

Synthesis and Crystal Structures of the Triphenylstannyl Esters of *N*-Phthaloyl-glycine and -leucine

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ABSTRACT

Triphenyltin N-phthaloylglycinate crystallizes from ethanol as a bridged, cyclic hexamer (rhombohedral $R\bar{3}$ space group, $a = 19.928(3)$ Å, $\alpha = 116.47(2)^\circ$; $Z = 6$) and triphenyltin N-phthaloylleucinate as a monomeric molecule (orthorhombic $Pbca$ space group, $a = 18.032(6)$, $b = 26.992(7)$, $c = 11.705(4)$ Å; $Z = 8$).

INTRODUCTION

Organotin(IV) derivatives of amino acids are best realized when the amino group is protected, thereby enhancing the solubility of the amino acid in organic solvents [1]. The phthaloyl derivative of glycine is known to possess auxin-like properties [2] and was therefore chosen in this study, along with the leucine analogue, to form the title compounds by condensation with triphenyltin hydroxide. Structurally, the triphenyltin *N*-phthaloylaminoacetates can be expected to form helical chains through

carboxylate bridges, a feature typical of triorganotin alkanooates [3], but triphenyltin *N*-phthaloylglycinate is unusual in crystallizing from ethanol as a cyclic hexamer.** The structure of this hexamer and that of triphenyltin *N*-phthaloylleucinate are described here.

EXPERIMENTAL

Synthesis

The method of Vogel [5] was used to prepare the phthaloyl derivatives of glycine and leucine. Triphenyltin *N*-phthaloylglycinate slowly precipitated as well-formed crystals when hot ethanolic solutions of triphenyltin hydroxide (3.67 g, 10 mmol) and *N*-phthaloylglycine (2.05 g, 10 mmol) were mixed and the mixture allowed to cool; mp 196–197°C. Anal. Calcd. for $C_{28}H_{21}NO_4Sn$: C, 60.68; H, 3.81; N, 2.52%. Found: C, 60.51; H, 3.71; N, 2.43%. The compound is soluble in ethanol, but a non-crystalline material results if it is recrystallized from this solvent. A saturated solution of $(C_6H_5)_3SnOC(O)CH_2NC(O)C_6H_4C(O)$ in $CDCl_3$ was used for the ^{13}C NMR spectrum (chemical shift δ in ppm (coupling constant J_{Sn-C} in Hz)): $C_{ipso} = 137.4$ (643.3), $C_{ortho} = 136.7$ (48.4), $C_{meta} = 129.1$ (64.5), $C_{para} = 130.3$ (13.2), $C(O)O = 172.5$, $C(O) = 167.6$, $C_6H_4 = 123.5, 132.3, 134.0$, $CH_2 = 39.4$. The com-

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**A preliminary report of the crystal structure of this compound is given in Ref. 4.

pound shows a ^{119}Sn NMR chemical shift $\delta = -90.5$ ppm, relative to $(\text{CH}_3)_3\text{Sn}^{\text{an}}$ (conc. 1% w/v CDCl_3). The tin-119m Mössbauer isomer shift (IS) and quadrupole splitting (QS) values at 80 K are 1.30 and 3.86 mm s^{-1} , respectively; $d[\ln f(T)]/dT$ (a) for 80 K $\leq T \leq 130$ K (6 points, corr. coeff. -0.998) is -0.0153 K^{-1} .

Triphenyltin hydroxide was similarly reacted with *N*-phthaloylleucine to afford triphenyltin *N*-phthaloylleucinate, $(\text{C}_6\text{H}_5)_3\text{SnOC}(\text{O})\text{CH}(\text{i-C}_4\text{H}_9)\text{NC}(\text{O})\text{C}_6\text{H}_4\text{C}(\text{O})$, mp 199–200°C. Anal. Calcd. for $\text{C}_{32}\text{H}_{29}\text{NO}_4\text{Sn}$: C, 62.98; H, 4.78; N, 2.29%. Found: C, 63.06; H, 4.84; N, 2.11%. Its crystal habit is unchanged by recrystallization from ethanol in which solvent the compound dissolves sparingly; its poor solubility in CDCl_3 precluded NMR measurements. Tin-119m Mössbauer data (80 K): IS 1.24, QS 2.67 mm s^{-1} ; a is -0.0164 K^{-1} in the 80 K $\leq T \leq 130$ K temperature range (6 points, corr. coeff. -0.998).

