

- 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, L_p and absorption corrections (analytical, relative transmission factors 0.572–1.000), 3764 independent reflections measured, 2683 with $I \geq 3\sigma(I)$, $\sigma^2(F_o^2) = [S^2(C + 4B) + (0.023F_o^2)^2]/L_p^2$, 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-

Table 1. Final atomic coordinates (fractional) and B_{eq} values (\AA^2)
$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
Sn	0.19756 (1)	$\frac{1}{4}$	0.39207 (2)	1.657 (4)
O(1)	0.2732 (1)	0.0769 (2)	0.6040 (2)	2.79 (5)
O(2)	0.3967 (1)	-0.0847 (2)	0.6989 (3)	2.97 (5)
C(1)	0.3198 (2)	$\frac{1}{4}$	0.1528 (5)	2.55 (8)
C(2)	0.0758 (2)	$\frac{1}{4}$	0.6275 (4)	3.2 (1)
C(3)	0.3646 (2)	0.0267 (2)	0.5974 (3)	2.65 (6)
H(1)	0.3882	$\frac{1}{4}$	0.2285	3.1
H(2)	0.3139	0.1623	0.0595	3.1
H(3)	0.1060	$\frac{1}{4}$	0.7775	3.9
H(4)	0.0322	0.1623	0.6076	3.9
H(5)	0.4158	0.0789	0.5047	3.2

Table 2. Bond lengths (\AA) and angles ($^\circ$) with *e.s.d.*'s in parentheses

Sn—C(1)	2.116 (2)	Sn—O(2)'	2.249 (1)
Sn—C(2)	2.097 (3)	O(1)—C(3)	1.249 (2)
Sn—O(1)	2.246 (2)	O(2)—C(3)	1.254 (2)
C(1)—Sn—C(2)	179.7 (2)	O(1)—Sn—O(2)'	92.78 (7)
C(2)—Sn—O(1)	86.09 (7)	O(1)''—Sn—O(2)'	173.07 (5)
C(2)—Sn—O(2)'	87.51 (7)	O(2)''—Sn—O(2)*	84.29 (8)
C(1)—Sn—O(1)	94.15 (7)	C(3)—O(1)—Sn	129.6 (1)
C(1)—Sn—O(2)'	92.23 (8)	C(3)—O(2)—Sn [#]	128.5 (1)
O(1)—Sn—O(1)''	89.4 (1)	O(1)—C(3)—O(2)	125.7 (2)

Symmetry operations: (') $\frac{1}{2} - x, -y, z - \frac{1}{2}$; (") $x, \frac{1}{2} - y, z$; (*) $\frac{1}{2} - x, \frac{1}{2} + y, z - \frac{1}{2}$; (#) $\frac{1}{2} - x, -y, \frac{1}{2} + z$.

matrix least squares on $w\Delta(F)^2$, $w = 4F_o^2/\sigma^2(F_o^2)$, hydrogen positions idealized from difference-map positions (C—H = 0.98 \AA , $B_{\text{H}} = 1.2$ times that of the parent C atom), scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–102, 149). *TEXSAN/TEXRAY* crystallographic software package (Molecular Structure Corporation, 1985); final $R = 0.030$, $wR = 0.037$ for 2683 reflections, $S = 1.79$, extinction parameter 1.65×10^{-7} , 47 parameters, $R = 0.058$ for all 3764 reflections, $\Delta/\sigma_{\text{max}} = 0.09$, maximum final difference density -1.1 to $+0.67 \text{ e \AA}^{-3}$.

