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19. ABSTRACT (Continue on reverse if necessary and identify by block number) Thermolysis of hexakis(2,6-diethylphenyl)cyclotristannane in xylenes at 200°C provides blue violet, crystalline 2,2,4,4,5,5-hexakis(2,6-diethylphenyl)pentastanna[1.1.1]propellane, 1, in 13% yield and hexakis(2,6-diethylphenyl)distannane, 2, after purification by column chromatography. Analytically pure 1 has been fully characterized by spectroscopic methods, including ¹¹⁹ Sn NMR, and a 2:1 mixed crystal of 1 and 2 has been crystallographically analyzed. Compound 2 has a Sn-Sn bond length of 3.05 Å which is the longest tin-tin bond length ever observed for a polystannane (previous longest value is 2.97 Å). The Sn-Sn bond lengths between the bridging and bridgehead tin atoms of 1 are in the range of 2.841 to 2.871 Å and the distance between the two bridgehead tin atoms is 3.367 Å. This latter distance falls well outside the range commonly observed for tin-tin bonds (including that of 2) and a structure with substantial singlet diradical character is proposed for 1. Solutions of 1 are intensely blue-violet in color with absorption maxima in pentane at 297 nm (14,500), 381 nm (2600), 495 nm (1000), and at 558 nm (1540) which extends beyond 700 nm (628).					
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2,2,4,4,5,5-Hexakis(2,6-diethylphenyl)pentastanna[1.1.1]propellane:
Characterization and Molecular Structure

by

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ABSTRACT

for

the Communication entitled,

2,4,5-HEXAKIS(2,6-DIETHYLPHENYL)PENTASTANNA[1.1.1]PROPELLANE:
CHARACTERIZATION AND MOLECULAR STRUCTURE

authored by

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Thermolysis of hexakis(2,6-diethylphenyl)cyclotristannane, 4, in xylenes at 200 °C provides blue violet, crystalline 2,4,5-hexakis(2,6-diethylphenyl)-pentastanna[1.1.1]propellane, 3, and hexakis(2,6-diethylphenyl)distannane, 5, after isolation and purification by column chromatography on silica gel (13% yield of 3). Analytically pure 3 has been fully characterized by spectroscopic methods, including ^{119}Sn NMR, and a 2:1 mixed crystal of 3 and 5 has been crystallographically analyzed. The tin-tin bond lengths between the bridging and bridgehead tin atoms of 3 are in the range of 2.841 (1) Å to 2.871 (1) Å and the distance between the two bridgehead tin atoms is 3.367 (1) Å. This latter distance falls well outside the range commonly observed for tin-tin bond lengths and a structure with substantial singlet diradical character is proposed for 3.

2,4,5-HEXAKIS(2,6-DIETHYLPHENYL)PENTASTANNA[1.1.1]PROPELLANE:
CHARACTERIZATION AND MOLECULAR STRUCTURE

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[1.1.1]propellane, 1 ($M = C$, $R = H$ in Scheme I), is an intriguing molecule that has received considerable experimental¹ and theoretical² attention regarding the extent to which bonding occurs between the bridgehead carbon atoms (i.e. 1a vs 1b). While this issue is still being debated, support is growing for a bond order of greater than zero.^{2c} Recently, *ab initio* calculations applied to the same question concerning bonding in pentasila[1.1.1]propellane, 2 ($M = Si$, $R = H$ in Scheme I) have shown that no minimum corresponding to a "classical" structure with a bond between the bridgehead atoms exists, and instead, the preferred structure for 2 appears to be one which possesses substantial singlet diradical character as in 2b.³ To date, no derivatives of 2 have been reported; however, herein, we report the isolation, characterization, and molecular structure of a pentastanna[1.1.1]propellane derivative, 3 ($M = Sn$, $R = 2,6$ -diethylphenyl in Scheme I), which provides the first insight concerning the nature of bonding for the tin analog of 1.

Insert Scheme I

Isolation of 3. The formation of 3 was accomplished by the thermolysis of hexakis(2,6-diethylphenyl)cyclotristannane 4⁴ in xylenes at 200 °C and isolation was effected through the use of flash chromatography on silica gel to provide 3 as an air-sensitive, dark blue violet, microcrystalline material (13% yield) and hexakis(2,6-diethylphenyl)distannane, 5, as a white crystalline co-product.⁵ Analytically pure 3 could be obtained through recrystallization from a 6:1 hexane/toluene mixture at -40 °C.

Characterization of 3.⁵ As shown in Figure 1, due to hindered rotation of the 2,6-diethylphenyl substituents, the ^1H NMR (300 MHz, toluene- d_8) spectrum of 3 at 20 $^\circ\text{C}$ exhibits two equally intense ABX_3 patterns produced by the diastereotopic character of the methylene hydrogen atoms. Above the coalescence temperature of 60 $^\circ\text{C}$, however, the ^1H NMR spectrum exhibits a single A_2X_3 pattern for the ethyl groups which is expected for all the aryl substituents being equivalent in 3 (Figure 1). Strong support for the [1.1.1]propellane structure of 3 is provided by ^{119}Sn NMR spectroscopy which is a powerful tool for elucidating tin atom connectivity in polystannanes.⁶ Thus, a ^{119}Sn (^1H) NMR (112 MHz, toluene- d_8 , δ relative to Me_4Sn) spectrum of 3 shows a resonance assigned to the equivalent bridging tin atoms (Sn_2 , Sn_4 , and Sn_5) at +356 ppm [$^1\text{J}(^{119}\text{Sn}-^{119}\text{Sn}) = 4159$ Hz (approximate relative intensity to parent peak, I = 8%), $^1\text{J}(^{119}\text{Sn}-^{117}\text{Sn}) = 3975$ Hz (I = 8%), and $^2\text{J}(^{119}\text{Sn}-^{117}\text{Sn}) = 262$ Hz (I = 8%)] and a resonance for Sn_1 and Sn_3 at -1751 ppm [$^1\text{J}(^{119}\text{Sn}-^{119}\text{Sn}) = 4159$ Hz (I = 14%), $^1\text{J}(^{119}\text{Sn}-^{117}\text{Sn}) = 3975$ Hz (I = 14%)].⁷ It is important to note that in the 1D ^{119}Sn NMR spectrum of 3, we do not observe a one-bond or two-bond coupling constant [$^n\text{J}(^{119}\text{Sn}_{1,3}-^{117}\text{Sn}_{3,1})$ ($n = 1$ or 2)] which are expected for coupling between Sn_1 and Sn_3 in the structures of 3a and 3b, respectively.⁸ Neither the proton-coupled ^{119}Sn NMR nor the IR (nujol) spectra show evidence for a directly bonded hydrogen atom at Sn_1 or Sn_3 (i.e. Sn-H)⁵, and at elevated temperatures up to 80 $^\circ\text{C}$, the ^{119}Sn NMR spectra remain the same indicating that 3 does not undergo dynamic exchange with other species at these temperatures.

Solutions of 3 are intensely blue-violet in color, showing absorption maxima in pentane at 297 nm (ϵ_{max} 14,500), 381 nm (ϵ_{max} 2600), 495 nm (ϵ_{max} 1000), and at 558 nm (ϵ_{max} 1540) which extends beyond 700 nm (ϵ 628). A mass spectrum of 3 (field desorption) reveals an isotope cluster pattern M^+ m/z (1385-1400) which is consistent with the molecular formula of $\text{C}_{60}\text{H}_{78}\text{Sn}_5$.⁵

Finally, solutions of 3 in the presence of air decolorize over a period of minutes to produce hexakis(2,6-diethylphenyl)cyclotristannoxane, 6⁴. In the solid state, however, 3 is much more robust, surviving for several hours upon exposure to air.