X-ray Crystallography

A $0.44 \times 0.49 \times 0.83$ mm prismatic triphenyltin *N*-phthaloyllycinate crystal was mounted on a computer-controlled Siemens AED diffractometer that was used to measure cell dimensions and collect intensity data with the use of nickel-filtered $\text{Cu-K}\alpha$ radiation ($\lambda 1.54178 \text{ \AA}$). Data collection was carried out with a triclinic cell of dimensions $a = 19.928$, $b = 20.985$, $c = 11.374 \text{ \AA}$, $\alpha = 111.18$, $\beta = 79.04$, and $\gamma = 121.77^\circ$. Before starting on structure analysis the transformation matrix $1\ 0\ 0/0\ 1\ 1/1\ 1\ 0$ was applied to give the final primitive rhombohedral cell. The space group $R\bar{3}$ was chosen on the basis of the Laue class ($\bar{3}$), the lack of any systematic absences, and the statistical analysis of the normalized structure factors. The unit-cell parameters and standard deviations obtained from the setting angles of 30 intense, accurately measured ($20^\circ < \theta < 38^\circ$) reflections were $a = 19.928(3) \text{ \AA}$ and $\alpha = 116.47(2)^\circ$ for $Z = 6$. A set of 14331 reflections was recorded by the θ - 2θ scan technique for $3^\circ < \theta < 70^\circ$. After merging duplicate and symmetry-equivalent data ($R_{\text{int}} = 0.0542$) there remained 3808 unique observed [$I > 2\sigma(I)$] reflections that were used for structure analysis. A standard reflection that was measured after every 50 reflections showed a small decrease in intensity during data collection. A correction for this effect was applied during data processing. Peak profile analysis was performed with a local modification of a published procedure [6], and the intensity data were corrected for Lorentz and polarization effects. Corrections for absorption were also applied after isotropic refinement using an empirical method [7]. The coordinates of the Sn atom were obtained by using the automatic centrosymmetric direct routine in the SHELX-86 program package [8], while the remaining non-H atoms were located through a combination of structure factor calculations and difference-Fourier syntheses.

Refinement was by full-matrix least-squares methods based on F , with the weights taken as unit weights. Anisotropic thermal parameters were used for all atoms except for those of the benzene ring of the phthalimido group (C(22) through C(27)), which were found to have large thermal motion, indicating a possible positional disorder. This was also confirmed by the presence in their neighborhood of the two largest peaks occurring in the final difference map. Attempts to treat these atoms anisotropically did not result in any significant improvement of the R index and led to physically unrealistic thermal motion for some atoms. All four aromatic rings were refined as rigid bodies of D_{6h} symmetry. No attempt was made to locate or calculate the H-atoms. Omitted from the last cycles of refinement was one reflection (1 0 3) with bad agreement between F_o and F_c . Some correlation matrix elements greater than 0.5 were observed. The final residual indices for 3807 data are $R (= R_w) 0.0566$, $R_g 0.0570$.

Crystal Data. $\text{C}_{28}\text{H}_{21}\text{NO}_4\text{Sn}$, $M_r = 554.17$, rhombohedral, $R\bar{3}$, $a = 19.928(3) \text{ \AA}$, $\alpha = 116.47(2)^\circ$, $U = 3769(5) \text{ \AA}^3$, $D = 1.465 \text{ g cm}^{-3}$, $F(000) = 1668$, $\mu_{\text{Cu}} = 85.38 \text{ cm}^{-1}$ for $Z = 6$.

For triphenyltin *N*-phthaloylleucinate, intensities were collected on a prismatic crystal ($0.36 \times 0.44 \times 0.56$ mm) with a computer-controlled four-circle Philips PW 1100 diffractometer using $\text{Mo-K}\alpha$ radiation ($\lambda 0.71069 \text{ \AA}$). The crystal belongs to the orthorhombic system with systematic absences ($0\ k\ l$: k odd, $h\ 0\ l$: l odd and $h\ k\ 0$: h odd) characteristic of the centrosymmetric space group *Pbca*. No evidence of crystal deterioration was observed during the course of the data collection. A total of 4851 reflections were measured by the θ - 2θ scan technique for $3^\circ \leq \theta \leq 24^\circ$, 2163 of which were considered observed ($I > 3\sigma(I)$). The reflections were processed with the peak-profile analysis procedure and corrected for Lorentz, polarization, and absorption effects. The structure was solved by a combination of heavy-atom and direct-methods techniques and refined by full-matrix least-squares. Unit weights were used in the refinement; anisotropic thermal parameters were used for all non-H atoms, and the phenyl rings (including H-atoms) were refined as rigid bodies of D_{6h} symmetry. The final residual index $R (= R_w)$ is 0.0533 and R_g is 0.0633.

