observed here for the U–C(2) bond distances, suggesting relatively similar bonding. It can be noted that while the $(\eta^6\text{-}C_6H_6)U(AlCl_4)_3$ complex is readily destroyed by THF, it is thermally stable to 110° (80° in vacuo),[32] suggesting a reasonably attractive interaction. Due to the contracted nature of the metal f orbitals, however, this bonding would seem to involve a predominantly donor role for the central carbon atom (and the entire allyl anion), analogous to the situation for silver(1+) olefin complexes.[42]

The above structural results shed some light on the nature of the bonding in actinide π-allyl complexes. There would seem to be a definite interaction taking place between the uranium atom and the central carbon atom of the allyl ligand, although the relative strength of this bonding is certainly open to question. A more explicit understanding of the bonding in these systems, however, will only be obtained through further chemical and physical studies.

Acknowledgment. R.D.E. wishes to express his graditude for partial support of this research from the donors of the Petroleum Research Fund, administered by the American Chemical Society, a Pennwalt Corporation Grant of Research Corp., and The University of Utah Research Committee. We wish to thank Professor Roger E. Cramer of the University of Hawaii for providing structural data in advance of publication.

Registry No. $(\eta^5\text{-}C_5(CH_3)_5)U(\eta^3\text{-}2\text{-}C_4H_7)_3$, 84895-68-1; $(\eta^5\text{-}C_5(CH_3)_5)U(\eta^3\text{-}C_3H_5)_3$, 84895-69-2.

Supplementary Material Available: Table II, a listing of atomic thermal parameters, and a table of observed and calculated structure factor amplitudes (14 pages). Ordering information is given on any current masthead page.

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[42] (a) Quinn, H. W.; Tsai, J. H. *Adv. Inorg. Chem. Radiochem.* **1969**, *12*, 217. (b) Salomon, R. G.; Kochi, J. K. *J. Organomet. Chem.* **1972**, *43*, C7.

Crystal and Molecular Structure of *N*-(Trimethylstannyl)succinimide, an Associated, Helical Polymer

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N-(Trimethylstannyl)succinimide, $C_7H_{13}NO_2Sn$, crystallizes in the monoclinic space group $P2_1/c$ with a = 10.599 (8) Å, b = 14.054 (12) Å, c = 13.558 (10) Å, β = 110.29 (7)°, V = 1894.26 Å^3, Z = 8, and ρ_{calcd} = 1.754 g cm^{-3}. The structure was solved by Patterson and Fourier techniques from 3903 reflections measured at 138 ± 2 K on an Enraf-Nonius CAD/4 automatic diffractometer using monochromated Mo Kα radiation to a final R value of 0.045 for the 3588 reflections included in the least-squares sums (R_w = 0.0581). The title compound contains five-coordinated, trigonal-bipyramidal tin groups with axial –O→Sn–N units [175.1° (mean)] bound through planar succinimide rings which use only one of their carbonyl groups in coordination. This C=O bond is longer [d(C=O) = 1.227 Å (mean)] and its contiguous C—N distance is shorter [d(C—N) = 1.366 Å (mean)] than those for the noncoordinated carbonyl group [d(C=O) = 1.211 Å (mean); d(C—N) = 1.394 Å (mean)] as expected for contributions by an >N=C(—O$^-$)– form in the coordinated carbonyl. The nitrogen atom is planar [the sum of the three angles subtended at nitrogen is 359.8° (mean)], but the ∠-Sn—N—C(=O)– angles are larger than the ∠-C(=O)—N—C(=O)– [110.3° (mean)] angles. The trimethyltin unit is not perfectly planar, with the sum of the three ∠sC—Sn—C being 354.3° (mean) and the tin atom lying out of the plane determined by the three carbon atoms at 0.30 Å (mean) toward the nitrogen atom. The chains are nearly linear at the tin atom, but bent through the –O=C—N– system of the succinimide ring at ∠-Sn←O=C< [136.5 (3)°, 142.3 (4)°], –O=C—N– [123.0° (mean)], and –C—N—Sn– [121.6° (mean)] angles. The planes containing the ←O=C—N– units of the succinimide rings are turned at each tin atom by 30.18° at Sn(1) and 38.72° at Sn(2). The chains thus propagate along the b axis of the crystal to produce a flattened helix which completes its rotation with each fifth molecule. There are two crystallographically independent molecules per asymmetric unit, two asymmetric units per helix, and two helixes per centrosymmetric unit cell. The two helixes, being related by the center of symmetry, turn in opposite hands to produce a meso form of the solid.