Insert Figure 1

Crystallographic Analysis of 3.⁵ Attempts to grow suitable crystals of 3 for X-ray analysis failed in a variety of solvents at -40 °C. However, on several occasions we observed by ¹H NMR that 3 had a tendency to co-crystallize with the hexaaryldistannane 5 from hexane at -40 °C when 5 was present as an impurity. Dark purple crystals comprised of a 2:1 ratio of 3 to 5 proved to be suitable for crystallographic analysis.^{6,10} As shown in Figure 2, the tin-tin bond lengths between the bridging and bridgehead tin atoms of 3 are longer than normal being in the range of 2.841 (1) Å to 2.871 (1) Å and this is to be expected for a ring-strained polystannane.¹⁰ Of particular interest is the distance of 3.367 (1) Å between Sn₁ and Sn₃. Prior to this investigation, the longest observed tin-tin bond length was on the order of 2.97 Å¹¹ and this has now been extended to 3.052 (1) Å for the hexaaryldistannane, 5.⁵ Thus, the distance of 3.367 Å falls well outside this new range which would appear to support substantial singlet diradical character for the structure of 3 (i.e. 3b), at least at 25 °C. The tin-carbon bond lengths are in the range of 2.16(1) - 2.22(1) Å and the bond angles of the pentastanna[1.1.1]propellane framework follow from the geometry of this structure and the observed tin-tin bond lengths mentioned above (Figure 2).

Insert Figure 2

In conclusion, the direct manner in which 3 can be prepared and isolated, together with its relatively high stability and the view provided by its molecular structure, will aid in future studies directed towards extending our

knowledge concerning bonding within the Group IVB elements. Both experimental and theoretical investigations of the electronic structure and chemical reactivity of pentastanna[1.1.1]propellane derivatives are currently in progress and will be reported in due course.

Acknowledgements. We thank Dr. Cynthia Day of Crystalytics Co. for the structural analysis of 3 and 5, and the Office of Naval Research for financial support. The high resolution mass spectrum of 3 was provided by Dr. Catherine Costello of the facility supported by the National Institutes of Health (Grant R R 00317; principal investigator, Professor K. Biemann), from the Division of Research Resources.

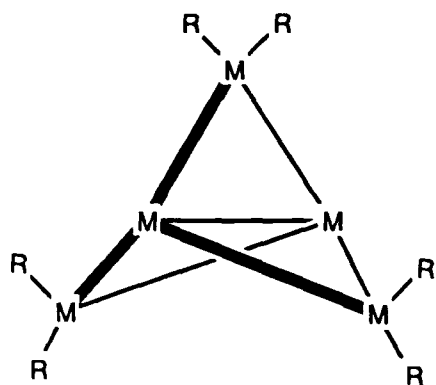
Supplementary Material Available. Detailed information concerning the isolation and the spectroscopic and crystallographic analysis of 3, including listings of atomic coordinates and temperature factors, bond lengths, bond angles, anisotropic temperature factors and an ORTEP representation of 3 and 5 (pages). Ordering information is given on any current masthead page

References and Footnotes

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4. Masamune, S.; Sita, L.R.; Williams, D.J. *J. Am. Chem. Soc.* 1983, 105, 630.
5. Detailed information is provided in the Supplementary Material.
6. Holt, M.S.; Wilson, W.L.; Nelson, J.H. *Chem. Rev.* 1989, 89, 11.
7. These unusual ^{119}Sn chemical shift values are in keeping with the observed downfield shift of the methylene carbon atoms and an upfield shift of the bridgehead carbon atoms in the ^{13}C NMR spectrum of 1 (see reference 1).
8. It is possible that the value of $\eta_{\text{J}}(^{119}\text{Sn}_{1,3} - ^{117}\text{Sn}_{1,3})$ is smaller than the line width of the parent peak ($w_{1/2} = 26 \text{ Hz}$).
9. The molecular structure of 5 is provided as part of the crystallographic analysis of 3 and while a detailed discussion of this compound will be made in a separate account, a crystal packing diagram shows no unusual intermolecular interactions between 3 and 5 (see Supplementary Material).
10. Cf. the tin-tin bond length range of 2.818 (1) - 2.931 (1) Å in a bicyclo[2.2.0]hexastannane derivative; Sita, L.R.; Bickstaff, R.D. *J. Am. Chem. Soc.* 1989, 111, 0000.
11. The Sn-Sn bond lengths in Sn_4^{-2} are 2.934 (3) - 2.972 (7) Å [Critchlow, S.C.; Corbett, J.D. *J. Chem. Soc., Chem. Commun.* 1981, 236]. Also see: a) Adams, S.; Drager, M. *Angew. Chem. Int. Ed. Engl.* 1987, 26, 1244. b) Puff, H.;

Breuer, B.; Gehrke-Brinkmann, G.; Kind, P.; Reuter, H.; Schuh, W.; Wald, W.;
Weidenbruck, G. J. *Organometal. Chem.* 1989, 365, 265.