Crystal Data. $\text{C}_{32}\text{H}_{29}\text{NO}_4\text{Sn}$, $M_r = 610.28$, orthorhombic, *Pbca*, $a = 18.032(6)$, $b = 26.992(7)$, $c = 11.705(4) \text{ \AA}$, $U = 5697(3) \text{ \AA}^3$, $D = 1.414 \text{ g cm}^{-3}$, $F(000) = 2448$, $\mu_{\text{Mo}} = 9.3 \text{ cm}^{-1}$ for $Z = 8$.

Atomic scattering factors were taken from the usual tabulation [9] and the anomalous dispersion terms were included for all atoms. All calculations were performed on a GOULD 6040 computer using the SHELX-76 [10], SHELX-86 [8], PARST [11] and ORTEP [12] program packages. Table 1 lists the atomic coordinates of triphenyltin *N*-phthaloyllycinate.

TABLE 1 Fractional Atomic Coordinates for Triphenyltin *N*-phthaloylglycinate

Atom	x	y	z
Sn	0.01740(5)	0.17983(5)	-0.14903(5)
O(1)	0.1410(4)	0.2177(4)	-0.1458(4)
O(2)	0.1385(4)	0.1013(4)	-0.1552(4)
O(3)	0.4505(6)	0.4948(6)	-0.1139(6)
O(4)	0.2164(6)	0.2266(6)	-0.2862(5)
N	0.3292(5)	0.3420(5)	-0.0898(5)
C(1)	0.1364(4)	0.2584(4)	0.0232(4)
C(2)	0.0872(4)	0.2123(4)	0.0454(4)
C(3)	0.1682(4)	0.2741(4)	0.1641(4)
C(4)	0.2984(4)	0.3821(4)	0.2607(4)
C(5)	0.3476(4)	0.4282(4)	0.2385(4)
C(6)	0.2666(4)	0.3664(4)	0.1198(4)
C(7)	0.0617(4)	0.3127(3)	-0.1267(4)
C(8)	0.0632(4)	0.3091(3)	-0.1975(4)
C(9)	0.0860(4)	0.3913(3)	-0.1891(4)
C(10)	0.1072(4)	0.4771(3)	-0.1100(4)
C(11)	0.1057(4)	0.4807(3)	-0.0392(4)
C(12)	0.0829(4)	0.3985(3)	-0.0476(4)
C(13)	-0.1483(4)	-0.0108(4)	-0.3392(4)
C(14)	-0.1466(4)	-0.0469(4)	-0.4159(4)
C(15)	-0.2547(4)	-0.1704(4)	-0.5420(4)
C(16)	-0.3645(4)	-0.2577(4)	-0.5914(4)
C(17)	-0.3662(4)	-0.2216(4)	-0.5146(4)
C(18)	-0.2581(4)	-0.0981(4)	-0.3885(4)
C(19)	0.1799(6)	0.1826(6)	-0.1403(6)
C(20)	0.2870(6)	0.2441(6)	-0.1123(7)
C(21)	0.4060(7)	0.4605(7)	0.0237(7)
C(22)	0.4202(4)	0.5271(5)	0.0077(5)
C(23)	0.4873(4)	0.6480(5)	0.0864(5)
C(24)	0.4791(4)	0.6845(5)	0.0407(5)
C(25)	0.4039(4)	0.5999(5)	-0.0837(5)
C(26)	0.3368(4)	0.4789(5)	-0.1625(5)
C(27)	0.3450(4)	0.4425(5)	-0.1168(5)
C(28)	0.2818(7)	0.3190(7)	-0.1847(7)

