups; (a) dimethyltin salts of strong protonic acids, like $(\text{CH}_3)_2\text{SnF}_2$ (Schlemper & Hamilton, 1966) and $(\text{CH}_3)_2\text{Sn}(\text{SO}_3\text{F})_2$ (Allen, Lerbscher & Trotter, 1971), where symmetrical anion bridges between linear C—Sn—C groups and short Sn—C distances are found, resulting in regular octahedral coordination about tin; (b) other dimethyltin derivatives, such as $(\text{CH}_3)_2\text{SnCl}_2$ (Davies, Milledge, Puxley & Smith, 1970), $(\text{CH}_3)_2\text{Sn}(\text{CN})_2$ (Konnert, Britton & Chow, 1972), $(\text{CH}_3)_2\text{Sn}(\text{NCS})_2$ (Chow, 1970) and $(\text{CH}_3)_2\text{Sn}(\text{MoO}_4)$ (Sasaki, Imoto & Nagano, 1984) where asymmetric bridges are encountered, with C—Sn—C angles departing noticeably from 180°, and longer Sn—C distances resulting in somewhat more distorted coordination

geometries about the Sn centre. The coordinative asymmetry of bridging groups such as —CN— or —NCS— may be seen as a plausible cause for the observed distortions, but this explanation is invalid for the MoO_4^{2-} groups.

To gain a better understanding of the reasons for the substantially disorted geometries when anions of weaker acids are involved, we became interested in structural studies of dimethyltin(IV) carboxylates of the type $(\text{CH}_3)_2\text{Sn}(\text{O}_2\text{CR})_2$ and selected the dimethyltin(IV) formate for this purpose because, with $R = \text{H}$ in this case, steric repulsion induced by the R group will be minimized.

Experimental. The dimethyltin bis(formate) was synthesized as reported previously (Okawara & Rochow, 1960). Colorless prisms, $0.25 \times 0.30 \times 0.42$ mm, Rigaku AFC6S diffractometer, lattice parameters from 25 reflections with $\theta = 31\text{--}33^\circ$. Intensities for $\theta < 50^\circ$, hkl : 0 to 27, 0 to 19, 0 to 12, ω - 2θ scan, ω -scan width $(1.5 + 0.30\tan\theta)^\circ$ at $32^\circ \text{ min}^{-1}$ (up to eight rescans), stationary backgrounds at each end of the scan counted 50% of the scan time, three standard reflections showed negligible variations in intensity, Lp and absorption corrections (analytical, relative transmission factors 0.572–1.000), 3764 independent reflections measured, 2683 with $I \geq 3\sigma(I)$, $\sigma^2(F_o^2) = [S^2(C + 4B) + (0.023F_o^2)^2]/Lp^2$, S = scan rate, C = scan count, B = background count. Structure by Patterson and Fourier syntheses in the non-centrosymmetric space group $Pn2_1a$, subsequent calculations in $Pnma$ after the presence of a mirror plane was verified, full-