Introduction

N-(Trimethylstannyl)succinimide has a number of features of potential structural interest. One is the structural consequence of the competition for the nitrogen atom lone pair among the d orbitals at tin and the double amide system of the succinimide ring. Populating the tin orbitals would lower its Lewis acid character, potentially leading to a monomeric structure containing four-coordinated tin, while delocalization of the lone pair through the five-atom O–C–N–C–O π system of the ring might allow coordination by both oxygen atoms to adjacent tins to give a rare, six-coordinated, R_3SnNO_2 system. Five-coordination in the ubiquitous, axially most-electronegative N–Sn←O configuration could lead to an n-meric ring association of coordinated molecules, the smallest of which would be a puckered tetramer, a situation of the type we have recently discovered in the hexameric triphenyltin(IV) diphenyl phosphate.[1] Carbonyl groups are usually weak donors toward tin, but the structures of the dimethyltin(IV) di-

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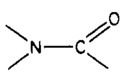
[1] Molloy, K. C.; Nasser, F. A. K.; Barnes, C. L.; van der Helm, D.; Zuckerman, J. J. *Inorg. Chem.* **1982**, *21*, 960.

chloride complexes with salicylaldehyde[2] and diphenylcyclopropenone[3] are known.[4] In both cases, however, charge dispersal is possible, as it is in the amide system



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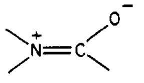

II

with canonical form II favorably disposed toward coordination through oxygen. A five-atom O–C–N–C–O π system would contain sp^2-hybridized oxygen atoms (two lone pairs plus the C–O bonding pair with the third lone pair in an atomic p orbital available for π interaction as in canonical form I). In this situation the oxygen lone pairs would lie in the plane of the succinimide ring and on coordination to tin would produce a geometry in which the Sn←O=C and ring planes were coincident, with an ∠Sn–O–C angle of 120°. Canonical form I could also lead to coordination by nitrogen as occurs in the associated trimethyltin (IV) azide through the α-nitrogen atom,[5,6] but amides generally coordinate to Lewis acids through the oxygen atom of the carbonyl group,[7] and this is thought to occur utilizing canonical form II.

In this paper we present some new spectroscopic data and report the crystal and molecular structure of *N*-(trimethylstannyl)succinimide.

Experimental Section

N-(Trimethylstannyl)succinimide was the fortuitous product of attempts to prepare halo-substituted cyclopentadienyltin derivatives. The compound was first prepared by the action of *N*-bromosuccinimide on *p*-tolyltrimethyltin.[8] Like this product, ours is a white crystalline solid, stable in air, but only slightly soluble in organic solvents. The solid, mp 146–147 °C (lit.[8] 148 °C), gives the tin-119m Mössbauer parameters: isomer shift (IS) = 1.31 ± 0.03; quadrupole splitting (QS) = 3.14 ± 0.06 mm s^{-1}. In the ^{1}H NMR the methylene bridge protons appear at δ 2.65 (s) (lit.[8] δ 2.70) and the methyltin protons at δ 0.60 (s) (lit.[8] δ 0.62) with $|^2J(^{119}\text{Sn–C–}^1\text{H})|$ = 59.7 Hz (lit.[8] 61 Hz) in CDCl$_3$ at 100 MHz. In the ^{13}C NMR spectrum the carbonyl carbon atoms appear at δ 185.93 (s), the methylene bridge atoms at δ 31.20 (s), and the methyltin atoms at δ −4.56 (s) vs. Me$_4$Si with $|^1J(^{119}\text{Sn–}^{13}\text{C})|$ = 394.96 Hz for the last.[9]