TABLE 2 Fractional Atomic Coordinates for Triphenyltin *N*-phthaloylleucinate

Atom	x	y	z
Sn	0.18483(5)	0.15114(3)	0.21101(8)
O(1)	0.2614(5)	0.1726(3)	0.0929(8)
O(2)	0.2929(5)	0.0943(4)	0.0810(8)
O(3)	0.5036(8)	0.1359(4)	0.053(1)
O(4)	0.3362(6)	0.0683(4)	-0.202(1)
N	0.4050(7)	0.1130(5)	-0.069(1)
C(1)	0.2312(5)	0.1087(3)	0.3441(7)
C(2)	0.2381(5)	0.0576(3)	0.3301(7)
C(3)	0.2642(5)	0.0284(3)	0.4198(7)
C(4)	0.2834(5)	0.0503(3)	0.5236(7)
C(5)	0.2765(5)	0.1015(3)	0.5377(7)
C(6)	0.2504(5)	0.1306(3)	0.4479(7)
C(7)	0.1017(4)	0.1146(3)	0.1145(7)
C(8)	0.1184(4)	0.0800(3)	0.0298(7)
C(9)	0.0616(4)	0.0586(3)	-0.0342(7)
C(10)	-0.0120(4)	0.0718(3)	-0.0135(7)
C(11)	-0.0287(4)	0.1064(3)	0.0712(7)
C(12)	0.0282(4)	0.1278(3)	0.1352(7)
C(13)	0.1516(5)	0.2237(3)	0.2580(7)
C(14)	0.1162(5)	0.2327(3)	0.3619(7)
C(15)	0.0930(5)	0.2805(3)	0.3896(7)
C(16)	0.1053(5)	0.3194(3)	0.3133(7)
C(17)	0.1407(5)	0.3104(3)	0.2094(7)
C(18)	0.1639(5)	0.2626(3)	0.1818(7)
C(19)	0.3001(8)	0.1358(6)	0.050(1)
C(20)	0.354(1)	0.1534(7)	-0.041(2)
C(21)	0.350(1)	0.1946(7)	-0.099(1)
C(22)	0.4068(9)	0.2103(6)	-0.189(1)
C(23)	0.372(1)	0.216(2)	-0.296(2)
C(24)	0.440(2)	0.256(1)	-0.158(3)
C(25)	0.477(1)	0.1090(6)	-0.025(1)
C(26)	0.5119(5)	0.0671(3)	-0.0747(7)
C(27)	0.5822(5)	0.0463(3)	-0.0611(7)
C(28)	0.6013(5)	0.0036(3)	-0.1215(7)
C(29)	0.5502(5)	-0.0182(3)	-0.1955(7)
C(30)	0.4800(5)	0.0027(3)	-0.2091(7)
C(31)	0.4608(5)	0.0453(3)	-0.1487(7)
C(32)	0.3927(9)	0.0743(6)	-0.146(2)

cininate, and Table 2 those of the *N*-phthaloylleucinate.

RESULTS AND DISCUSSION

A notable feature of triphenyltin *N*-phthaloylglycinate (Figure 1) is its cyclic hexameric nature (Figure 2). The molecular structure consists of $[(C_6H_5)_3SnOC(O)CH_2NC(O)C_6H_4C(O)]_6$ units in which each of the six crystallographically-equivalent tin atoms resides in a trigonal bipyramidal environment where the *ipso*-carbon atoms of the three phenyl rings form the equatorial set and two oxygen atoms (one from the bridging carboxylate group) occupy the axial sites. The hexameric units are discrete, displaying no significant interactions with neighboring clusters, the closest contact being $O(3) \cdots C(16)$ ($1 + x, 1 + y, 1 + z$) 3.33(1) Å. The six tin atoms are arranged at the corners of a flattened chair-shaped ring with $Sn \cdots Sn$ distances of

5.584(2) Å. Figure 1 shows the coordination environment about the tin atom and Figure 2 is a perspective view of the hexamer. This structure again illustrates the marked tendency of triphenyltin derivatives to assume a five-coordinate, trigonal bipyramidal arrangement with the phenyl rings in the equatorial girdle. The tin-carbon bond distances are practically identical at 2.121(7), 2.121(8), and 2.127(5) Å and lie just in the middle of the range 2.075 to 2.181 Å of lengths observed in other structurally characterized trigonal bipyramidal triphenyltin derivatives [13]. The more significant angular distortions are in the equatorial plane, with the $C(1)-Sn-C(13)$ angle greatly enlarged to 130.9(3)° and the other two correspondingly compressed to 114.0(2) and 114.9(2)°; such angular widenings have also been noted in triphenyltin acetate and triphenyltin chloroacetate, whose largest C-Sn-C an-