Scheme I



1a

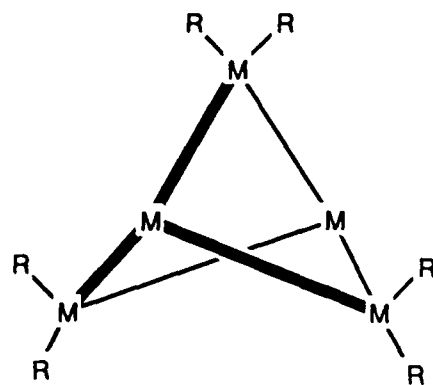
$M = C$ and $R = H$

2a

$M = Si$ and $R = H$

3a

$M = Sn$ and $R = 2,6\text{-diethylphenyl}$



1b

2b

3b

FIGURE 1

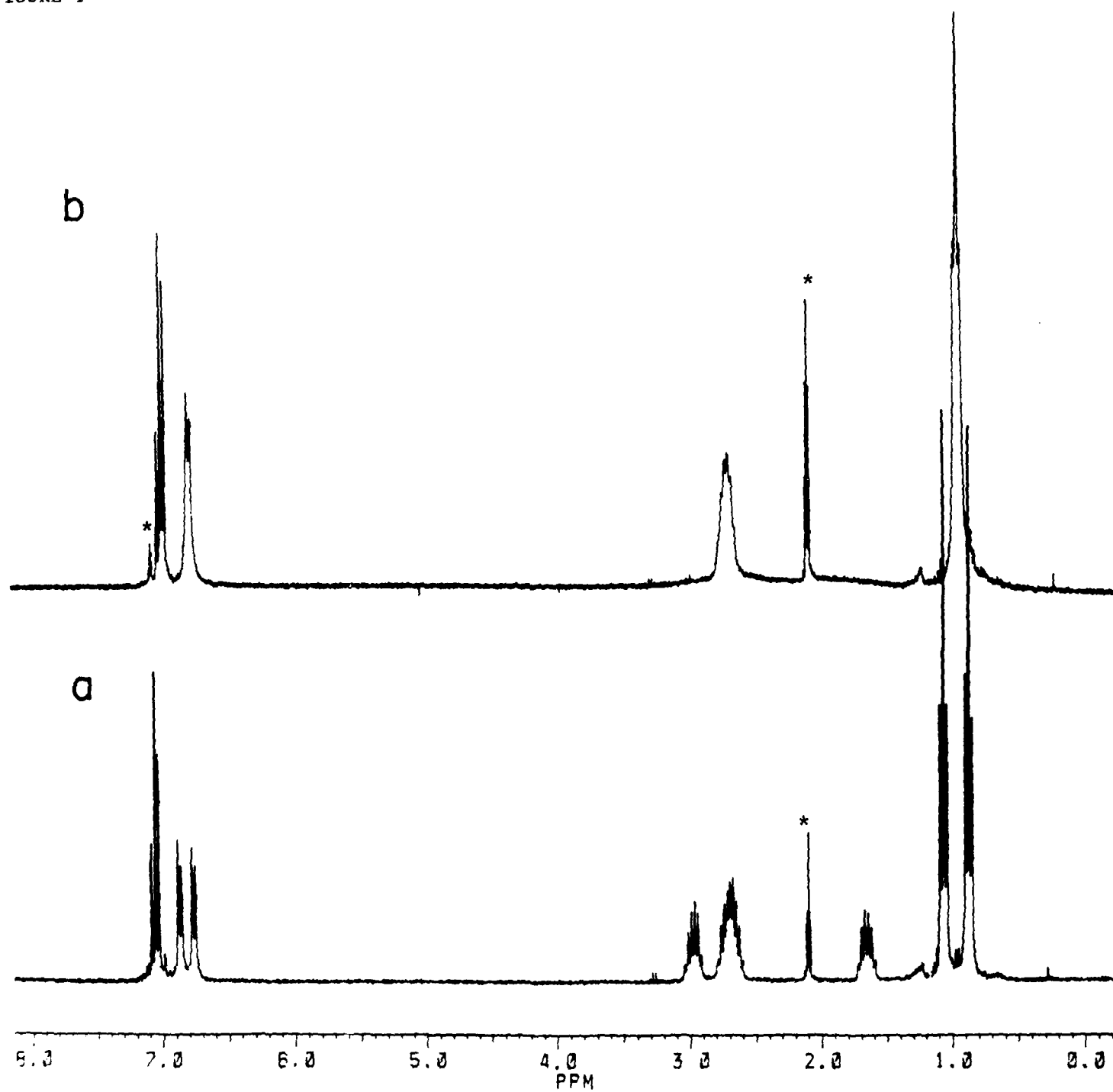
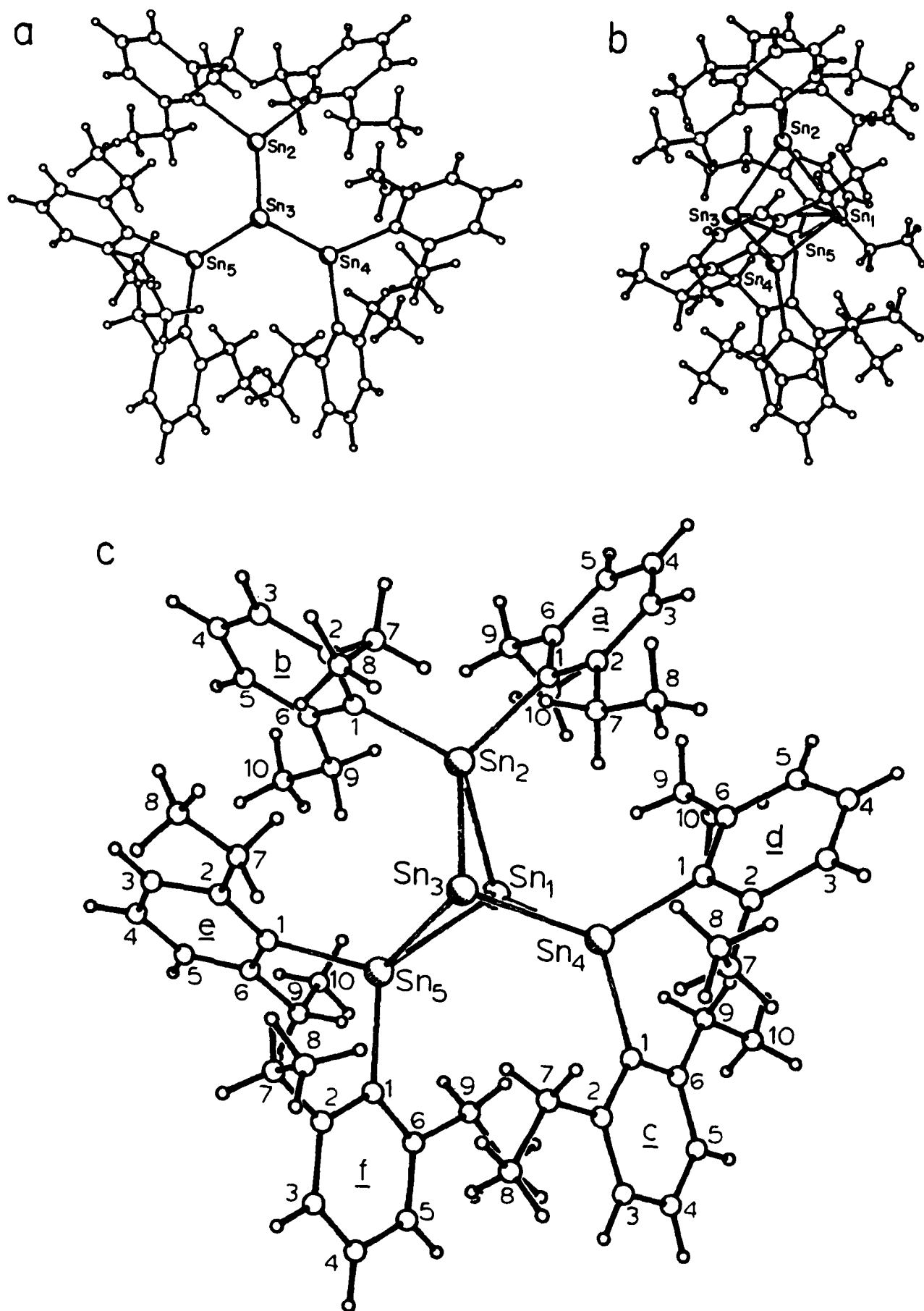


Figure 1. ^1H NMR (300 MHz, toluene- d_8) spectra of 3 taken at 20 °C (spectrum a) and at 70 °C (spectrum b). Solvent peaks are marked with an asterisk.

