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Structure of Triphenyltin 2-(4-Pyridylthio)acetate

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Abstract. Triphenyl[2-(4-pyridylthio)acetato]tin, $C_{25}H_{21}NO_2SSn$, $M_r = 518.21$, monoclinic, $P2_1/a$, $a = 16.525$ (2), $b = 15.386$ (2), $c = 19.751$ (2) Å, $\beta = 113.82$ (1)°, $V = 4594$ (1) Å³, $Z = 8$, $D_x = 1.498$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 12.22$ cm⁻¹, $F(000) = 2080$, $T = 298$ K, $R = 0.034$ for 4981 $I \geq 3\sigma(I)$ reflections. The geometry around the Sn atoms in the two independent molecules is a distorted *trans*-C₃SnNO trigonal bipyramid.

Experimental. The carboxylate was prepared by heating equimolar amounts of triphenyltin hydroxide and 2-(4-pyridylthio)acetic acid in ethanol. Crystals suitable for single-crystal analysis were obtained by cooling the solution slowly. A crystal measuring 0.18 × 0.18 × 0.22 mm was used; the instrument was an Enraf–Nonius diffractometer. Cell dimensions were fixed from the 25 most intense reflections in the $14 \leq \theta \leq 16^\circ$ thin shell. Intensity data were collected up to a $2\theta_{max}$ of 50° (h 19, k 18, $\pm l$ 23) by using the ω - 2θ scan technique; 8395 data, 7755 unique data. Crystal decay was checked by monitoring three reflections hourly (loss in intensity = 15.7% for the 72h of collection), and a linear decay correction was applied to the data (min./max. correction = 1.0001/1.0889). The data were corrected for absorption effects by using the ψ -scan data (min./max. correction = 0.9454/0.9992). The structure was solved by vector methods from the 4981 reflections satisfying the $I \geq 3\sigma(I)$ criterion. All non-H atoms were refined anisotropically; H atoms were generated geometrically (C–H = 0.95 Å, $B = 5$ Å²) and were allowed to ride on their respective C atoms. Refinement based on F

for the 541 variables converged at $R = 0.034$, $wR = 0.038$; $S = 0.458$, $w = [\sigma(F)^2 + (0.02F)^2 + 1]^{-1}$ for the 541 variables, and the largest Δ/σ was less than 0.01. The highest and lowest ($\Delta\rho$) were 0.40 (5) and -0.13 (5) e Å⁻³. Scattering factors were taken from Tables 2.2B and 2.3.1. of *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were performed using the *MoIEN* structure determination package (Fair, 1990) on a DEC MicroVAX minicomputer. The atomic coordinates are listed in Table 1,* and selected bond distances and angles in Table 2. The two symmetry-independent molecules are depicted in Fig. 1.

Related literature. The two molecules of the title compound are bridged through their pyridyl groups to form polymeric helical chains parallel to b . The coordination polyhedron around the Sn atom is a distorted *trans*-C₃SnNO trigonal bipyramid; one apical position is occupied by the carboxyl O and the other by the N belonging to a symmetry-related ($\frac{1}{2} - x, y - \frac{1}{2}, z$; $1\frac{1}{2} - x, y - \frac{1}{2}, z$) anionic group. The distortion from ideal geometry is shown by the large difference between the axial Sn←:N and Sn—O distances. The Sn←:N distance is, however, somewhat shorter than that in the pyridyl-bridged tri-

* Lists of structure factors, anisotropic thermal parameters, calculated H positional parameters, and complete bond lengths and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55257 (61 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0579]