The infrared (1700–500 cm^{-1}) [Raman (700–80 cm^{-1})] absorptions are 1735 (m), 1640 (s, br), 1345 (s), 1280 (s), 1213 (s), 1186 (s), 1008 (m), 980 (w), 880 (w), 780 (s), 651 (m) (652 (m)), 540 (m) (549 (m)), 515 (w) (517 (s)), (441 (w)), 310 (vw), 260 (vw), (148 (m)), (115 (sh)), (113 (m)), (98 (m)), and (78 (m)) cm^{-1} in Nujol (solid) or CHCl$_3$. Bands at 785 and 550 cm^{-1} have been assigned to the methyl group and δ(Sn–C) modes, respectively.[8] The mass spectrum at 70 eV gave *m/e* 509 (2.0) $[2P - CH_3]^{+\cdot *}$, 248 (100) $[P - CH_3]^{+*}$, 218 (45.8) $[P - 3CH_3]^{+*}$, 190 (26.6) $[P - 3CH_3CO]^{+*}$, 177 (7.5) $[SnNC(O)CH_3]^{+*}$, 162 (68.4) $[SnNC(O)]^{+*}$, 150 (11.6) $[(CH_3)_2Sn]^{+\cdot *}$, 135 (77.1) $[CH_3Sn]^{+\cdot *}$, 99 (2.6) $[P - (CH_3)_3Sn]^+$, 70 (38.9) $[P - (CH_3)_3SnCO]^+$, 56 (26.3) $[NC(O)CH_2]^+$, 55 (30.7) $[NC(O)CH]^{+\cdot}$, and 42 (12.8) $[NC(O)]^+$ with the tin-bearing fragments asterisked. Anal. Calcd for C$_7$H$_{13}$NO$_2$Sn: C, 32.19; H, 4.97; Sn, 45.87. Found: C, 32.38; H, 4.79; Sn, 44.92.

Table I. Crystal Data for (CH$_3$)$_3$SnNC(O)CH$_2$CH$_2$C(O)[a]

formula	C$_7$H$_{13}$NO$_2$Sn
fw	261.9
cryst system	monoclinic
a, Å	10.599 (8)
b, Å	14.054 (12)
c, Å	13.558 (10)
β, deg	110.29 (7)
V, Å^3	1894.26
space group[b]	$P2_1/c$
Z	8
F(000)	1024
ρ_{calcd}, gm cm^{-3}	1.836
μ, cm^{-1}	24.49
cryst size, mm	0.15 × 0.25 × 0.3

[a] Estimated standard deviations in parentheses; the radiation used was Mo Kα_1 (λ = 0.70926 Å). [b] Based upon systematic absences: $0k0$, $k = 2n + 1$; $h0l$, $l = 2n + 1$.

Table II. Data Collection Parameters for (CH$_3$)$_3$SnNC(O)CH$_2$CH$_2$C(O)

diffractometer	Enraf-Nonius CAD/4
radiation	Mo K$\bar{\alpha}$ (λ = 0.710 69 Å)
temp, K	138 ± 2
scan technique	θ-2θ
2θ limit, deg	0 < 2θ < 53°
max scan time, s	40
scan angle, deg	0.9 + 0.2 tan θ
aperture width, mm	3.0 + 0.86 tan θ
aperture height, mm	6
aperture dist, mm	173
monitor reflection[c]	3
orientation monitors	150 reflections[a]
no. of unique data[b]	3903
no. of observed data	3588
corrections	Lorentz-polarization, anomalous dispersion

[a] New orientation matrix if angular change >0.12°; orientation matrix based upon 25 reflections. [b] $I > 2\sigma(I)$. [c] Three monitor reflections measured after each 7200s of X-ray exposure time. No deterioration of the crystal was observed.

Crystals of *N*-(trimethylstannyl)succinimide were obtained by sublimation as colorless cubes. Although the compound is air stable, the crystals became cloudy when exposed to air.

The unit cell dimensions and intensity data were obtained as in ref 10 and Tables I and II. No absorption correction was applied (the minimum and maximum transmittances are 0.48 and 0.69, respectively). Each structure factor was assigned an individual weight.[10] The final lattice parameters were determined from a least-squares refinement of the angular setting of 48 stray reflections.

The positions of the two tin atoms (two molecules per asymmetric unit) were determined from a three-dimensional Patterson map. All 22 non-hydrogen atoms in the asymmetric unit were obtained by successive difference Fourier syntheses. The least-square refinement of the structure was carried out in stages by using first isotropic and later anisotropic thermal parameters. All 20 hydrogen atoms were located from a difference Fourier map and were refined isotropically.

The final *R* factor ($R = \sum |k|F_o| - |F_c|| / \sum k|F_o|$) is 0.045 and R_w = 0.0581 for the 3588 reflections included in the least-squares calculations. The standard deviation of an observation of unit weight, or goodness of fit, σ, is 2.71. Refinement was discontinued when the maximal parameter shift in the final cycle of least squares refinement was less than 37% of the corresponding standard deviation for non-hydrogen atoms, while the average

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(2) Cunningham, D.; Douek, I.; Frazer, M. J.; McPartlin, M.; Matthews, J. D. *J. Organomet. Chem.* **1975**, *90*, C23.

