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Trimethylstannyl 2-Furancarboxylate*

By Edward R. T. Tiekink

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, South Australia 5001, *Australia*

and G. K. Sandhu and S. P. Verma

Department of Chemistry, Guru Nanek Dev University, Amritsar 143005, *India*

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Abstract. $[C_8H_{12}O_3Sn]_n$, $M_r = 274{\cdot}9$, monoclinic, $P2_1/n$, $a = 10{\cdot}120\,(3)$, $b = 10{\cdot}145\,(1)$, $c = 10{\cdot}854\,(3)$ Å, $\beta = 106{\cdot}09\,(2)°$, $V = 1071\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}705$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 2{\cdot}173$ mm^{-1}, $F(000) = 536$, $T = 295\,(1)$ K, $R = 0{\cdot}034$ for 2233 observed reflections. The Sn atom in the title compound is five coordinate, forming there bonds to methyl groups [Sn—C: 2·129 (5) − 2·138 (5) Å], a bond to the carboxylate ligand [Sn—O(1) 2·191 (3) Å] and an intermolecular bond of 2·430(4) Å to a symmetry-related $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$ O atom, thereby generating an infinite polymeric chain; the O atom of the heterocyclic ring does not interact significantly with the Sn atom. The Sn-atom geometry is described as distorted trigonal bipyramidal with O atoms occupying axial positions; O(1)—Sn—O(2′) 172·3 (2)°.

Experimental. The title compound was prepared as in the literature (Sandhu & Verma, 1987). Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; $\omega{:}2\theta$ scan technique. Cell parameters on crystal (grown from ethanol solution) 0·42 × 0·42 × 0·52 mm by least squares on 25 reflections ($7 \leq \theta \leq 14°$) (de Boer & Duisenberg, 1984). Analytical absorption correction applied; max. and min. transmission factors 0·494 and 0·421 (Sheldrick, 1976). Total of 2743 reflections ($1{\cdot}5 \leq \theta \leq 27{\cdot}5°$) measured in the range $-13 \leq h \leq 13$, $0 \leq k \leq 13$, $-14 \leq l \leq 1$. No significant variation in the net intensities of three reference reflections ($\bar{3}16$, $\bar{2}15$, $\bar{1}34$) measured every 7200 s. 2460 unique reflections ($R_{\rm int} = 0{\cdot}023$) and 2233 satisfied $I \geq 2{\cdot}5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement of 111 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included at their calculated positions. Owing to the relatively high thermal motion associated with the atoms comprising the five-membered ring, the alternate conformation of the furan ring was investigated. The position of the O atom was confirmed by three independent means: (i) a higher residual was achieved when the O(3) and C(5) atoms were interchanged, (ii) the appearance of a residual electron density peak consistent with an H atom in the vicinity of C(5), and (iii) the orientation of O(3) towards the Sn atom. Some evidence of disorder in atomic parameters is evident, however; *e.g.* the rather short C(3)—C(4) bond distance of 1·283 (7) Å when compared to that found in 2-furoic acid (Gilmore, Mallinson & Speakman, 1983). At convergence $R = 0{\cdot}034$, $wR = 0{\cdot}047$, $w = [\sigma^2(F) + 0{\cdot}0031F^2]^{-1}$, $S = 1{\cdot}18$, $(\Delta/\sigma)_{\max} \leq 0{\cdot}002$, $\Delta\rho_{\max} = 0{\cdot}79$, $\Delta\rho_{\min} = -1{\cdot}00$ e Å^{-3}; extinction correction applied [coefficient 0·0025 (3)]. Scattering factors for H, C and O given in *SHELX*76 (Sheldrick, 1976) and those for neutral Sn corrected for f' and f'' (Hamilton & Ibers, 1974). All calculations on SUN4/280 computer system. Atomic parameters are given in Table 1, interatomic parameters in Table 2† and the numbering scheme used is shown in Fig. 1 [drawn with *ORTEP* (Johnson, 1971) at 25% probability ellipsoids].

Related literature. The polymeric structure found here for $[Me_3Sn(O_2CC_4H_3O)]_n$ (which arises as a result of carboxylate bridges) is similar to those reported triorganotin carboxylates as summarized recently (Ng, Chen Wei & Kumar Das, 1988). Noteworthy in the structure is the absence of any significant interaction between the O atom of the heterocyclic ring and the Sn atom [Sn···O 3·584 (4) Å].

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* Structural Chemistry of Organotin Carboxylates. III.

† Lists of structure factors, anisotropic thermal parameters, H-atom parameters and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52063 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates and B_{eq} values* (Å²)

$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$

	x	y	z	B_{eq}
Sn	0·27553 (2)	0·10188 (2)	0·19474 (2)	4·07
O(1)	0·3028 (3)	0·2527 (3)	0·0593 (3)	5·01
O(2)	0·2596 (5)	0·4142 (3)	0·1789 (4)	6·26
O(3)	0·2976 (6)	0·5952 (2)	0·0005 (4)	7·29
C(1)	0·2872 (4)	0·3709 (4)	0·0820 (4)	4·36
C(2)	0·3044 (4)	0·4630 (3)	−0·0207 (3)	4·16
C(3)	0·3109 (6)	0·6547 (4)	−0·1094 (5)	6·80
C(4)	0·3233 (6)	0·5682 (4)	−0·1918 (5)	6·01
C(5)	0·3230 (5)	0·4408 (5)	−0·1333 (4)	5·53
C(6)	0·3255 (8)	−0·0448 (5)	0·0736 (6)	7·46
C(7)	0·4400 (5)	0·1666 (6)	0·3517 (5)	6·27
C(8)	0·0639 (5)	0·1432 (6)	0·1725 (6)	6·75