Table 1. Positional parameters and their estimated standard deviations

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

	x	y	z	$B_{eq}(\text{\AA}^2)$
Sn1a	0.20143 (2)	0.22048 (3)	0.95893 (2)	3.782 (8)
Sn1a	-0.0549 (9)	0.4386 (1)	0.86046 (9)	5.29 (4)
O1a	0.0791 (2)	0.2898 (3)	0.9036 (2)	5.0 (1)
O2a	0.0928 (3)	0.2877 (3)	0.7967 (2)	6.0 (1)
N1a	0.1627 (3)	0.6392 (3)	0.9599 (3)	4.3 (1)
C1a	0.1647 (3)	0.1063 (4)	0.8932 (3)	3.9 (1)
C2a	0.0769 (4)	0.0837 (5)	0.8566 (3)	5.5 (2)
C3a	0.0514 (5)	0.0096 (5)	0.8147 (4)	7.3 (2)
C4a	0.1133 (5)	-0.0421 (5)	0.8056 (4)	7.0 (2)
C5a	0.1998 (5)	-0.0213 (5)	0.8391 (3)	6.2 (2)
C6a	0.2261 (4)	0.0518 (4)	0.8835 (3)	5.1 (2)
C7a	0.2845 (3)	0.3124 (4)	0.9393 (3)	3.8 (1)
C8a	0.3007 (4)	0.3066 (4)	0.8753 (3)	4.7 (1)
C9a	0.3497 (4)	0.3677 (5)	0.8591 (3)	5.8 (2)
C10a	0.3852 (4)	0.4363 (5)	0.9060 (4)	6.0 (2)
C11a	0.3709 (4)	0.4431 (5)	0.9691 (4)	6.0 (2)
C12a	0.3213 (4)	0.3813 (4)	0.9860 (3)	5.1 (2)
C13a	0.1742 (4)	0.2255 (4)	1.0558 (3)	4.5 (1)
C14a	0.2337 (5)	0.2552 (5)	1.1224 (3)	6.0 (2)
C15a	0.2118 (6)	0.2590 (5)	1.1825 (4)	8.6 (2)
C16a	0.1327 (5)	0.2303 (6)	1.1789 (4)	10.3 (2)
C17a	0.0749 (5)	0.1998 (6)	1.1136 (4)	9.0 (2)
C18a	0.0931 (4)	0.1975 (5)	1.0517 (4)	6.4 (2)
C19a	0.0558 (4)	0.3109 (4)	0.8363 (3)	4.5 (1)
C20a	-0.0247 (4)	0.3703 (4)	0.8026 (3)	4.7 (2)
C21a	0.0299 (3)	0.5147 (4)	0.8949 (3)	4.1 (1)
C22a	0.1081 (3)	0.5124 (4)	0.8850 (3)	4.5 (1)
C23a	0.1706 (4)	0.5748 (4)	0.9175 (3)	4.6 (1)
C24a	0.0871 (4)	0.6410 (4)	0.9694 (3)	4.8 (2)
C25a	0.0206 (4)	0.5814 (4)	0.9381 (3)	4.8 (1)
Sn1b	0.73378 (2)	0.20656 (3)	0.53764 (2)	3.962 (8)
Sn1b	0.57503 (9)	0.4266 (1)	0.63542 (8)	4.92 (4)
O1b	0.6663 (2)	0.2786 (3)	0.5921 (2)	4.45 (9)
O2b	0.7850 (2)	0.2785 (3)	0.6988 (2)	5.0 (1)
N1b	0.7041 (3)	0.6221 (4)	0.5379 (3)	5.6 (1)
C1b	0.7577 (4)	0.0966 (4)	0.6084 (3)	4.4 (1)
C2b	0.7053 (4)	0.0843 (4)	0.6469 (3)	5.6 (2)
C3b	0.7181 (6)	0.0138 (5)	0.6927 (4)	8.5 (2)
C4b	0.7827 (6)	-0.0439 (5)	0.7013 (5)	10.5 (3)
C5b	0.8354 (6)	-0.0317 (5)	0.6651 (5)	9.5 (3)
C6b	0.8235 (5)	0.0377 (4)	0.6189 (4)	6.4 (2)
C7b	0.8340 (3)	0.2995 (4)	0.5529 (3)	4.0 (1)
C8b	0.9137 (3)	0.2961 (4)	0.6134 (3)	4.6 (1)
C9b	0.9776 (4)	-0.3576 (5)	0.6239 (3)	5.4 (2)
C10b	0.9627 (4)	0.4247 (5)	0.5741 (4)	6.0 (2)
C11b	0.8856 (4)	0.4294 (5)	0.5150 (4)	7.0 (2)
C12b	0.8218 (4)	0.3663 (5)	0.5033 (4)	5.9 (2)
C13b	0.6134 (4)	0.2077 (4)	0.4396 (3)	4.3 (1)
C14b	0.6089 (5)	0.2161 (7)	0.3691 (4)	8.6 (2)
C15b	0.5294 (5)	0.2084 (8)	0.3085 (4)	11.1 (3)
C16b	0.4541 (5)	0.1937 (7)	0.3170 (4)	8.7 (3)
C17b	0.4566 (4)	0.1856 (6)	0.3847 (4)	7.3 (2)
C18b	0.5355 (4)	0.1932 (5)	0.4458 (3)	5.7 (2)
C19b	0.7096 (3)	0.3004 (4)	0.6604 (3)	4.2 (1)
C20b	0.6614 (4)	0.3568 (4)	0.6946 (3)	4.6 (1)
C21b	0.6297 (4)	0.5012 (4)	0.6024 (3)	4.3 (1)
C22b	0.7176 (4)	0.4943 (5)	0.6116 (3)	5.9 (2)
C23b	0.7494 (4)	0.5555 (5)	0.5786 (4)	6.4 (2)
C24b	0.6204 (4)	0.6293 (4)	0.5315 (3)	5.3 (2)
C25b	0.5821 (4)	0.5716 (4)	0.5627 (3)	5.2 (2)