(3) Ng, S.-W.; Kumar Das, V.-G.; Barnes, C. L.; Hossain, M. B.; van der Helm, D.; Zuckerman, J. J. *J. Am. Chem. Soc.* **1982**, *104*, 5359.

(4) Ng, S.-W.; Zuckerman, J. J. *J. Chem. Soc., Chem. Commun.* **1982**, 475.

(5) Allmann, R.; Hohlfeld, R.; Waskowska, A.; Lorberth, J. *J. Organomet. Chem.* **1980**, *192*, 353.

(6) Molloy, K. C.; Hossain, M. B.; van der Helm, D.; Zuckerman, J. J., unpublished results.

(7) Randall, E. W.; Silcox Yoder, C. M.; Zuckerman, J. J. *Inorg. Chem.* **1966**, *5*, 2240.

(8) Maire, J. C.; Prosperini, R.; van Rietschoten, J. *J. Organomet. Chem.* **1970**, *21*, P41.

(9) Petrosyan, V. S. *Prog. Nucl. Magn. Reson. Spectrosc.* **1977**, *11*, 115.

(10) Molloy, K. C.; Hossain, M. B.; van der Helm, D.; Zuckerman, J. J. *Inorg. Chem.* **1979**, *18*, 3507.

Table III. Final Positional Parameters for $(CH_3)_3Sn\overline{NC(O)CH_2CH_2C(O)}$ $(\times 10^4)^a$

atom	x	y	z
	Molecule A		
Sn(1)	4145.7 (3)	5583.8 (3)	2076.5 (3)
O(1)	3166 (5)	6984 (4)	−53 (4)
O(2)	1772 (5)	4046 (3)	485 (3)
N(1)	2624 (5)	5544 (3)	487 (4)
C(1)	4000 (8)	7043 (5)	2422 (6)
C(2)	3285 (8)	4615 (6)	2855 (5)
C(3)	5733 (6)	5048 (5)	1618 (5)
C(12)	1807 (5)	4794 (4)	31 (4)
C(13)	987 (6)	5015 (5)	−1108 (5)
C(14)	1358 (7)	6025 (5)	−1259 (5)
C(15)	2477 (6)	6272 (4)	−238 (5)
	Molecule B		
Sn(2)	1740.5 (3)	2239.8 (3)	56.7 (3)
O(3)	−161 (4)	209 (3)	−1344 (3)
O(4)	3956 (4)	710 (3)	1098 (3)
N(2)	1869 (5)	701 (3)	−146 (4)
C(4)	1834 (7)	2168 (5)	1657 (5)
C(5)	−160 (6)	2534 (5)	−1112 (5)
C(6)	3440 (7)	2642 (5)	−329 (6)
C(22)	3015 (6)	263 (4)	480 (4)
C(23)	2966 (7)	−790 (5)	314 (6)
C(24)	1598 (7)	−947 (5)	−558 (5)
C(25)	962 (6)	25 (4)	−753 (4)

[a] Estimated standard deviations in parentheses.

parameter shift for all atoms was 10%. An analysis of the function $[\sum w(F_o - F_c)^2/\sum w]^{1/2}$ showed no significant variation for various ranges of sin θ, $|F_o|$, and the indicies h,k,l.

Crystal data for *N*-(trimethylstannyl)succinimide are gathered in Table I and the data collection parameters in Table II. The final position parameters are in Table III and the intramolecular angles in Table IV. Table V lists the intermolecular angles and Table VI the short nonbonded contacts. Thermal parameters are tabulated in Table VII and hydrogen parameters in Table VIII. The hydrogen–carbon distances are found in Table IX and the least-squares planes in Table X.

Figure 1 gives the atomic numbering scheme for the two independent molecules in the asymmetric unit and lists the internuclear distances adjacent to each corresponding bond. Numbers 1–3 and 4–6 are used to label the methyltin carbons. Numbers 12–15 and 22–25 are used to label corresponding succinimide ring carbon atoms for ease of comparison. Figure 2 is a stereoview of the contents of the unit cell seen down the *a* axis. Figures 3 and 4 are views down the Sn(1)–N(1) and Sn(2)–N(2) axes, respectively.

