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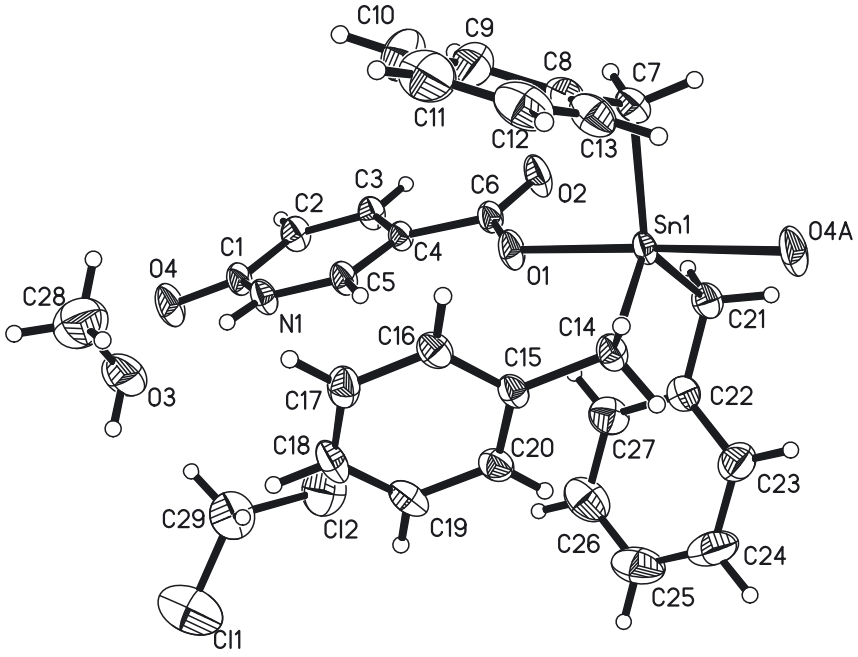
Crystal structure of *catena*-poly[tribenzyl-κ^1*C*-(μ$_2$-6-oxidopyridin-1-ium-3-carboxylato-κ^2*O*:*O*')tin(IV)-dichloromethane-methanol (1/1/1), $C_{29}H_{31}Cl_2NO_4Sn$



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Abstract

$C_{29}H_{31}Cl_2NO_4Sn$, monoclinic, $P2_1/n$ (no. 14), a = 10.638(4) Å, b = 17.168(7) Å, c = 18.399(5) Å, β = 120.573(16)°, V = 2893.1(18) Å^3, Z = 4, $R_{gt}(F)$ = 0.0314, $wR_{ref}(F^2)$ = 0.0875, T = 298 K.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.49 × 0.35 × 0.26 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.10 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14894, 5365, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 4501
$N(param)_{refined}$:	335
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Source of material

A mixture of tribenzyltin oxide (1.6003 g, 2.0 mmol) and 6-hydroxy-nicotinic acid (0.5564 g, 4.0 mmol), in methanol (40 ml) was heated under reflux for 6 h. The obtained clear