Discussion. Final positional and equivalent isotropic thermal parameters are given in Table 1, and geometrical data appear in Table 2. Stereoviews of the polymer and of the coordination about the Sn atom are shown in Figs. 1 and 2.*

The structure of $(\text{CH}_3)_2\text{Sn}(\text{O}_2\text{CH})_2$ consists of infinite sheets (Fig. 1), the shortest distances between non-H atoms of adjacent layers being O(2)—C(2) ($\frac{1}{2} - x, -y, \frac{1}{2} + z$) = 3.007 (2) \AA and O(2)—C(1)

* Lists of anisotropic thermal parameters, torsion angles, intermolecular contacts, least-squares planes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53168 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

($\frac{1}{2} - x, -y, \frac{1}{2} + z$) = 3.148 (3) \AA . The coordination about the Sn atom is that of a relatively undistorted octahedron [exact mirror symmetry with Sn, C(1) and C(2) in the mirror plane] with Sn—O = 2.246 (2) and 2.249 (1) \AA , Sn—C = 2.097 (3) and 2.116 (2) \AA , and C—Sn—C = 179.7 (2) $^\circ$. The structure thus falls into the same category as those of $(\text{CH}_3)_2\text{SnF}_2$ and $(\text{CH}_3)_2\text{Sn}(\text{SO}_3\text{F})_2$ (see *Introduction*) although the Sn—C distances in the present structure (mean 2.107 \AA), while short, are probably longer than the corresponding distances of 2.08 (1) \AA reported for both $(\text{CH}_3)_2\text{SnF}_2$ and $(\text{CH}_3)_2\text{Sn}(\text{SO}_3\text{F})_2$. The small, but significant difference between the two Sn—C distances probably arises from intra-layer steric interactions between the formate H atom and the methyl H atom H(1) bound to C(1) [H(1)⋯H(5) = 2.31 \AA].

It is noteworthy that the Sn—O bonds involving each $\mu\text{-O}_2\text{CH}$ ligand are different even though the two Sn—O distances are essentially equal. The Sn—O(1) bond is *cis* to the formate H atom H(5) (with respect to the C—O bond) while Sn[#]—O(2) is *trans* to H(5). Sn and Sn[#] are displaced by -0.337 and -0.255 \AA from the plane of the formate ligand.

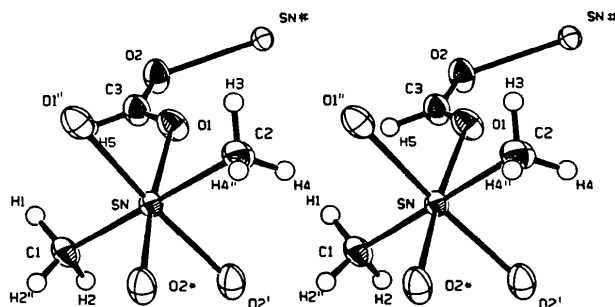


Fig. 1. Stereoview down the *a* axis of the structure of $[\text{Me}_2\text{Sn}(\text{O}_2\text{CH})_2]_n$.

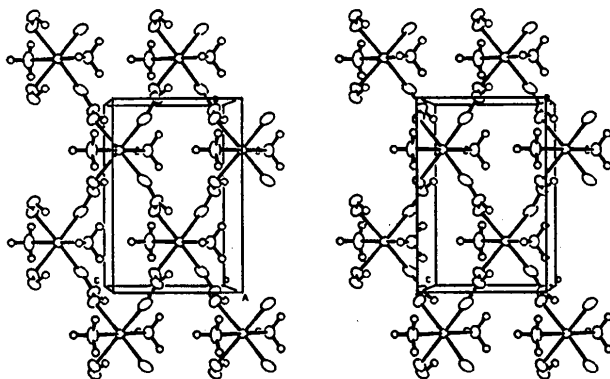


Fig. 2. Stereoview of the coordination about Sn in $[\text{Me}_2\text{Sn}(\text{O}_2\text{CH})_2]_n$. Shaded non-H atoms comprise the asymmetric unit; 50% probability thermal ellipsoids are shown for the non-H atoms.

An H(5)⋯H(5) separation of 2.58 Å suggests that compounds of the type (CH₃)₂Sn(O₂CR)₂ with R different from H would not have the same structure as that described here for the formate.

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

References

ALLEN, F. H., LERBSCHER, J. & TROTTER, J. (1971). *J. Chem. Soc. A*, pp. 2507–2509.

CHOW, Y. M. (1970). *Inorg. Chem.* **9**, 794–796.

DAVIES, A. G., MILLEDGE, H. J., PUXLEY, D. C. & SMITH, P. J. (1970). *J. Chem. Soc. A*, pp. 2862–2866.