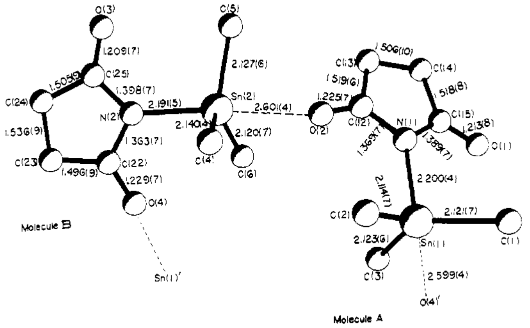

Figure 1. Internuclear distances in $(CH_3)_3Sn\overline{NC(O)CH_2CH_2C(O)}$ in angstroms. The two independent molecules in the asymmetric unit are shown. Estimated standard deviations are in parentheses. The primed atoms represent symmetrically equivalent positions of the corresponding atoms ($\bar{x}$, $^1/_2 + y$, $^1/_2 - z$).

Description and Discussion of the Structure

N-(Trimethylstannyl)succinimide crystallizes as an infinite, helical polymer in which roughly planar trimethyltin(IV) units are axially bridged by three-atom $\leftarrow$O=C—N– linkages of the succinimide ligand as shown in Figure 1. Use of only one oxygen atom per succinimide ring in coordination produces trigonal-bipyramidal geometry about tin. The internuclear angles in the –Sn$\leftarrow$O=C—N– system and the dihedral angles between the $\leftarrow$O=C—N– planes create helical chains propagating through the crystal around the twofold screw axis.

The associated nature of the solid helps rationalize the air stability of the material in this state and is corroborated by the slight solubility and the appearance in the mass spectrum of a polyisotopic fragment at *m/e* 509 which would correspond to a dimer minus one methyl group. In solution, however, an NMR $|^2J(^{119}Sn\text{–}C\text{–}^1H)|$ value of only 59.7 Hz $[|^1J(^{119}Sn\text{–}^{13}C)| = 394.96$ Hz] signifies a four-coordinated situation at tin,[9] and hence the solution must contain monomers.

The geometry at each tin atom is distorted from perfect trigonal bipyramidal, since one of the ∠C–Sn–C angles in each of the two crystallographically independent molecules is greater than 120° while the other two are less, and the tin atom lies out of the plane determined by the three methyl groups toward the nitrogen atom by 0.30 Å (mean). The sum of the three ∠-C–Sn–C- angles is 354.3° (mean). This together with the ∠-N–Sn–O- angle of 175.1° (mean) produces the distorted trigonal-bipyramidal geometry seen.

Table IV. Intramolecular Angles (deg) in $(CH_3)_3\overline{NC(O)CH_2CH_2C(O)}^a$

molecule A		molecule B	
C(1)-Sn(1)-C(2)	115.8 (3)	C(4)-Sn(2)-C(5)	117.6 (2)
C(1)-Sn(1)-C(3)	122.2 (3)	C(4)-Sn(2)-C(6)	121.2 (2)
C(2)-Sn(1)-C(3)	116.2 (3)	C(5)-Sn(2)-C(6)	115.5 (2)
N(1)-Sn(1)-C(1)	99.0 (2)	N(2)-Sn(2)-C(4)	95.6 (2)
N(1)-Sn(1)-C(2)	100.1 (2)	N(2)-Sn(2)-C(5)	100.5 (2)
N(1)-Sn(1)-C(3)	95.2 (2)	N(2)-Sn(2)-C(6)	97.9 (2)
Sn(1)-N(1)-C(12)	126.6 (3)	Sn(2)-N(2)-C(22)	116.6 (3)
Sn(1)-N(1)-C(15)	122.6 (4)	Sn(2)-N(2)-C(25)	133.2 (4)
C(12)-N(1)-C(15)	110.5 (4)	C(22)-N(2)-C(25)	110.1 (5)
O(1)-C(15)-N(1)	123.3 (5)	O(3)-C(25)-N(2)	124.2 (5)
O(1)-C(15)-C(14)	126.9 (5)	O(3)-C(25)-C(24)	126.0 (5)
O(2)-C(12)-N(1)	124.0 (4)	O(4)-C(22)-N(2)	122.1 (5)
O(2)-C(12)-C(13)	125.4 (5)	O(4)-C(22)-C(23)	125.9 (5)
N(1)-C(12)-C(13)	110.6 (5)	N(2)-C(22)-C(23)	112.0 (5)
C(12)-C(13)-C(14)	104.3 (5)	C(22)-C(23)-C(24)	103.4 (5)
C(13)-C(14)-C(15)	104.4 (5)	C(23)-C(24)-C(25)	104.6 (5)
C(14)-C(15)-N(1)	109.7 (5)	C(24)-C(25)-N(2)	109.7 (5)

[a] Estimated standard deviations in parentheses.