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.15603 (2)	0.25443 (2)	0.99415 (2)	0.03244 (9)
O1	0.3616 (2)	0.26285 (12)	0.99845 (14)	0.0446 (5)
O2	0.5139 (3)	0.23656 (14)	1.13394 (15)	0.0586 (6)
O4	0.9140 (3)	0.25531 (14)	0.97169 (18)	0.0624 (7)
N1	0.6867 (2)	0.27838 (15)	0.94526 (15)	0.0416 (5)
H1	0.664332	0.294523	0.895866	0.050*
C1	0.8280 (3)	0.25667 (16)	0.99955 (19)	0.0402 (7)
C2	0.8619 (3)	0.23765 (17)	1.0825 (2)	0.0437 (7)
H2	0.957580	0.225427	1.123133	0.052*
C3	0.7563 (3)	0.23705 (16)	1.10335 (19)	0.0400 (6)
H3	0.781090	0.224768	1.158304	0.048*
C4	0.6087 (3)	0.25482 (14)	1.04280 (18)	0.0312 (6)
C5	0.5800 (3)	0.27601 (17)	0.96456 (17)	0.0379 (6)
H5	0.485028	0.289113	0.923545	0.045*
C6	0.4885 (3)	0.25093 (14)	1.06240 (19)	0.0368 (6)
C7	0.1870 (4)	0.36473 (17)	1.05591 (19)	0.0466 (7)
H7A	0.114665	0.371213	1.072650	0.056*
H7B	0.283002	0.366407	1.106361	0.056*
C8	0.1730 (4)	0.42922 (18)	0.99772 (19)	0.0480 (7)
C9	0.2933 (5)	0.4664 (2)	1.0043 (3)	0.0712 (10)
H9	0.386388	0.451898	1.046705	0.085*
C10	0.2769 (6)	0.5254 (3)	0.9481 (3)	0.0953 (15)
H10	0.358764	0.550373	0.953540	0.114*
C11	0.1404 (7)	0.5467 (2)	0.8850 (3)	0.0902 (15)
H11	0.129761	0.585651	0.847146	0.108*
C12	0.0212 (6)	0.5112 (2)	0.8777 (3)	0.0783 (12)
H12	−0.071270	0.526278	0.835053	0.094*
C13	0.0353 (4)	0.4528 (2)	0.9328 (2)	0.0594 (9)
H13	−0.047824	0.428745	0.926779	0.071*
C14	0.0323 (3)	0.25772 (16)	0.85884 (18)	0.0410 (7)
H14A	−0.030413	0.212217	0.838847	0.049*
H14B	−0.030255	0.303296	0.841483	0.049*
C15	0.1211 (3)	0.25991 (15)	0.81650 (17)	0.0355 (6)
C16	0.1675 (3)	0.32986 (17)	0.79970 (17)	0.0419 (7)
H16	0.142100	0.376700	0.814260	0.050*
C17	0.2515 (3)	0.33054 (19)	0.76137 (19)	0.0484 (7)
H17	0.280408	0.377822	0.749851	0.058*
C18	0.2921 (4)	0.2619 (2)	0.7403 (2)	0.0510 (8)
H18	0.347915	0.262578	0.714524	0.061*
C19	0.2491 (3)	0.1925 (2)	0.75797 (18)	0.0508 (8)
H19	0.277176	0.145837	0.744688	0.061*
C20	0.1645 (3)	0.19142 (18)	0.79533 (18)	0.0440 (7)
H20	0.136080	0.143833	0.806526	0.053*
C21	0.1906 (3)	0.13886 (17)	1.04616 (19)	0.0448 (7)
H21A	0.285568	0.135967	1.097301	0.054*
H21B	0.116831	0.126948	1.060592	0.054*
C22	0.1829 (3)	0.08103 (17)	0.98349 (19)	0.0451 (7)
C23	0.0519 (4)	0.0442 (2)	0.9271 (2)	0.0626 (9)
H23	−0.032079	0.055173	0.928970	0.075*
C24	0.0457 (6)	−0.0087 (2)	0.8682 (3)	0.0843 (14)
H24	−0.042286	−0.033141	0.831453	0.101*
C25	0.1663 (6)	−0.0254 (2)	0.8634 (3)	0.0851 (14)
H25	0.160854	−0.061045	0.823830	0.102*
C26	0.2961 (5)	0.0108 (2)	0.9175 (3)	0.0795 (12)
H26	0.378872	0.000039	0.914375	0.095*
C27	0.3039 (4)	0.0634 (2)	0.9769 (2)	0.0607 (9)
H27	0.392487	0.087463	1.013261	0.073*
O3	0.6395 (3)	0.33764 (18)	0.78776 (16)	0.0820 (8)
H3A	0.609829	0.315078	0.742637	0.123*
C28	0.6180 (6)	0.4162 (3)	0.7735 (3)	0.1045 (16)
H28A	0.515588	0.426538	0.737753	0.157*
H28B	0.669587	0.434351	0.746579	0.157*
H28C	0.653664	0.442832	0.826360	0.157*
Cl1	0.6663 (2)	0.07753 (11)	0.72801 (10)	0.1408 (7)
Cl2	0.63999 (17)	0.08200 (8)	0.87653 (10)	0.1133 (4)
C29	0.6466 (5)	0.1346 (3)	0.7981 (3)	0.0893 (13)
H29A	0.557529	0.164824	0.767348	0.107*
H29B	0.727612	0.170925	0.824303	0.107*

solution was evaporated under vacuum. The product was crystallized from a mixture of dichloromethane/ethanol (1:1). Yield 1.8892 g, 73%, m.p. 462 K.

Experimental details

H atoms were included in the riding model approximation with C–H 0.93–0.97 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The O- and N-bound H atoms were refined with O–H = 0.82+−0.01 Å and N–H = 0.86%A, and with U_{iso}(H) = 1.5U_{eq}(O) or 1.2U_{eq}(N).

Comment

Studies on organotin(IV) compounds having carboxylate ligands with additional donor atom, such as N, O, or S, revealed new structural types which may lead to compounds with different activities [4–8]. In order to exploring whether or not the hetero-atoms (N, O or S) of the carboxylic acid derivatives influence the coordination mode and extension of triaryltin compounds with heteroatom carboxylate ligands this study was undertaken.

In the title compound, the Sn atom exists in a distorted trigonal bipyramidal, surrounded axially by two oxygen atoms and equatorialy by the carbon atoms of the benzyl groups. It has an infinite one-dimensional polymeric chain structure, due to the Sn1–O1 and Sn1–O4A [symmetry operation: *x*–1, *y*, *z*] distances of 2.153(2) and 2.3391(2) Å, respectively, comparable to a related structure [9]. Translational pseudosymmetry is found in the title structure (klassengleich, c/2). The pseudosymmetry is mainly caused by the solvent molecules. Such a translational

symmetry is an often found feature of reported structures [10].

In detail the distortions from ideal trigonal bipyramidal symmetry for the compound is reflected by the bond angles around the tin atom: C7–Sn1–O1 92.39(10)°, C7–Sn1–O4A 87.01(10)°, C14–Sn1–O1 93.04(10)°, C14–Sn1–O4A 80.10(11)°, C21–Sn1–O1 96.01(10)° and C21–Sn1–O4A 90.48(10)°, respectively. The bond angle O1–Sn1–O4A is 172.05(8)° which is deviating from the linear angle by 7.95°. The sum of the C7–Sn1–C21 129.26(13)°, C14–Sn1–C7 115.45(11)° and C14–Sn1–C21 113.93(11)° bond angles is 358.64°, which shows that the atoms of Sn1, C7, C14 and C21 are almost in the same plane. Therefore the tin atom forms a distorted trigonal bipyramidal structure. Due to the protonation at N, a coordination at this site is impossible, which is different to the tribenyltin 4-picolinate and similar others [11–14].

There are strong intermolecular N–H···O hydrogen bonds (N1–O3 = 2.865 Å) involving pyridinium–N–H and the methanol hydroxyl O atom. There are also intermolecular O–H···O hydrogen bonds ($O3–O2^{i}$ = 2.752 Å) [symmetry code: x, $-y+0.5$, $z-0.5$] between the methanol hydroxyl O atom and the picolinic carboxyl O atom noncoordinating to Sn center.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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