FIGURE 1 The asymmetric unit of triphenyltin *N*-phthaloylglycinate. Selected bond distances and angles: Sn–C(1) 2.121(7), Sn–C(7) 2.121(8), Sn–C(13) 2.127(5), Sn–O(1) 2.191(10), Sn–O(2') 2.320(10) Å; C(1)–Sn–C(7) 114.0(2), C(1)–Sn–C(13) 130.9(3), C(1)–Sn–O(1) 93.8(2), C(1)–Sn–O(2') 84.0(3), C(7)–Sn–C(13) 114.9(2), C(7)–Sn–O(1) 86.8(4), C(7)–Sn–O(2') 90.8(4), C(13)–Sn–O(1) 94.0(2), C(13)–Sn–O(2') 90.1(3), O(1)–Sn–O(2') 175.9(2), Sn–O(1)–C(19) 129.1(9), Sn^{II}–O(2)–C(19) 141.5(9)° (symmetry transformation: $i = \bar{y}, \bar{z}, \bar{x}; ii = \bar{z}, \bar{x}, \bar{y}$).

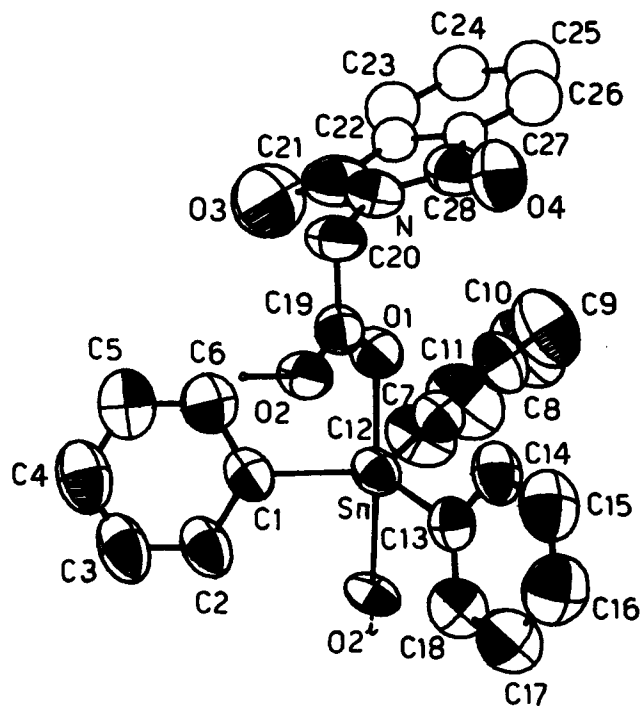
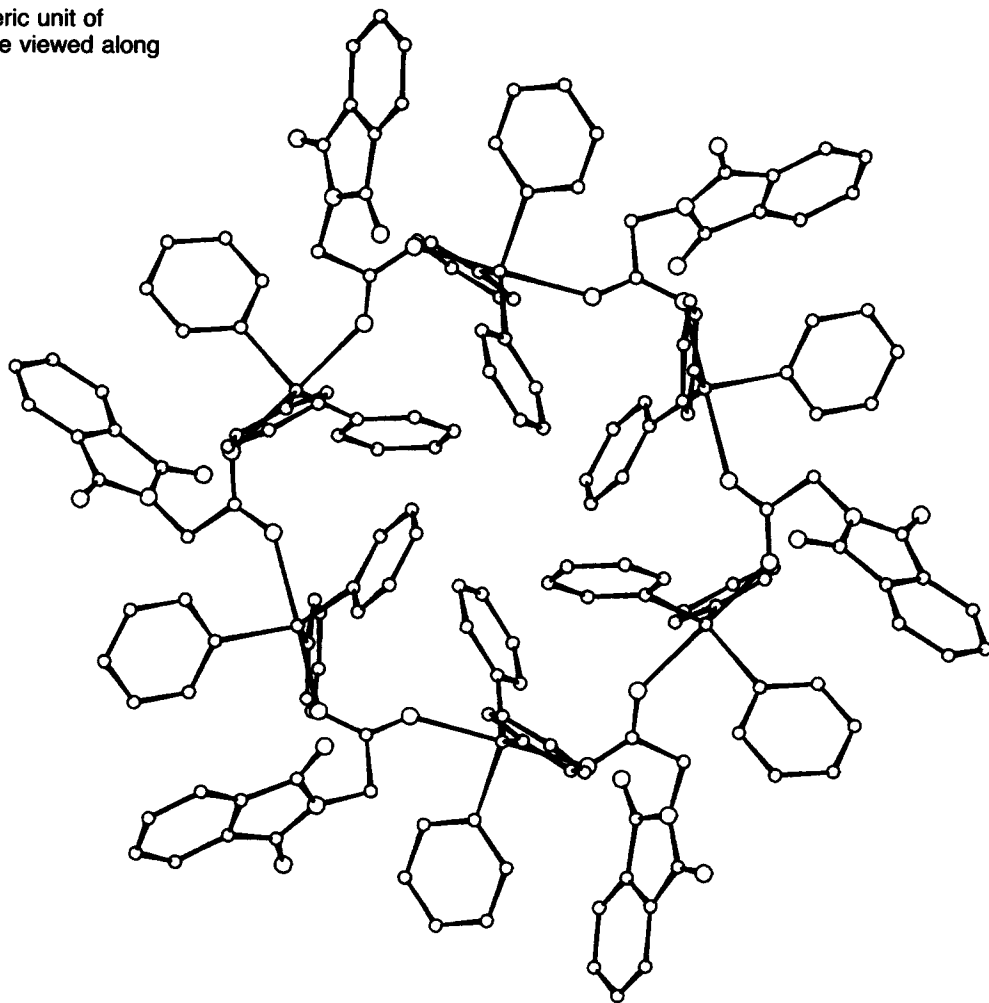