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

Sn1a—O1a	2.153 (4)	Sn1b—O1b	2.144 (4)
Sn1a—N1a ⁱ	2.503 (5)	Sn1b—N1b ⁱⁱ	2.490 (5)
Sn1a—C1a	2.122 (6)	Sn1b—C1b	2.127 (6)
Sn1a—C7a	2.113 (6)	Sn1b—C7b	2.115 (6)
Sn1a—C13a	2.137 (6)	Sn1b—C13b	2.143 (6)
O1a—Sn1a—N1a ⁱ	171.1 (2)	O1b—Sn1b—N1b ⁱⁱ	173.5 (2)
O1a—Sn1a—C1a	97.0 (2)	O1b—Sn1b—C1b	94.5 (2)
O1a—Sn1a—C7a	97.0 (2)	O1b—Sn1b—C7b	96.6 (2)
O1a—Sn1a—C13a	86.7 (2)	O1b—Sn1b—C13b	88.2 (2)
N1a ⁱ —Sn1a—C1a	86.5 (2)	N1b ⁱⁱ —Sn1b—C1b	88.1 (2)
N1a ⁱ —Sn1a—C7a	88.3 (2)	N1b ⁱⁱ —Sn1b—C7b	87.1 (2)
N1a ⁱ —Sn1a—C13a	84.4 (2)	N1b ⁱⁱ —Sn1b—C13b	85.4 (2)
C1a—Sn1a—C7a	119.2 (2)	C1b—Sn1b—C7b	121.1 (2)
C1a—Sn1a—C13a	117.8 (2)	C1b—Sn1b—C13b	117.3 (2)
C7a—Sn1a—C13a	121.8 (2)	C7b—Sn1b—C13b	120.7 (2)

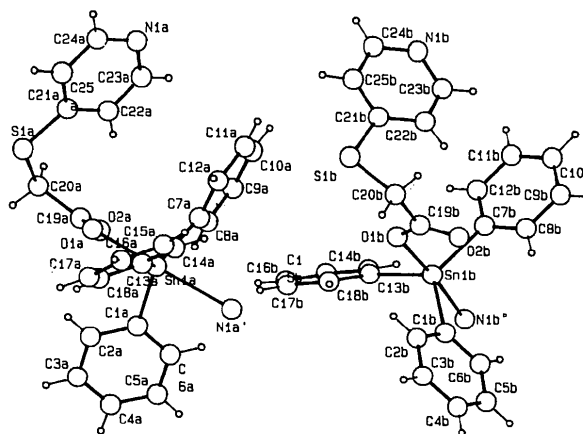
Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, z$; (ii) $1\frac{1}{2} - x, y - \frac{1}{2}, z$.

Fig. 1. Atom-numbering scheme.

Kumar Das, 1989; Tiekink, 1991). This feature is found in the isoelectronic triphenyltin 2-(2-pyrimidylthio)acetate (Ng, Kumar Das, Yip & Mak, 1992); a chain structure resulting from bridging by the pyridyl group is not realized, possibly because the pyrimidyl N is not a sufficiently strong Lewis base to coordinate to Sn.

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phenyltin nicotinate [2.568 (7) Å] (Ng, Kumar Das, van Meurs, Schagen & Straver, 1989) and triphenyltin isonicotinate [2.582 (3) Å] (Ng & Kumar Das, 1992) compounds.

Triorganotin carboxylates generally adopt carboxylate-bridged chain structures (Ng, Chen &