Figure 2. Crystal structure of 3. a) Perspective of 3 viewed perpendicular to the triangular plane specified by Sn₂, Sn₄, and Sn₅. b) Perspective of 3 viewed parallel to plane specified by Sn₁, Sn₂, and Sn₃. c) Perspective of 3 viewed nearly perpendicular to plane specified by Sn₂, Sn₄ and Sn₅. Tin atoms are represented by large shaded spheres while carbon and hydrogen atoms are represented by medium and small open spheres, respectively. The aryl ligand carbons are labeled with a number (from 1 to 10) with a literal subscript (a-f) identifying their particular ligand. Bond lengths (Å): Sn₁-Sn₂ 2.871(1), Sn₁-Sn₄ 2.852(1), Sn₁-Sn₅ 2.849(1), Sn₃-Sn₂ 2.841(1), Sn₃-Sn₄ 2.871(1), Sn₃-Sn₅ 2.864(1), Sn₁-Sn₃ 3.367(1), Sn-C 2.16(1) - 2.22(1). Bond Angles (°); Sn₂Sn₁Sn₃ 53.5(1), Sn₃Sn₁Sn₄ 54.2(1), Sn₃Sn₁Sn₅ 54.1(1), Sn₁Sn₃Sn₂ 54.3(1), Sn₁Sn₃Sn₄ 53.7(1), Sn₁Sn₃Sn₅ 53.7(1), Sn₁Sn₂Sn₃ 72.2(1), Sn₁Sn₄Sn₃ 72.2(1), Sn₁Sn₅Sn₃ 72.2(1), Sn₂Sn₁Sn₄ 88.4(1), Sn₂Sn₁Sn₅ 88.2(1), Sn₄Sn₁Sn₅ 89.9(1), Sn₂Sn₃Sn₄ 88.6(1), Sn₂Sn₃Sn₅ 88.5(1), Sn₄Sn₃Sn₅ 89.3(1)

FIGURE 2

-10-



Supplementary Material

for

the Communication entitled,

2,4,5-HEXAKIS(2,6-DIETHYLPHENYL)PENTASTANNA[1.1.1]PROPELLANE:
CHARACTERIZATION AND MOLECULAR STRUCTURE

authored by

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Detailed information on the isolation and characterization of 2,4,5-hexakis-(2,6-diethylphenyl)pentastanna[1.1.1]propellane, 3, and the crystallographic analysis of a 2:1 mixed crystal of 3 and hexakis(2,6-diethylphenyl)-distannane, 5.

Isolation of 3: In a typical experiment, a mixture of 5.0 g (4.4 mmol) of 4 and 3.5 ml of dry xylenes were placed in a sealed tube under nitrogen and heated to 200 °C for 10 min whereupon a dark reddish brown solution was obtained. At this time, the solution was cooled rapidly to room temperature and then immersed into liquid nitrogen. After warming to room temperature, removal of the xylenes under reduced pressure (10^{-6} torr) and washing the residue with pentane (3 x 5 ml) provided recovered 4 as a bright yellow solid and a dark purple residue was obtained from the pentane extracts upon removal of the solvent *in vacuo*.

Repetition of this process twice more by utilizing the amount of recovered 4 each time, resulted in a combined yield of 2.5 g of crude material and then this material was divided into five 0.5 g portions. In a glove box, compound 3 was isolated from each portion through flash chromatography on silica gel (400 mesh) using a 6:1 mixture of hexane:toluene as the elutant. Collection of the dark purple fractions and removal of the solvents provided approximately 100 mg of 3 from each portion for a combined yield of 470 mg (13% yield based on 4).

Spectroscopic Data for 3: ^1H NMR (300 MHz, 20 °C, toluene- d_8) δ (ppm from reference solvent peak at 2.09 ppm) 0.87 (3H, t, $J_{ax} = 7.4$ Hz), 1.06 (3H, t, $J_{ax} = 7.4$ Hz), 1.65 (1H, dq, $J_{ax} = 7.4$ Hz, $J_{ab} = 15.3$ Hz), 2.69 (2H, m), 2.97 (1H, dq, $J_{ax} = 7.4$ Hz, $J_{ab} = 14.5$ Hz), 6.76 (1H, d, $J = 7.6$ Hz), 6.86 (1H, d, $J = 7.5$ Hz), 7.05 (1H, t, $J = 7.5$ Hz). ^{13}C (^1H) NMR (75 MHz, toluene- d_8) δ 15.59, 15.65, 33.61, 35.71, 125.15, 125.99, 128.5, 148.58, 155.51. ^{119}Sn (^1H) NMR (see text); IR (nujol) 3188 (w), 3050(s), 2726 (ws), 1929 (ws), 1857 (ws), 1790 (ws), 1566 (s), 1541 (ws), 1321 (s), 1274 (ws), 1242 (ws), 1229 (ws), 1069 (s), 1043 (s), 1012 (s), 971 (s), 779 (s), 760 (s). UV (pentane) (see text).

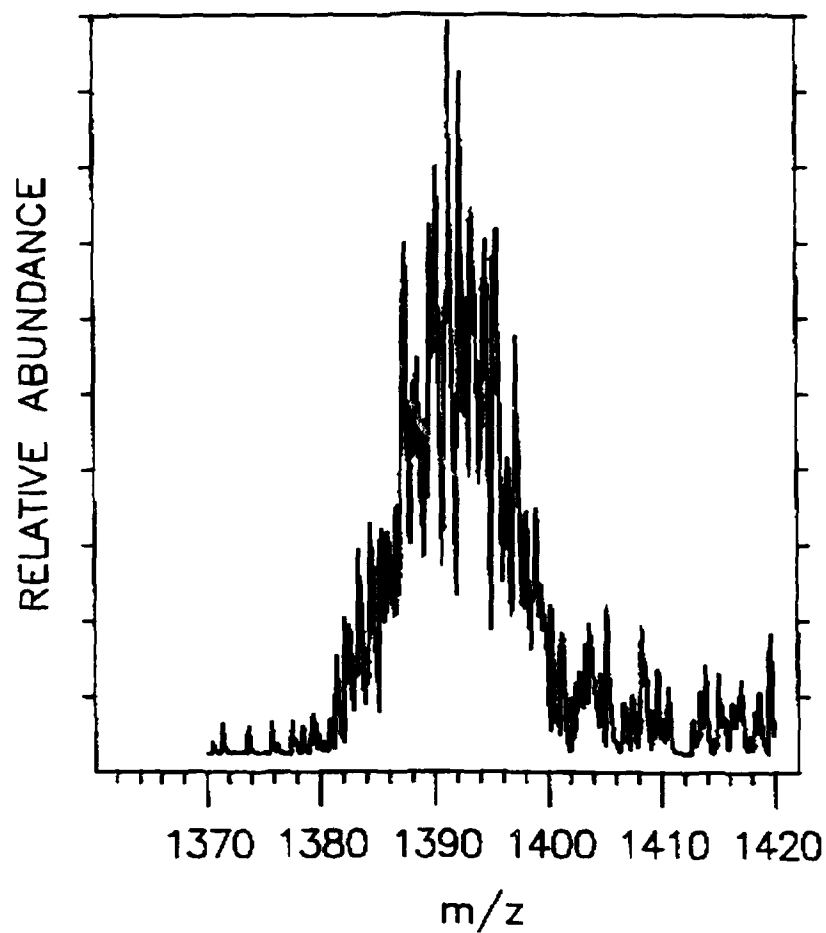


Figure 3. Molecular ion cluster pattern for 3 from the field desorption mass spectrum.