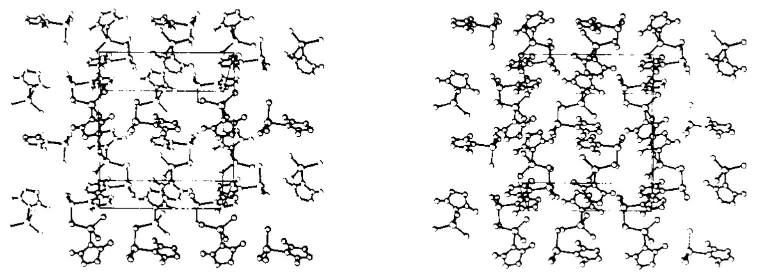

Figure 2. A stereoview of the packing.

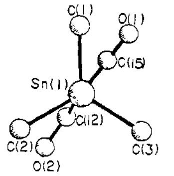

Figure 3. A view down the Sn(1)–N(1) axis. Atom N(1) is covered by Sn(1).

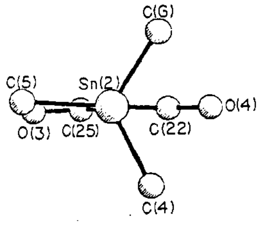

Figure 4. A view down the Sn(2)–N(2) axis. Atom N(2) is covered by Sn(2).

Table V. Intermolecular Angles (deg) in $(CH_3)_3\overline{SnNC(O)CH_2CH_2C}(O)^a$

O(2)-Sn(2)-N(2)	174.2 (1)	O(4)′[b]-Sn(1)-N(1)	175.9 (1)
O(2)-Sn(2)-C(4)	80.1 (2)	O(4)′-Sn(1)-C(1)	79.3 (2)
O(2)-Sn(2)-C(5)	84.9 (2)	O(4)′-Sn(1)-C(2)	83.9 (2)
O(2)-Sn(2)-C(6)	81.3 (2)	O(4)′-Sn(1)-C(3)	82.7 (2)
Sn(2)-O(2)-C(12)	136.5 (3)	Sn(1)-O(4)′-C(22)′	142.3 (4)

[a] Estimated standard deviations in parentheses. [b] The primed atoms represent symmetrically equivalent positions $(1 - x, 1/2 + y, 1/2 - z)$.

Table VI. Short Nonbonded Contact Distances (Å)[a]

Sn(1)-O(1)	3.347 (4)
-O(2)	3.449 (5)
Sn(2)-O(3)	3.627 (4)
-O(4)	3.135 (4)
O(1)-C(1)	3.161 (6)
O(2)-C(2)	3.156 (6)
-C(4)	3.070 (7)
-C(5)	3.212 (8)
-C(6)	3.096 (8)
O(3)-C(5)	3.283 (8)
O(4)-C(4)	3.319 (7)
-C(6)	3.268 (8)

[a] Estimated standard deviations in parentheses.

The observations of both ν_{sym} and ν_{asym} (Sn–C) modes in both the infrared (540, 515 cm^{-1}) and Raman (549, 517 cm^{-1}) spectra reflect the nonplanar nature of the $(CH_3)_3Sn$ system.

Although the near linearity of the axial -N-Sn←O connections requires linearity of the polymer in the vicinity of the tin atoms, the chains are bent through the ←O═C—N- bonds of the succinimide rings at angles ∠Sn←O═C- of 136.5 (3)°, -O═C—N- of 123.8° (mean), and -C—N—Sn of 121.6° (mean). In addition, the dihedral angle between the planes determined by the ←O═C—N- units bonded to Sn(1) is 30.18°, while the angle between the planes containing the ←O═C—N- units bonded to Sn(2) is 38.72°. The chains thus propagate through the crystal along the *b* axis in a flattened helical fashion. Each fifth molecule completes one turn of the helix. There are two crystallographically independent molecules per asymmetric unit, two asymmetric units per helix, and two helixes per centrosymmetric unit cell. These two helixes are related by the center of symmetry so that each one turns in the opposite hand (mirror images) to produce a meso form of the solid.