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

Primed atoms are related by $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

Sn—O(1)	2·191 (3)	Sn—C(6)	2·138 (5)
Sn—C(7)	2·129 (5)	Sn—C(8)	2·130 (5)
Sn—O(2′)	2·430 (4)	Sn—O(2)	3·175 (4)
Sn—O(3)	3·584 (4)	C(1)—O(1)	1·244 (5)
C(1)—O(2)	1·240 (5)	C(1)—C(2)	1·502 (5)
C(2)—O(3)	1·365 (4)	C(2)—C(5)	1·306 (5)
C(3)—O(3)	1·377 (6)	C(3)—C(4)	1·283 (7)
C(4)—C(5)	1·440 (6)		
O(1)—Sn—C(6)	88·7 (2)	O(1)—Sn—C(7)	96·1 (2)
O(1)—Cn—C(8)	95·5 (2)	O(1)—Sn—O(2′)	172·4 (2)
C(6)—Sn—C(7)	116·2 (3)	C(6)—Sn—C(8)	118·0 (3)
C(6)—Sn—O(2′)	84·2 (2)	C(7)—Sn—C(8)	124·6 (2)
C(7)—Sn—O(2′)	89·7 (2)	C(8)—Sn—O(2′)	85.3 (2)
Sn—O(1)—C(1)	119·6 (2)	Sn—O(2′)—C(1′)	149·9 (3)
O(1)—C(1)—O(2)	125·5 (4)	O(1)—C(1)—C(2)	114·0 (3)
O(2)—C(1)—C(2)	120·6 (4)	C(1)—C(2)—O(3)	117·6 (3)
C(1)—C(2)—C(5)	131·5 (4)	O(3)—C(2)—C(5)	110·8 (4)
C(2)—O(3)—C(3)	105·2 (3)	O(3)—C(3)—C(4)	110·8 (4)
C(3)—C(4)—C(5)	107·1 (4)	C(4)—C(5)—C(2)	106·0 (4)

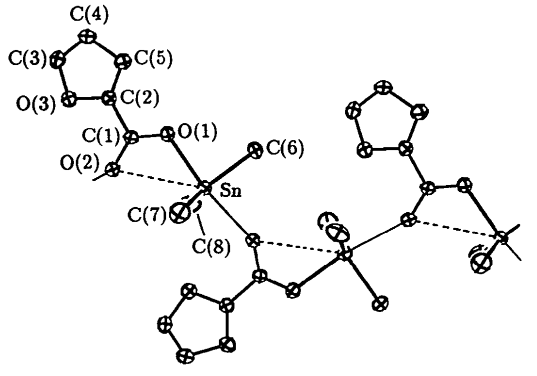

Fig. 1. Molecular structure and numbering scheme for $[Me_3Sn(O_2CC_4H_3O)]_n$ (Johnson, 1971).

References

Boer, J. L. de & Duisenberg, A. J. M. (1984). Enraf-Nonius CAD-4F diffractometer software update. February 1984. Groningen and Utrecht, The Netherlands.

Gilmore, C. J., Mallinson, P. R. & Speakman, J. C. (1983). *Acta Cryst.* C39, 1111–1113.

Hamilton, W. C. & Ibers, J. A. (1974). Editors. *International Tables for X-ray Crystallography*, Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1971). *ORTEP*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

Ng, S. W., Chen Wei & Kumar Das, V.G. (1988). *J. Organomet. Chem.* **345**, 59–64.

Sandhu, G. K. & Verma, S.P. (1987). *Polyhedron*, **6**, 587–592.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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Structure of *trans*-Dichlorotetrakis(diphenylphosphine)ruthenium(II) Chloroform (1/2)

By Ewan B. McAslan, Alexander J. Blake* and T. Anthony Stephenson

Department of Chemistry, University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

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Abstract. $[RuCl_2(C_{12}H_{11}P)_4].2CHCl_3$, $M_r = 1155·7$, triclinic, $P\overline{1}$, $a = 10·4314\ (11)$, $b = 10·5379\ (28)$, $c = 13·2413\ (8)$ Å, $\alpha = 70·106\ (13)$, $\beta = 73·208\ (7)$, $\gamma = 77·191\ (13)$°, $V = 1298·0$ Å³, $Z = 1$, $D_x = 1·460$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·865$ mm^{-1}, $F(000) = 586$, $T = 298$ K, $R = 0·0311$ for 3154 unique observed reflections. The Ru occupies a crystallographic inversion centre and is octahedrally coordinated by the resulting *trans* arrangement of the ligands.

Experimental. Compound prepared by isomerization of the corresponding *cis* complex in chloroform solution on standing in air, crystals obtained by partial evaporation of this solution. Pale-yellow ingot, 0·6 × 0·4 × 0·3 mm, CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell parameters by least-squares refinement on diffractometer angles

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* Author for correspondence.

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