FIGURE 2 The cyclic hexameric unit of triphenyltin *N*-phthaloylglycinate viewed along the [1 1 1] direction.



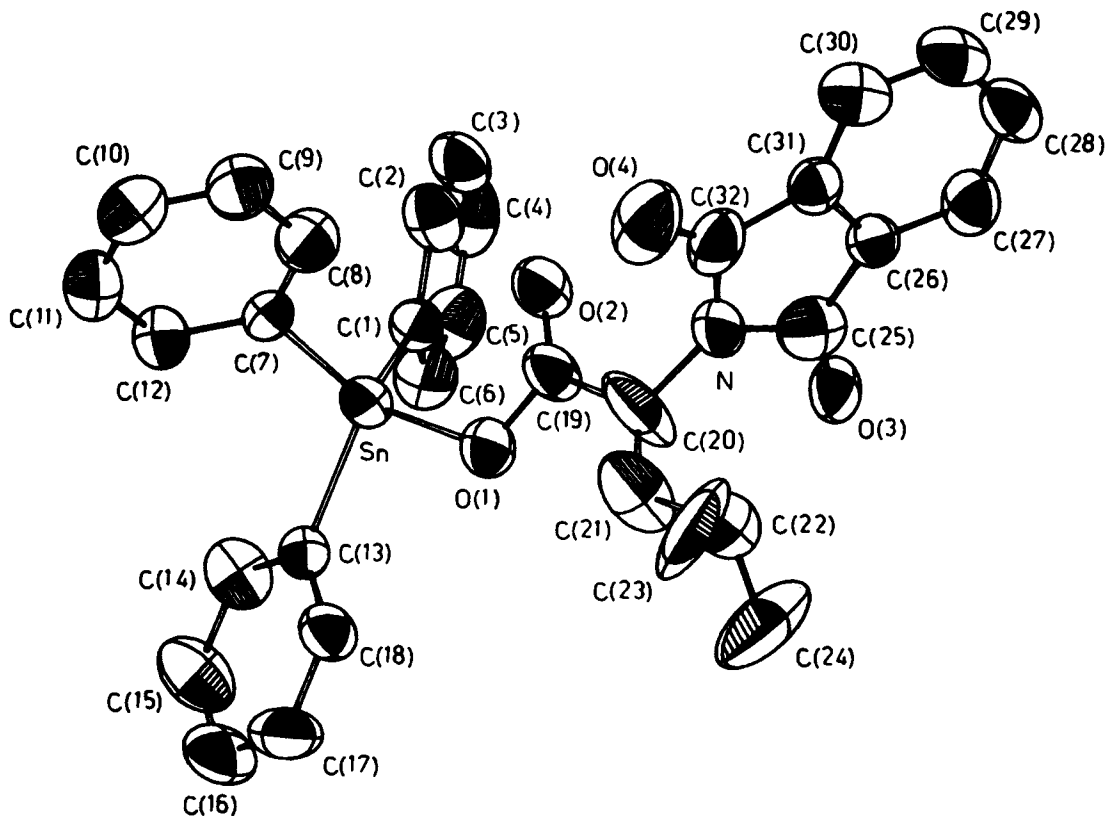


FIGURE 3 The asymmetric unit of triphenyltin *N*-phthaloylleucinate. Selected bond distances and angles: Sn–C(1) 2.107(8), Sn–C(7) 2.120(8), Sn–C(13) 2.121(7), Sn–O(1) 2.038(9) Å; C(1)–Sn–C(7) 115.0(3), C(1)–Sn–C(13) 115.0(3), C(1)–Sn–O(1) 112.8(4), C(7)–Sn–C(13) 111.6(3), C(7)–Sn–O(1) 104.5(4), C(13)–Sn–O(1) 96.0(3), Sn–O(1)–C(19) 113.7(9)°.

gles are 135.2 [14] and 135.1° [15], respectively. As has been previously discerned, polymeric triorganotin carboxylates characteristically possess a short tin–oxygen bond (2.14 to 2.22 Å) and a long intermolecular tin–oxygen bond (2.32 to 2.49 Å, except for tribenzyltin acetate) [3]. In triphenyltin *N*-phthaloylglycinate, the carboxylate ligand is seen to be unsymmetrically bound to tin, with Sn–O bond lengths of 2.191(10) and 2.320(10) Å. Another interesting feature which is common to these compounds is the significant difference in the two tin–oxygen–carbon angles, with that involving the more strongly bonded oxygen atom being near 120° and the other being considerably larger, in order to accommodate the bulky organic groups. Bond distances and angles in the phthalimido group are in good agreement with the corresponding values found in *N*-phthaloyl-glycinehydroxamic acid [16].

Inspection of the unit cell contents of triphenyltin *N*-phthaloylglycinate (Figure 2) reveals that the placement of a bulky group on the C(20) atom would lead to steric crowding with the phenyl ring (defined by C(7)–C(12)) of the adjacent molecule, and thereby possibly disrupt the hexameric conformation. This expectation is indeed borne out in triphenyltin *N*-phthaloylleucinate, the *i*-butyl-sub-