Virtually no truly relevant data exist that can be used to compare the distances in our -N—Sn←O- system where tin is covalently bonded to nitrogen and coordinatively bonded by oxygen,[11] especially considering the specially delocalized nature of the nitrogen lone pair in the succinimide and that the oxygen donor atom is part of an amide system. Most closely relevant to the structure of the title compound is that of (trimethylstannyl)nitromethylamine, $(CH_3)_3SnN(CH_3)NO_2$, which is also an associated polymer with distorted trigonal-bipyramide coordination at tin with *d*(Sn–N) = 2.34 and *d*(Sn←O) = 2.39 Å (mean).[12] Our mean tin distances are, on the other hand, shorter to the nitrogen [2.196 Å (mean)] and longer from the oxygen atom [2.600 Å (mean)], reflecting the weaker coordination of the succinimide vs. the nitrate group oxygen atoms to tin. The trimethyltin system is perfectly planar in the nitrate amine, but the ∠C–Sn–C angles sum to only 354.3° (mean) in our compound. Further, in the nitrate amine the ∠N–Sn←O angle is 172° (mean) vs. 175.1° (mean) in the title compound. Thus, although the trimethyltin system is more planar in the nitrate amine, the N–Sn←O system in our compound is more linear.

The trigonal $(CH_3)_3Sn(1)$ group is staggered with respect to the planar O═C—N(1)—C═O system to which it is attached as can be seen in Figure 3, but the corresponding trimethyltin(IV) group eclipses the succinimide ring along the Sn(2)–N(2) axis (see Figure 4). The disparity may be associated with the large difference in the Sn–N–C angles in the two succinimide rings, especially with the large value [133.2 (4)°] of the Sn(2)–N(2)–C(25) angle.

The geometry at nitrogen is nearly planar in each of the two independent molecules (the sum of the angles is 359.7° and 359.9° in molecules A and B, respectively), with the nitrogen atom out of the plane of the two carbonyl carbons and the tin atom by only 0.045 Å (mean). The ∠sSn–N–C angles involving the coordinated carbonyl group are more acute (by 4.0° in molecule A, but by 16.6° in molecule B) than the corresponding angles involving the noncoordinating carbonyl group, reflecting the contribution of can-

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[11] Zubieta, J. A.; Zuckerman, J. J. *Prog. Inorg. Chem.* **1978**, *24*, 251. Smith, P. J. *J. Organomet. Chem. Libr.* **1981**, *12*, 98.

[12] Domingos, A. M.; Sheldrick, G. M. *J. Organomet. Chem.* **1974**, *69*, 207.

Table XI. Comparison Dimensions (Average Values) of Selected Simple Succinimide Rings[a]

compd	$\angle$(C—N—C)	$\angle$(C—C(=O)—C)	$\angle$(C—C—C)	*d*(C=O)	*d*(N—C(=O))	*d*(C(=O)—C)	*d*(C—C)	ref
1	112.6	108.3	105.4	1.227	1.385	1.507	1.505	13
2[b]	115	107	106	1.22	1.39	1.48	1.55	14
3[c]	108	116	98	1.212	1.38	1.49	1.79	15
4	113.6	107.7	102.8	1.200	1.380	1.530	1.553	16
5	114.3	106.8	105.4	1.21	1.39	1.50	1.52	17
6	112.6	107.5	102.9	1.207	1.397	1.528	1.552	18
7	112.8	107.6	105.8	1.198	1.403	1.494	1.519	19
8	113	109	105	1.23	1.37	1.54	1.51	20
9[d]	111.6	107.4	106.6	1.202	1.423	1.476	1.511	21
10	112.1	108.0	105.9	1.200	1.397	1.488	1.500	22
11	114.6	107.8	103.5	1.21	1.36	1.52	1.55	23
12	113.8	107.9	104.6	1.214	1.378	1.517	1.536	24
13	114.6	107.3	103.9	1.208	1.370	1.527	1.534	25
title	110.3 (5)	110.5 (5)	104.2 (5)	1.219 (8)	1.380 (7)	1.510 (9)	1.521 (10)	*d*

[a] Distances (*d*) in Å; angles ($\angle$) in deg. [b] Solved from projection data. [c] From projection data; the data were calculated by us from the atomic coordinates listed in ref 15. [d] The data were calculated by us from the atomic coordinates in ref 21. [e] This work.

onical form II in the former case. Bond distance parameters also mirror the difference in the bonding modes of the two carbonyl groups. The coordinating carbonyl exhibits a longer *d*(C=O) [1.227 Å (mean) vs. 1.211 Å (mean)], while for the coordinating carbonyl the contiguous *d*(N–C) [1.366 Å (mean)] is shorter than in the noncoordinating carbonyl [1.394 Å (mean)] as expected from canonical form II. The hybridization at nitrogen is thus close to sp^2, but the $\angle$s-Sn-N-C(=O) angles [124.8° (mean)] are larger than the $\angle$-C(=O)—N—C(=O)- [110.3° (mean)] angles. Aside from the predominance of canonical form II for the coordinating carbonyl, and the steric requirements of the ring, the influence of the relative electronegativities of the tin atom and the carbonyl groups must contribute to the larger angle opening where the electropositive tin is involved and the angle closing where the nitrogen is connected to two electronegative carbonyl groups.