Crystallographic Analysis of 2:1 Mixed Crystal of 3 and 5:

Single crystals of $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 1/2 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$ are, at $20 \pm 1^\circ\text{C}$, triclinic, space group $\text{P}\bar{1}-\text{C}_1^1$ (No. 2) with $a = 12.727(4)\text{\AA}$, $b = 17.272(5)\text{\AA}$, $c = 19.760(8)\text{\AA}$, $\alpha = 97.43(3)^\circ$, $\beta = 96.71(3)^\circ$, $\gamma = 101.51(3)^\circ$, $V = 4175(3)\text{\AA}^3$ and $Z = 2$ ($d_{\text{calcd}} = 1.521\text{gcm}^{-3}$; $\mu_a(\text{MoK}\alpha) = 1.82\text{mm}^{-1}$). A total of 11462 independent reflections having $2\theta_{\text{MoK}\alpha} < 45.8^\circ$ (the equivalent of 0.6 limiting $\text{CuK}\alpha$ spheres) were collected on a computer-controlled Nicolet autodiffractometer using ω scans and graphite-monochromated $\text{MoK}\alpha$ radiation. The structure was solved using Direct Methods techniques with the Nicolet SHELXTL software package as modified at Crystalytics Company. The resulting structural parameters have been refined to convergence $\{R_1 \text{ (unweighted, based on } F) = 0.051 \text{ for } 7008 \text{ independent reflections having } 2\theta_{\text{MoK}\alpha} < 45.8^\circ \text{ and } I > 3\sigma(I) \}$ using counter-weighted cascade block-diagonal least-squares techniques and a structural model which incorporated anisotropic thermal parameters for all nonhydrogen atoms and isotropic thermal parameters for all included hydrogen atoms. Due to program limitations, only 13 of the 18 terminal methyl groups could be included in the structural model as idealized rigid rotors; the five which were omitted belonged to the dimer. The remaining hydrogen atoms were fixed at idealized sp^2 - or sp^3 -hybridized positions with a C-H bond length of $0.96\text{\AA}$.

Table I. Atomic Coordinates for Nonhydrogen Atoms in Crystalline
 $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]^a$

Atom Type ^b	Fractional Coordinates			Equivalent Isotropic Thermal Parameter, B, Å ² x 10 ^c
[Sn ₅ (C ₁₀ H ₁₃) ₆]				
Sn ₁	2778(1)	2821(1)	2088(1)	35(1)
Sn ₂	2082(1)	3570(1)	952(1)	35(1)
Sn ₃	4381(1)	3854(1)	1121(1)	38(1)
Sn ₄	4223(1)	2206(1)	1273(1)	38(1)
Sn ₅	4404(1)	4239(1)	2577(1)	40(1)
C _{1a}	1080(9)	2763(7)	52(6)	41(4)
C _{2a}	1504(11)	2546(8)	-557(7)	52(5)
C _{3a}	834(13)	2029(9)	-1109(7)	66(6)
C _{4a}	-243(13)	1771(9)	-1064(8)	72(6)
C _{5a}	-662(12)	1980(8)	-478(8)	61(5)
C _{6a}	17(10)	2495(7)	88(7)	47(4)
C _{7a}	2720(11)	2819(9)	-621(6)	65(5)
C _{8a}	3136(15)	2508(13)	-1205(9)	109(9)
C _{9a}	-545(10)	2638(7)	713(6)	51(4)
C _{10a}	-621(12)	1968(8)	1125(8)	70(6)
C _{1b}	1413(8)	4628(7)	1079(6)	39(4)
C _{2b}	1426(9)	5091(7)	550(7)	44(4)
C _{3b}	997(12)	5783(8)	616(8)	63(5)
C _{4b}	568(12)	6024(9)	1207(9)	72(6)
C _{5b}	585(10)	5579(8)	1710(7)	55(5)
C _{6b}	977(8)	4879(7)	1697(6)	38(3)
C _{7b}	1916(11)	4879(8)	-82(7)	59(5)
C _{8b}	2866(23)	5415(13)	-165(13)	208(16)

Table I. (continued)

Atom Type ^b	Fractional Coordinates			Equivalent Isotropic Thermal Parameter, $B, \text{\AA}^2 \times 10^3$ ^c
$10^4 x$	$10^4 y$	$10^4 z$		
C _{9b}	977(9)	4414(7)	2279(6)	45(4)
C _{10b}	593(12)	4739(9)	2928(7)	66(5)
C _{1c}	5488(10)	1797(8)	1881(6)	46(4)
C _{2c}	6564(11)	2287(9)	2047(6)	58(5)
C _{3c}	7317(11)	1984(10)	2474(7)	73(6)
C _{4c}	6996(12)	1241(10)	2690(7)	79(7)
C _{5c}	6003(12)	797(10)	2507(8)	74(6)
C _{6c}	5231(12)	1066(9)	2105(6)	53(5)
C _{7c}	6870(10)	3055(9)	1811(8)	70(6)
C _{8c}	7986(12)	3553(10)	2081(11)	105(8)
C _{9c}	4108(12)	573(8)	1951(7)	64(6)
C _{10c}	3883(17)	-204(11)	2200(12)	122(10)
C _{1d}	3657(10)	1291(7)	343(6)	44(4)
C _{2d}	4472(10)	1129(8)	-47(7)	56(5)
C _{3d}	4126(13)	507(8)	-640(7)	67(6)
C _{4d}	3080(13)	108(9)	-834(8)	73(6)
C _{5d}	2315(12)	302(8)	-441(7)	62(5)
C _{6d}	2579(9)	886(7)	156(6)	44(4)
C _{7d}	5650(10)	1557(10)	120(7)	69(6)
C _{8d}	6054(19)	1992(17)	-340(11)	198(15)
C _{9d}	1673(10)	998(7)	561(7)	56(5)
C _{10d}	1273(14)	369(10)	935(10)	90(8)
C _{1e}	3827(10)	5274(7)	3067(6)	46(4)
C _{2e}	3691(9)	5910(7)	2732(7)	46(4)

Table 1. (continued)

Atom Type ^b	Fractional Coordinates			Equivalent Isotropic Thermal Parameter, $B, \text{\AA}^2 \times 10^3$ ^c
	10^4x	10^4y	10^4z	
C _{3e}	3355(12)	6546(8)	3117(8)	68(6)
C _{4e}	3185(14)	6530(9)	3773(8)	79(6)
C _{5e}	3290(12)	5899(8)	4082(8)	69(6)
C _{6e}	3627(10)	5252(8)	3743(7)	51(4)
C _{7e}	3904(12)	5950(7)	1987(7)	59(5)
C _{8e}	3906(18)	6680(11)	1707(11)	117(10)
C _{9e}	3703(13)	4560(10)	4114(7)	71(6)
C _{10e}	2699(21)	4128(15)	4288(17)	209(19)
C _{1f}	5951(9)	4302(7)	3203(6)	45(4)
C _{2f}	6754(10)	4986(8)	3224(8)	66(5)
C _{3f}	7771(12)	5071(10)	3637(8)	86(6)
C _{4f}	7979(12)	4506(10)	4012(8)	82(6)
C _{5f}	7193(11)	3819(10)	3986(7)	73(6)
C _{6f}	6183(11)	3728(9)	3604(6)	56(5)
C _{7f}	6640(12)	5632(8)	2781(8)	71(6)
C _{8f}	7137(16)	5568(11)	2157(10)	107(9)
C _{9f}	5318(12)	2953(9)	3573(7)	63(5)
C _{10f}	5638(15)	2364(12)	4002(10)	105(9)
0.5 [Sn ₂ (C ₁₀ H ₁₃) ₆]				
Sn ₆	-209(1)	-881(1)	4683(1)	33(1)
C _{1g}	-1682(8)	-1191(6)	3856(5)	36(3)
C _{2g}	-2569(10)	-1827(8)	3878(7)	52(4)
C _{3g}	-3472(11)	-1978(9)	3336(8)	64(5)
C _{4g}	-3489(12)	-1518(9)	2821(8)	76(6)
C _{5g}	-2623(11)	-916(8)	2822(7)	64(5)
C _{6g}	-1714(9)	-750(7)	3327(6)	42(4)