stituted triphenyltin *N*-phthaloylglycinate (Figure 3), whose carbonyl oxygen is 2.910(10) Å from the four-coordinate tin atom.

The structure of triphenyltin *N*-phthaloylleucinate can be compared with the structures of other triphenyltin arylcarboxylates with typically four-coordinate tin and intramolecular tin–oxygen contacts in the range 2.463–2.861 Å [17]. The intramolecular contact here is seen to exceed this range, and attests to the pronounced steric congestion in the molecule. In tri-*p*-anisyltin acetate the tin is also four-coordinate (Sn···O 2.747 Å), but here the monomeric nature is ascribed to the decreased Lewis acidity of tin arising from the electron-donating ability of the methoxy group [18]. The tetrahedral geometry of the *N*-phthaloylleucinate is also indicated by its tin-119m Mössbauer QS of 2.67 mm s⁻¹; for the five-coordinate *N*-phthaloylglycinate derivative, the QS value is significantly larger at 3.86 mm s⁻¹.

Triphenyltin *N*-phthaloylglycinate dissociates into monomers in chloroform solution, as evidenced by its one-bond coupling constant $J(^{119}\text{Sn}-^{13}\text{C}) = 643.3 \text{ Hz}$ and $\delta(^{119}\text{Sn}) = -90.5 \text{ ppm}$, which compare favorably with values found for other four-coordinate triphenyltin carboxylates [19].

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SUPPLEMENTARY MATERIAL AVAILABLE

Anisotropic thermal displacement factors, F_{obs} and F_{calc} tables, have been deposited with the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England, and are available on request.

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