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Structure of Triphenyltin Glyoxalate *O*-Methyloxime

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Abstract. *catena*-Poly[(triphenyltin)-μ-(methoxyiminoacetato-*O*:*O*′)], $[Sn(C_3H_4NO_3)(C_6H_5)_3]_n$, M_r = 452.08, orthorhombic, $P2_12_12_1$, a = 9.7484 (7), b = 10.3086 (6), c = 19.2656 (8) Å, V = 1936.0 (2) Å^3, Z = 4, D_x = 1.551 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 13.41 cm^{-1}, $F(000)$ = 904, T = 298 K, R = 0.032 for 2239 reflections [$I \geq 3\sigma(I)$]. The compound exists as a five-coordinate, *trans*-C_3SnO_2 trigonal bipyramidal carboxylate-bridged polymer.

Experimental. A stoichiometric amount of glyoxalic acid monohydrate was added to an ethanol solution of methoxyamine, prepared from equimolar amounts of sodium metal and methoxyamine hydrochloride in absolute ethanol. To this was added triphenyltin hydroxide, and the solution heated briefly. The solvent was then removed, and the solid obtained was purified by recrystallization from ethanol; m.p. 440–441 K. Analysis found: C 55.79, H 4.19, N 3.03%; calculated for $C_{21}H_{19}NO_3Sn$: C 55.79, H 4.23, N 3.09%.

A crystal measuring approximately 0.20 × 0.25 × 0.30 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. The cell dimensions were fixed from 25 reflections in the $17 \leq \theta \leq 19°$ thin shell. For data collection (ω–2θ-scan mode), the $2\theta_{max}$ value was set at 54°, with the hkl ranges being h 0–12, k 0–13, l 0–24; 2404 reflections were measured, of which 2239 obeyed $I \geq 3\sigma(I)$. Three standard reflections (377, 6,0,12, 4,4,13) monitored hourly showed negligible intensity variation. Direct phase determination yielded the heavy atom and only one phenyl ring; the remaining non-H atoms were derived from successive difference Fourier syntheses. The non-H atoms were refined anisotropically; H atoms were generated (C—H = 0.95 Å, B = 5 Å^2) and included in the structure-factor calculations. The refinement was based on F. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1). Computations were performed by using the *MolEN* structure determination system (Fair, 1990) on a DEC MicroVAX minicomputer. The final least-squares cycle was calculated with 235 variables; unit weights were used. The residuals were: R = 0.032 (R = 0.038 for all reflections) and S = 1.96; Δ/σ = 0.03; $(\Delta\rho)_{max}$ = 0.715 e Å^{-3} about 1 Å from the Sn atom. Fractional coordinates are given in Table 1* and bond dimensions in Table 2. Fig. 1 shows the asymmetric unit.

* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positional parameters, and complete bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55060 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0552]

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Table 1. *Positional parameters and equivalent isotropic displacement parameters* ($Å^2$)

$B_{eq} = (4/3)[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{1,} + ac(\cos\beta)B_{1,} + bc(\cos\alpha)B_{2,3}]$.

	x	y	z	B_{eq}
Sn	0.51399 (5)	0.06664 (5)	0.78956 (3)	2.261 (7)
O1	0.5984 (6)	−0.0926 (5)	0.8511 (3)	3.1 (1)
O2	0.5522 (5)	−0.2481 (5)	0.7748 (3)	3.0 (1)
O3	0.8697 (7)	−0.3398 (7)	0.9403 (4)	4.7 (2)
N	0.7781 (8)	−0.2506 (7)	0.9151 (4)	3.4 (1)
C1	0.3028 (8)	0.0222 (8)	0.8118 (4)	2.6 (1)
C2	0.2139 (9)	0.1313 (9)	0.8185 (5)	3.4 (2)
C3	0.0765 (9)	0.107 (1)	0.8415 (5)	3.9 (2)
C4	0.0340 (9)	−0.020 (1)	0.8569 (5)	4.2 (2)
C5	0.124 (1)	−0.123 (1)	0.8488 (6)	4.8 (2)
C6	0.2592 (9)	−0.1012 (8)	0.8252 (5)	3.5 (2)
C7	0.5926 (8)	0.1979 (8)	0.8654 (4)	2.6 (1)
C8	0.7090 (9)	0.2747 (9)	0.8539 (5)	3.5 (2)
C9	0.755 (1)	0.357 (1)	0.9071 (6)	4.5 (2)
C10	0.687 (1)	0.364 (1)	0.9700 (5)	4.6 (2)
C11	0.570 (1)	0.288 (1)	0.9813 (5)	5.0 (3)
C12	0.526 (1)	0.2035 (9)	0.9295 (4)	3.8 (2)
C13	0.6480 (8)	0.0255 (7)	0.7045 (5)	2.7 (1)
C14	0.608 (1)	0.0080 (9)	0.6378 (5)	3.9 (2)
C15	0.706 (1)	−0.015 (1)	0.5850 (5)	5.1 (2)
C16	0.846 (1)	−0.024 (1)	0.6045 (6)	4.9 (2)
C17	0.885 (1)	−0.005 (1)	0.6701 (6)	5.0 (2)
C18	0.7883 (9)	0.019 (1)	0.7205 (5)	4.1 (2)
C19	0.6145 (8)	−0.2068 (7)	0.8274 (4)	2.4 (1)
C20	0.7186 (9)	−0.2909 (8)	0.8611 (5)	3.2 (2)
C21	0.936 (1)	−0.294 (1)	1.0024 (6)	6.3 (3)