Table I. (continued)

Atom Type ^b	Fractional Coordinates			Equivalent Isotropic Thermal Parameter, $B, \text{\AA}^2 \times 10^3$ ^c
	10^4x	10^4y	10^4z	
C _{7g}	-2646(11)	-2425(9)	4361(7)	72(6)
C _{8g}	-3725(14)	-2687(15)	4565(13)	166(13)
C _{9g}	-780(10)	-85(7)	3248(6)	52(4)
C _{10g}	-582(14)	45(10)	2516(7)	78(6)
C _{1h}	1223(9)	-1135(7)	4193(5)	38(4)
C _{2h}	2235(9)	-887(7)	4586(6)	43(4)
C _{3h}	3157(11)	-962(9)	4299(8)	71(6)
C _{4h}	3078(14)	-1343(9)	3622(9)	77(7)
C _{5h}	2043(14)	-1627(9)	3248(7)	67(6)
C _{6h}	1122(11)	-1542(7)	3523(7)	50(4)
C _{7h}	2439(10)	-501(8)	5347(6)	54(4)
C _{8h}	3563(11)	-91(11)	5695(8)	85(7)
C _{9h}	65(12)	-1968(9)	3073(7)	78(6)
C _{10h}	-152(19)	-2841(11)	2873(14)	145(11)
C _{1i}	-391(8)	-1637(6)	5502(5)	34(3)
C _{2i}	190(9)	-2240(7)	5575(6)	45(4)
C _{3i}	121(11)	-2614(8)	6184(8)	57(5)
C _{4i}	-487(15)	-2418(9)	6671(8)	74(6)
C _{5i}	-1120(12)	-1876(8)	6550(7)	65(5)
C _{6i}	-1090(10)	-1484(7)	5983(6)	44(4)
C _{7i}	782(11)	-2613(8)	5060(7)	62(5)
C _{8i}	235(15)	-3440(10)	4740(9)	91(7)
C _{9i}	-1874(11)	-912(7)	5880(7)	57(5)
C _{10i}	-2659(18)	-831(13)	6360(12)	132(11)

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.

^b Atoms are labeled in agreement with Figures 1 and 2.

^c This is one-third of the trace of the orthogonalized B_{ij} tensor.

Table II. Anisotropic Thermal Parameters for Nonhydrogen Atoms
in Crystalline $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$ ^{a,b}

Atom	Anisotropic Thermal Parameter ($\text{\AA}^2 \times 10$)					
Type ^c	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
$[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6]$						
Sn_1	38(1)	39(1)	30(1)	11(1)	9(1)	2(1)
Sn_2	37(1)	36(1)	29(1)	11(1)	2(1)	-2(1)
Sn_3	38(1)	40(1)	34(1)	6(1)	9(1)	-1(1)
Sn_4	37(1)	40(1)	35(1)	12(1)	5(1)	-5(1)
Sn_5	41(1)	42(1)	33(1)	11(1)	-1(1)	-9(1)
C_{1a}	46(6)	44(6)	37(6)	21(5)	9(5)	-2(5)
C_{2a}	67(8)	56(8)	43(7)	28(6)	15(6)	9(6)
C_{3a}	87(10)	63(9)	34(7)	5(8)	-7(7)	-6(6)
C_{4a}	74(10)	72(10)	45(8)	-9(8)	-22(7)	-16(7)
C_{5a}	65(8)	50(8)	57(8)	4(6)	0(7)	-5(6)
C_{6a}	50(7)	38(6)	47(7)	9(5)	-0(5)	-8(5)
C_{7a}	72(8)	94(10)	28(6)	17(7)	20(6)	-4(6)
C_{8a}	86(11)	158(18)	82(12)	25(11)	42(10)	-11(12)
C_{9a}	45(6)	43(6)	58(7)	3(5)	10(5)	-13(5)
C_{10a}	86(10)	55(8)	70(9)	13(7)	35(8)	2(7)
C_{1b}	30(5)	40(6)	38(6)	-1(4)	-12(4)	2(5)
C_{2b}	41(6)	41(6)	46(7)	3(5)	-6(5)	8(5)
C_{3b}	82(9)	59(8)	54(8)	25(7)	-9(7)	28(7)
C_{4b}	84(10)	60(9)	79(11)	33(8)	7(8)	20(8)
C_{5b}	55(7)	54(8)	52(7)	27(6)	2(6)	-20(6)
C_{6b}	35(5)	34(6)	39(6)	5(4)	2(4)	-16(4)
C_{7b}	70(8)	67(9)	42(7)	14(7)	1(6)	23(6)
C_{8b}	309(33)	106(16)	180(23)	-72(18)	188(24)	-43(15)

Table II. (continued)

Atom Type ^c	Anisotropic Thermal Parameter ($\text{\AA}^2 \times 10$)					
	B ₁₁	B ₂₂	B ₃₃	B ₁₂	B ₁₃	B ₂₃
C _{9b}	38(6)	53(7)	45(6)	14(5)	13(5)	-8(5)
C _{10b}	81(9)	63(8)	62(8)	32(7)	34(7)	-3(7)
C _{1c}	48(7)	65(8)	30(6)	28(6)	8(5)	-8(5)
C _{2c}	52(7)	91(10)	36(6)	39(7)	4(5)	-7(6)
C _{3c}	45(7)	130(13)	39(7)	30(7)	3(5)	-18(7)
C _{4c}	86(10)	113(13)	53(8)	62(9)	-2(7)	9(8)
C _{5c}	92(10)	79(10)	64(9)	53(9)	1(8)	8(7)
C _{6c}	71(8)	62(8)	35(6)	36(7)	11(6)	5(6)
C _{7c}	46(7)	80(10)	79(10)	16(7)	-5(7)	3(8)
C _{8c}	48(8)	95(12)	147(16)	-1(8)	12(9)	-46(11)
C _{9c}	83(10)	59(8)	56(8)	28(7)	13(7)	11(6)
C _{10c}	131(16)	103(15)	154(19)	47(13)	49(14)	44(14)
C _{1d}	56(6)	42(6)	41(6)	25(5)	12(5)	-3(5)
C _{2d}	54(7)	54(7)	58(8)	7(6)	26(6)	-4(6)
C _{3d}	90(10)	50(8)	56(8)	10(7)	26(7)	-14(6)
C _{4d}	73(9)	70(10)	59(9)	2(8)	1(8)	-26(7)
C _{5d}	64(8)	54(8)	52(8)	0(6)	-1(6)	-18(6)
C _{6d}	46(6)	35(6)	43(6)	-4(5)	3(5)	4(5)
C _{7d}	60(8)	104(11)	40(7)	12(7)	28(6)	-7(7)
C _{8d}	147(19)	309(34)	78(15)	-78(20)	-25(14)	51(18)
C _{9d}	48(6)	44(7)	68(8)	2(5)	14(6)	-6(6)
C _{10d}	90(11)	85(12)	113(14)	30(9)	46(10)	33(10)
C _{1e}	53(6)	37(6)	47(7)	18(5)	2(5)	-8(5)
C _{2e}	46(6)	33(6)	51(7)	9(5)	5(5)	-16(5)

Table II. (continued)