The situation in the title compound can be compared to that for several simple succinimide ring structures which have appeared in the literature. These include the parent compound (1),[13] *N*-chloro-(2),[14] *N*-(*p*-bromophenyl)-(3),[15] and α-(*p*-chlorophenyl)-α-methyl-α'-cyanosuccinimide (4),[16] *N*,*N'*-bisuccinimidyl (5),[17] *N*-phenyl-, tetramethylsuccinimide (6),[18] *N*,*N'*-dithiodisuccinimide (7),[19] 3-ethylsulfonyl, 4-methylsuccinimide (8),[20] *N*-bromosuccinimide (9),[21] *N*-*tert*-butyldithiosuccinimide (10),[22] α-(3-bromo-4-ethoxyphenyl)succinimide (11),[23] and 2-(1-methoxyethyl)-3-methylsuccinimide in the *RRS*-*SSR* (12)[24] and *SRS*-*RSR* (13)[25] forms (see Table XI). Complexes involving succinimides have been excluded. The most direct comparison, however, is with mercury(II) succinimide (14),[26] for which no relevant structural data are available. Judging by the circumference of the five-membered rings, our system [7.301 (10) Å] lies between the largest, the *N*-*p*-bromophenyl (3)[15] (7.53 Å) and the smallest, the *N*-*tert*-butyldithio (10)[22] (7.27 Å), near the parent succinimide (1)[13] (7.289 Å). The interior angles in the title succinimide ring sum to 539.7 (5)°, within experimental error of 540°, as do all but a few of the dozen examples in Table XI, the most deviant of which are the α-*p*-chlorophenyl)-α-methyl-α'-cyano (4)[16] (532.6°) and the *N*-phenyl-, tetramethyl (6)[18] (533.4°). The interior angle at nitrogen in 110.3 (5)° in the title compound; the smallest is in the *N*-*p*-bromophenyl (3)[15] (108°), while the largest is in the *N*-chloro (2)[14] (115°).

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Supplementary Material Available: Tables of thermal parameters (Table VII) and hydrogen parameters (Table VIII), of the hydrogen–carbon distances (Table IX), and of the least-squares planes (Table X) and a listing of structure factor amplitudes for the title compound (19 pages). Ordering information is given on any current masthead page.

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(13) Mason, R. *Acta Crystallogr.* **1961**, *14*, 720.

(14) Brown, R. N. *Acta Crystallogr.* **1961**, *14*, 711.

(15) Barassin, J.; Tsoucaris, G.; Lumbroso, H. *C. R. Hebd. Seances Acad. Sci.* **1961**, *213*, 2546.

(16) Baudour, J. L.; Messager, J. C. *Acta Crystallogr., Sect. B* **1971**, *B27*, 799.

(17) King, G. S. D. *J. Chem. Soc. B* **1966**, 1224.

(18) Wong, W.; Watkins, S. F. *J. Chem. Soc., Chem. Commun.* **1973**, 888.

(19) Mazhar-Ul-Haque; Behforouz, M. *J. Chem. Soc., Perkin Trans. 2* **1974**, 1459.

(20) Lotter, H.; Klein, G., Rüdiger, W.; Scheer, H. *Tetrahedron Lett.* **1977**, 2317.

(21) Jabay, O.; Pritzkow, H.; Jander, J. *Z. Naturforsch., B: Anorg. Chem., Org. Chem.* **1977**, *B32*, 1416.

(22) Mazhar-Ul-Haque, *Acta Crystallogr., Sect. B* **1980**, *B36*, 2082.

(23) Karapetyan, H. A.; Andrianov, V. G.; Struchkov, Yu. T. *Cryst. Struct. Commun.* **1980**, *9*, 417.

(24) Sheldrick, W. S. *Acta Crystallogr., Sect. B* **1981**, *B37*, 299.

(25) Sheldrick, W. S. *Acta Crystallogr., Sect. B* **1981**, *B37*, 300.

(26) Grdenić, D.; Kamenar, B.; Sikiriea, M.; Duplancić, T.; Govedic, S.; Hergold, A.; Matković, P. *Acta. Crystallogr., Sect. A* **1975**, *A31*, S132.