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

Sn—O1	2.185 (5)	Sn—O2′	2.367 (5)
Sn—C1	2.152 (7)	Sn—C7	2.134 (7)
Sn—C13	2.139 (7)	O1—C19	1.273 (8)
C19—O2	1.254 (8)	C19—C20	1.48 (1)
C20—N	1.26 (1)	N—O3	1.371 (8)
O3—C21	1.44 (1)		
O1—Sn—O2′	173.2 (2)	O1—Sn—C1	95.3 (2)
O1—Sn—C7	88.2 (2)	O1—Sn—C13	92.1 (2)
O2′—Sn—C1	90.9 (2)	O2′—Sn—C7	86.9 (2)
O2′—Sn—C13	85.7 (2)	C1—Sn—C7	110.0 (3)
C1—Sn—C13	134.0 (3)	C7—Sn—C13	115.6 (3)
Sn—O1—C19	123.1 (4)	Sn—O2′—C19′	146.0 (5)
O1—C19—O2	123.0 (7)	O1—C19—C20	117.8 (6)
O2—C19—C20	119.1 (6)	N—C20—C19	118.9 (7)
O3—N—C20	111.8 (7)	N—O3—C21	111.6 (7)

Symmetry code: (′) $1-x, \frac{1}{2}-y, \frac{3}{2}-z$.

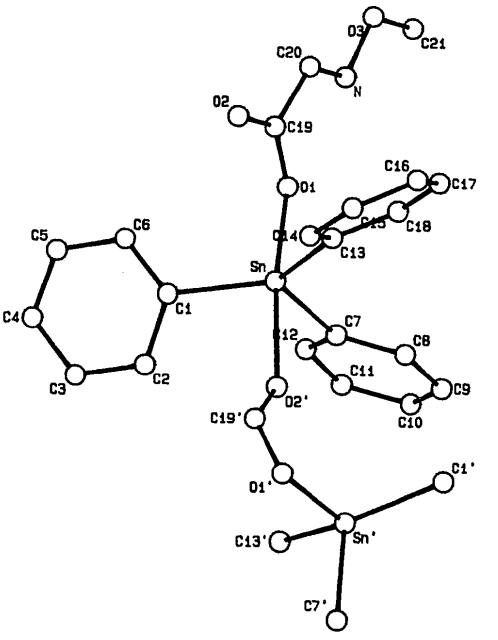

Fig. 1. Structure of the asymmetric unit of the polymer.

Related literature. Tiekink (1991) has reviewed the structures of triorganotin carboxylates.

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Structure of $[IrCl_2(CO)(PEt_3)_2(SOCl)]$

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Abstract. Carbonyldichloro(sulfur monoxide chloride)bis(triethylphosphine)iridium(III), $[IrCl_2(CO)(C_6H_{15}P)_2(SOCl)]$, $M_r = 610.96$, monoclinic, $P2_1/c$, $a = 14.5752$ (17), $b = 9.7304$ (8), $c = 15.163$ (5) Å, $\beta = 97.454$ (15)°, $V = 2132$ Å^3, $Z = 4$, $D_x = 1.903$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 7.09$ mm^{-1}, $F(000) = 1192$, $T = 183$ K, $R = 0.0448$ for 2803 unique observed reflections. The coordinated SOCl group has Ir—S 2.304 (3), S—Cl 2.168 (5), S—O 1.462 (10) Å and Ir—S—Cl 102.22 (17), Ir—S—O 113.9 (4), Cl—S—O 106.5 (4)°.

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