Atom	Anisotropic Thermal Parameter ($\text{\AA}^2 \times 10$)					
Type ^c	B ₁₁	B ₂₂	B ₃₃	B ₁₂	B ₁₃	B ₂₃
C _{3e}	80(9)	47(8)	76(10)	17(7)	11(8)	1(7)
C _{4e}	115(12)	60(9)	65(9)	38(8)	29(9)	-22(7)
C _{5e}	84(9)	63(9)	58(8)	15(7)	28(7)	-8(7)
C _{6e}	52(7)	51(7)	47(7)	8(5)	10(5)	-7(5)
C _{7e}	80(9)	43(7)	46(7)	1(6)	4(6)	1(5)
C _{8e}	164(18)	91(14)	95(14)	19(13)	24(12)	21(11)
C _{9e}	92(10)	84(11)	46(8)	28(8)	27(7)	8(7)
C _{10e}	198(27)	127(21)	359(42)	76(20)	103(27)	124(25)
C _{1f}	39(6)	60(7)	32(6)	22(5)	-3(4)	-17(5)
C _{2f}	43(6)	64(8)	77(9)	23(6)	-11(6)	-40(7)
C _{3f}	58(8)	82(10)	94(11)	13(7)	-19(8)	-46(8)
C _{4f}	61(8)	96(11)	77(10)	24(8)	-9(7)	-28(8)
C _{5f}	74(9)	92(11)	52(8)	46(8)	-10(7)	-17(7)
C _{6f}	53(7)	75(9)	35(6)	24(6)	-12(5)	-9(6)
C _{7f}	71(9)	49(8)	87(10)	8(7)	13(8)	-6(7)
C _{8f}	108(13)	94(13)	117(15)	6(10)	44(12)	9(11)
C _{9f}	77(9)	77(10)	40(7)	35(8)	2(6)	1(6)
C _{10f}	98(13)	127(16)	98(14)	30(11)	14(10)	38(12)
0.5 [Sn ₂ (C ₁₀ H ₁₃) ₆]						
Sn ₆	40(1)	31(1)	26(1)	9(1)	5(1)	-2(1)
C _{1g}	41(5)	30(5)	35(5)	13(4)	-1(4)	-7(4)
C _{2g}	51(7)	53(7)	44(7)	6(5)	1(5)	-6(6)
C _{3g}	49(7)	59(9)	67(9)	-2(6)	-17(6)	-7(7)
C _{4g}	68(9)	73(10)	66(9)	12(7)	-34(7)	-21(8)
C _{5g}	74(8)	51(8)	54(8)	9(6)	-26(6)	3(6)
C _{6g}	54(6)	37(6)	31(5)	14(5)	-4(5)	-5(4)

Atom Type ^c	Anisotropic Thermal Parameter ($\text{\AA}^2 \times 10$)					
	B ₁₁	B ₂₂	B ₃₃	B ₁₂	B ₁₃	B ₂₃
C _{7g}	66(8)	72(10)	56(8)	-19(7)	-14(7)	-3(7)
C _{8g}	70(11)	209(24)	215(25)	-32(13)	3(13)	139(21)
C _{9g}	62(7)	41(6)	51(7)	14(5)	5(6)	-3(5)
C _{10g}	117(12)	76(10)	36(7)	7(9)	21(7)	2(7)
C _{1h}	54(6)	36(6)	27(5)	15(5)	13(5)	4(4)
C _{2h}	41(6)	36(6)	49(7)	6(5)	15(5)	-1(5)
C _{3h}	51(7)	73(9)	84(10)	11(6)	23(7)	-9(8)
C _{4h}	80(10)	68(9)	96(12)	24(8)	62(9)	9(8)
C _{5h}	95(11)	73(9)	38(7)	35(8)	27(7)	-13(6)
C _{6h}	69(8)	40(6)	48(7)	32(6)	12(6)	-6(5)
C _{7h}	60(7)	55(7)	45(7)	20(6)	11(6)	-11(5)
C _{8h}	40(7)	133(14)	71(10)	25(8)	-8(6)	-26(9)
C _{9h}	88(10)	112(12)	31(6)	47(9)	-2(6)	-24(7)
C _{10h}	153(18)	64(12)	187(22)	21(12)	-56(15)	-18(13)
C _{1i}	36(5)	34(5)	29(5)	2(4)	-3(4)	7(4)
C _{2i}	43(6)	33(6)	51(7)	-5(5)	-8(5)	9(5)
C _{3i}	62(8)	47(7)	65(9)	14(6)	4(7)	23(7)
C _{4i}	115(12)	62(9)	49(8)	23(9)	11(8)	17(7)
C _{5i}	95(10)	55(8)	47(8)	9(7)	29(7)	13(6)
C _{6i}	51(6)	44(6)	35(6)	4(5)	10(5)	5(5)
C _{7i}	72(8)	52(8)	80(9)	31(6)	34(7)	29(7)
C _{8i}	101(12)	63(10)	92(12)	8(8)	1(9)	-24(8)
C _{9i}	66(8)	43(7)	60(8)	5(6)	26(6)	4(6)
C _{10i}	152(17)	149(17)	166(19)	94(14)	132(15)	87(15)

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.

^b The form of the anisotropic thermal parameter is given in reference 8 on page 6 of the structure report.

^c Atoms are labeled in agreement with Figures 1 and 2.

Table III. Atomic Coordinates for Hydrogen Atoms in Crystalline
 $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]^a$

Atom Type ^b	Fractional Coordinates		
	10^4x	10^4y	10^4z
$[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6]$			
H _{3a}	1121	1855	-1516
H _{4a}	-713	1437	-1453
H _{5a}	-1413	1778	-451
H _{7aa}	3124	2691	-225
H _{7ab}	2861	3389	-602
H _{8aa}	3882	2633	-1272
H _{8ab}	2694	2642	-1581
H _{8ac}	2919	1946	-1188
H _{9aa}	-1264	2698	560
H _{9ab}	-145	3122	1002
H _{10aa}	-904	2178	1522
H _{10ab}	53	1827	1271
H _{10ac}	-1130	1502	880
H _{3b}	997	6095	247
H _{4b}	273	6495	1251
H _{5b}	301	5756	2118
H _{7ba}	1376	4835	-476
H _{7bb}	2103	4370	-62
H _{8ba}	3422	5207	-366
H _{8bb}	3157	5704	289
H _{8bc}	2604	5768	-447
H _{9ba}	524	3894	2110
H _{9bb}	1709	4364	2405
H _{10ba}	505	4369	3251
H _{10bb}	-105	4811	2740
H _{10bc}	1028	5244	3160

Table III. (continued)

Atom Type ^b	Fractional Coordinates		
	10^4x	10^4y	10^4z
H _{3c}	8045	2290	2615
H _{4c}	7515	1050	2980
H _{5c}	5820	285	2653
H _{7ca}	6808	2952	1317
H _{7cb}	6360	3369	1940
H _{8ca}	7984	3963	1796
H _{8cb}	8619	3337	2043
H _{8cc}	7993	3778	2552
H _{9ca}	3914	468	1458
H _{9cb}	3647	889	2149
H _{10ca}	3136	-448	2034
H _{10cb}	3997	-113	2695
H _{10cc}	4331	-553	2036
H _{3d}	4663	369	-909
H _{4d}	2878	-297	-1235
H _{5d}	1571	28	-580
H _{7da}	6070	1160	178
H _{7db}	5733	1913	547
H _{8da}	6804	2233	-178
H _{8db}	5658	2403	-397
H _{8dc}	5991	1653	-774
H _{9da}	1076	1057	242
H _{9db}	1928	1482	887
H _{10da}	609	457	1084
H _{10db}	1785	358	1329
H _{10dc}	1134	-132	631