KONNERT, J., BRITTON, D. & CHOW, Y. M. (1972). *Acta Cryst.* **B28**, 180–187.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX77381, USA.

OKAWARA, R. & ROCHOW, E. G. (1960). *J. Am. Chem. Soc.* **82**, 3285–3287.

SASAKI, Y., IMOTO, H. & NAGANO, O. (1984). *Bull. Chem. Soc. Jpn*, **57**, 1417–1418.

SCHLEMPER, E. O. & HAMILTON, W. C. (1966). *Inorg. Chem.* **5**, 995–998.

Acta Cryst. (1990). **C46**, 2093–2095

Structure of *trans*-Dichlorotetra(2-furaldehyde oxime)cobalt(II)

BY B. NORÉN AND Å. OSKARSSON

Inorganic Chemistry 1, Chemical Center, University of Lund, Box 124, S-221 00 Lund, Sweden

AND K. C. DASH AND M. MOHAPATRA

Department of Chemistry, Utkal University, Vani Vihar, 751004 Bhubaneswar, India

(Received 2 February 1990; accepted 5 March 1990)

Abstract. [CoCl₂(C₅H₅NO₂)₄], *M*_r = 574.24, triclinic, *P* $\bar{1}$, *a* = 6.9707 (8), *b* = 9.4963 (12), *c* = 10.7001 (11) Å, α = 77.691 (12), β = 70.739 (11), γ = 81.750 (11)°, *V* = 651.23 (10) Å³, *Z* = 1, *D*_x = 1.464 Mg m⁻³, λ (Mo *K*α) = 0.7107 Å, μ = 0.91 mm⁻¹, *F*(000) = 293, room temperature. The refinements converged to *R* = 0.036 for 2028 unique reflections with *I* > 3σ(*I*). The Co atom is bonded to four N atoms from four furaldehyde oxime ligands in a square-planar geometry and to two Cl atoms in a *trans* position thus forming an octahedral complex. The compound consists of van der Waals packed molecules. Each Cl atom forms two internal hydrogen bonds to the OH group from the two different oxime ligands. Acceptor–donor distances observed are: Co—Cl = 2.4377 (8) and Co—N = 2.171 (2)–2.173 (2) Å.

Introduction. Oxime compounds contain the group C=N—OH and possess two potential donor sites, *i.e.* the N and the O atoms. Hence, they can act as ambidentate ligands in coordinating metal ions (Mehrotra, Rai, Singh & Bohra, 1975; Chakravorty, 1974; Chatterjee, 1978; Nakamura, Konishi & Otsuka, 1979; Singh, Gupta, Srivastava & Mehrotra, 1974). The various modes of possible metal–oxime bonding include monodentate as well as bidentate oxime ligands coordinating *via* the N and/or O atom.

In the second case the oximes preferably react as the conjugate base, *i.e.* the oximate anion. Polynuclear complexes could also be formed in which different metal atoms are bonded to the N atom and the O atom, respectively. Intra- or intermolecular hydrogen bonds are also important for the formation of discrete or polymeric coordination compounds.

The present investigation was performed to establish the donor behaviour of furaldehyde oxime towards cobalt(II). Moreover there seem to be only few earlier X-ray investigations of cobalt(II) oxime compounds (Bekaroglu, Sarisaban, Koray & Ziegler, 1977; Alcock, Atkins, Curzon, Golding & Sellars, 1980; Kubel & Strähle, 1981, 1982).

Experimental. The title compound was prepared by reacting CoCl₂·6H₂O (1 mmol) with furaldehyde oxime (FDH, C₅H₅NO₂, 4 mmol) in ethanol at 333 K. Cooling to room temperature and recrystallization from a mixture of methanol and ethanol gave single crystals. The intensity data set was collected at room temperature with the aid of a CAD-4 diffractometer. The intensities in one half of the reflection sphere (3 ≤ θ ≤ 28°) were measured with ω–2θ scans. The ratio σ(*I*)/*I* requested in a scan was 0.028. Three standard reflections were measured at regular intervals. The intensities decreased linearly by 0.24% in one hour exposure time (total 34%). All the collected