CRYSTAL STRUCTURE OF TRIBUTYLTIN *N*-PHTHALOYLLEUCINATE

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Abstract

The two symmetry-independent molecules of tributyltin *N*-phthaloylleucinate are linked by long tin-amide bridges [Sn-O = 2.897(7), 2.960(7) Å] into a linear chain that propagates along the *a-c* diagonal of the monoclinic cell. The tin atoms are five-coordinate in *trans*-C_3SnO_2 trigonal bipyramidal geometries.

Discussion

The tributyltin derivative of *N*-phthaloylglycine exists as two symmetry-independent hydrates, and the coordinated water molecules engage in hydrogen bonding interactions that consolidate the crystal structure [1]. The incorporation of an *i*-propyl group in the protected amino acid appears to render the tin atom less Lewis acidic in the resulting carboxylate; unlike trialkyltin carboxylates that generally are linked by strong carboxylate bridges [2,3], the molecules of tributyltin *N*-phthaloylleucinate are connected by a long tin-amide interaction into a linear chain. A similar long interaction is also found in tricyclohexyltin *N*-phthaloylglycinate [4].

Table 1. Crystal data for tributyltin *N*-phthaloylleucinate.

Formula	$C_{26}H_{41}NO_4Sn$
Crystal system	Monoclinic
Formula weight	550.29
Space group	$P2_1$
Cell constants	a = 10.609(1), b = 23.023(1), c = 11.951(1) Å; β = 101.73(1)°
Z	4
Temperature	25°C
Density (calculated)	1.279 g cm^{-3}
μ	0.921 mm^{-1}
F(000)	1144
Diffractometer	*CAD*-4 (λ = 0.71073 Å)
Theta range for data collection	1.74 to 24.98°.
Index ranges	$0 \le h \le 12$, $-27 \le k \le 0$, $-14 \le l \le 13$
Total refelctions measured	5455
Unique reflections measured	5166 (R_{int} = 0.028)
Transmission factors	0.724 - 0.803
Data / restraints / parameters	5166 / 246 / 579
Reflections with $I > 2\sigma(I)$	4278
R (wR) for $I > 2\sigma(I)$ data	0.042 (0.110)
R (wR) for all data	0.057 (0.121)
Goodness-of-fit	1.039
ρ, eÅ^{-3}	- 0.371 to 495
Programs used	*SHELXS*-97 [6] *SHELXL*-97 [7]
Deposition number	CCDC

Experimental

N-Phthaloylleucinate was synthesized by melting phthalic anhydride with leucine according to the procedure for preparing *N*-phthaloylglycine [5]. The acid and bis(tributyltin) oxide (2:1 molar stoichiometry) were heated in a small volume of ethanol to afford the organotin carboxylate, which was isolated and recrystallized from ethanol. A specimen, 0.22 x 0.14 x 0.14 mm, was used in the diffraction measurements. The refinement of the crystal structure necessitated imposing several restraints as the butyl groups in molecule *a* were disordered. Each butyl group was refined as two butyl groups sharing a common C_α atom. Distances in all butyl groups and in the two *i*-propyl groups were restrained (C-C = 1.54±0.01; C···C = 2.52±0.02 Å, and the temperature factors in these groups were restrained to be nearly isotropic by an *ISOR* 0.02 command.

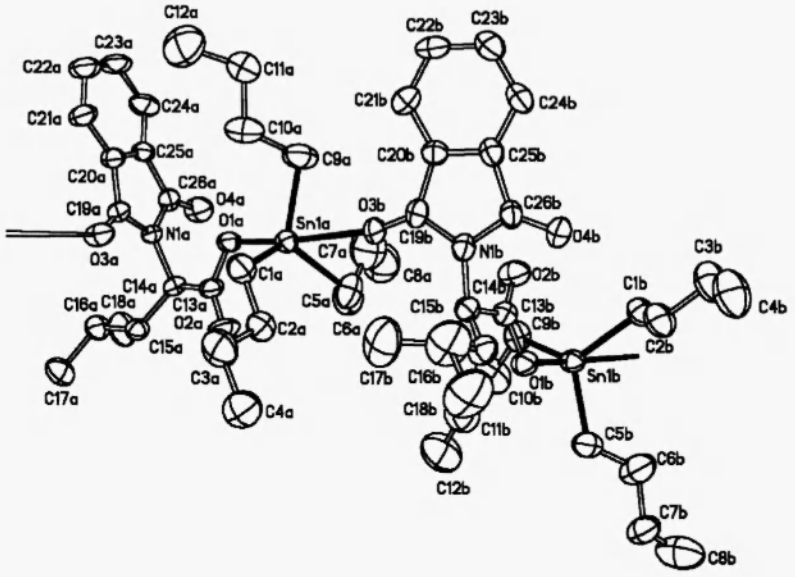

Figure 1. *ORTEP* [8] plot of tributyltin *N*-phthaloylleucinate. Selected bond distances and angles: Sn1*a*-C1*a* = 2.14(1), Sn1*a*-C5*a* = 2.13(1), Sn1*a*-C9*a* = 2.14(1), Sn1*a*-O1*a* = 2.068(6), Sn1*a*-O3*b* = 2.960(7), Sn1*b*-C1*b* = 2.14(1), Sn1*b*-C5*b* = 2.10(1), Sn1*b*-C9*b* = 2.12(1), Sn1*b*-O1*b* = 2.139(7), Sn1*b*-O3*a*^{i} = 2.897(7) Å; C1*a*-Sn1*a*-C5*a* = 119.0(5), C1*a*-Sn1*a*-C9*a* = 115.7(6), C1*a*-Sn1*a*-O1*a* = 102.7(4), C1*a*-Sn1*a*-O3*b* = 79.2(4), C5*a*-Sn1*a*-C9*a* = 115.3(6), C5*a*-Sn1*a*-O1*a* = 105.2(4), C5*a*-Sn1*a*-O3*b* = 77.2(4), C9*a*-Sn1*a*-O1*a* = 93.4(4), C9*a*-Sn1*a*-O3*b* = 81.9(4), O1*a*-Sn1*a*-O3*b* = 175.3(2), C1*b*-Sn1*b*-C5*b* = 114.3(6), C1*b*-Sn1*b*-C9*b* = 123.5(5), C1*b*-Sn1*b*-O1*b* = 100.7(4), C1*b*-Sn1*b*-O3*a*^{i} = 76.9(3), C5*b*-Sn1*b*-C9*b* = 115.7(7), C5*b*-Sn1*b*-O1*b* = 91.6(5), C5*b*-Sn1*b*-O3*a*^{i} = 89.0(4), C9*b*-Sn1*b*-O1*b* = 102.4(4), C9*b*-Sn1*b*-O3*a*^{i} = 79.4(4), O1*b*-Sn1*b*-O3*a*^{i} = 177.5(3)°. Translational code: ^{i} = 1 + *x*, *y*, 1 + *z*.

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