Table III. (continued)

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Atom Type ^b	Fractional Coordinates		
	10 ⁴ x	10 ⁴ y	10 ⁴ z
H _{3e}	3245	6997	2903
H _{4e}	2984	6978	4025
H _{5e}	3133	5892	4545
H _{7ea}	3355	5543	1694
H _{7eb}	4601	5825	1958
H _{8ea}	4051	6608	1239
H _{8eb}	4458	7104	1978
H _{8ec}	3212	6816	1716
H _{9ea}	4209	4758	4530
H _{9eb}	3978	4183	3822
H _{10ea}	2840	3698	4523
H _{10eb}	2197	3914	3873
H _{10ec}	2396	4484	4582
H _{3f}	8325	5543	3649
H _{4f}	8669	4581	4294
H _{5f}	7350	3405	4237
H _{7fa}	6972	6142	3054
H _{7fb}	5881	5605	2652
H _{8fa}	6737	5926	1968
H _{8fb}	6901	5044	1887
H _{8fc}	7896	5760	2148
H _{9fa}	5143	2695	3101
H _{9fb}	4686	3101	3726
H _{10fa}	5025	1936	3998
H _{10fb}	5876	2641	4465
H _{10fc}	6216	2150	3834

Table III. (continued)

Atom Type ^b	Fractional Coordinates		
	10^4x	10^4y	10^4z
0.5 [Sn ₂ (C ₁₀ H ₁₃) ₆]			
H _{3g}	-4077	-2411	3333
H _{4g}	-4104	-1620	2465
H _{5g}	-2637	-593	2462
H _{7ga}	-2146	-2192	4774
H _{7gb}	-2435	-2889	4145
H _{8ga}	-3716	-2941	4970
H _{8gb}	-4313	-2983	4221
H _{8gc}	-3820	-2151	4682
H _{9ga}	-905	406	3478
H _{9gb}	-130	-195	3477
H _{3h}	3858	-751	4569
H _{4h}	3714	-1406	3422
H _{5h}	1972	-1891	2782
H _{7ha}	2188	-918	5605
H _{7hb}	2002	-109	5388
H _{9ha}	40	-1740	2655
H _{9hb}	-510	-1852	3316
H _{3i}	524	-3019	6247
H _{4i}	-477	-2650	7087
H _{5i}	-1602	-1767	6872
H _{7ia}	1486	-2625	5289
H _{7ib}	860	-2289	4702
H _{9ia}	-2286	-1091	5428
H _{9ib}	-1433	-388	5896

Table III. (continued)

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- ^a Due to program limitations, only 13 of the 18 terminal methyl groups could be included in the structural model as rigid rotors with idealized sp^3 -geometry and a C-H bond length of 0.96Å; the methyl hydrogens which were omitted (those on C_{10g}, C_{8h}, C_{10h}, C_{8i} and C_{10i}) all belonged to the dimer. Of these 13 methyl groups, only 10 behaved well during least-squares refinement and gave final C-C-H angles which ranged from 91° to 125°; the remaining three methyl groups (C_{8d}, C_{8e}, and C_{10e}) were included in the structure factor calculations at fixed idealized staggered positions. For the 10 methyl groups which were refined as rigid rotors (C_{8a}, C_{10a}, C_{8b}, C_{10b}, C_{8c}, C_{10c}, C_{10d}, C_{8f}, C_{10f}, C_{10g} and their hydrogens), the initial orientation of each methyl group was determined from difference Fourier positions for the hydrogen atoms. The final orientation of each methyl group was determined by three rotational parameters. The remaining hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp^2 - or sp^3 -hybridization of the carbon atoms and a C-H bond length of 0.96Å) "riding" on their respective carbon atoms. The isotropic thermal parameter of each hydrogen atom was fixed at 1.2 times the equivalent isotropic thermal parameter of the carbon atom to which it is covalently bonded.
- ^b Hydrogen atoms are labeled with the same numerical and literal subscripts as their carbon atoms with an additional literal subscript (a, b or c) where necessary to distinguish between hydrogen atoms bonded to the same carbon.

Table IV. Bond Lengths and Angles Involving the Coordination Groups of Sn Atoms in Crystalline $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6]^- 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]^+$ ^a

Type ^b	Length, Å	Type ^b	Length, Å
$\text{Sn}_1\text{-Sn}_2$	2.871(1)	$\text{Sn}_3\text{-Sn}_2$	2.841(1)
$\text{Sn}_1\text{-Sn}_4$	2.852(1)	$\text{Sn}_3\text{-Sn}_4$	2.871(1)
$\text{Sn}_1\text{-Sn}_5$	2.849(1)	$\text{Sn}_3\text{-Sn}_5$	2.864(1)
$\text{Sn}_1\text{-Sn}_3$	3.367(1)	$\text{Sn}_6\text{-Sn}_6$, ^c	3.052(1)
$\text{Sn}_2 \cdots \text{Sn}_4$	3.990(1)		
$\text{Sn}_2 \cdots \text{Sn}_5$	3.981(1)		
$\text{Sn}_4 \cdots \text{Sn}_5$	4.029(1)		
$\text{Sn}_2\text{-C}_{1a}$	2.20(1)	$\text{Sn}_6\text{-C}_{1g}$	2.27(1)
$\text{Sn}_2\text{-C}_{1b}$	2.16(1)	$\text{Sn}_6\text{-C}_{1h}$	2.25(1)
$\text{Sn}_4\text{-C}_{1c}$	2.18(1)	$\text{Sn}_6\text{-C}_{1i}$	2.21(1)
$\text{Sn}_4\text{-C}_{1d}$	2.21(1)		
$\text{Sn}_5\text{-C}_{1e}$	2.22(1)		
$\text{Sn}_5\text{-C}_{1f}$	2.17(1)		
Type ^b	Angle, deg.	Type ^b	Angle, deg.
$\text{Sn}_2\text{Sn}_1\text{Sn}_3$	53.5(1)	$\text{Sn}_1\text{Sn}_2\text{C}_{1a}$	116.1(3)
$\text{Sn}_3\text{Sn}_1\text{Sn}_4$	54.2(1)	$\text{Sn}_3\text{Sn}_2\text{C}_{1a}$	121.2(3)

Table IV. (continued)

Type ^b	Angle, deg.	Type ^b	Angle, deg.
Sn ₃ Sn ₁ Sn ₅	54.1(1)	Sn ₁ Sn ₂ C _{1b}	123.0(3)
Sn ₁ Sn ₃ Sn ₂	54.3(1)	Sn ₃ Sn ₂ C _{1b}	115.4(3)
Sn ₁ Sn ₃ Sn ₄	53.7(1)	C _{1a} Sn ₂ C _{1b}	106.5(4)
Sn ₁ Sn ₃ Sn ₅	53.7(1)	Sn ₁ Sn ₄ C _{1c}	113.1(3)
Sn ₁ Sn ₂ Sn ₃	72.2(1)	Sn ₃ Sn ₄ C _{1c}	122.6(3)
Sn ₁ Sn ₄ Sn ₃	72.1(1)	Sn ₁ Sn ₄ C _{1d}	122.9(3)
Sn ₁ Sn ₅ Sn ₃	72.2(1)	Sn ₃ Sn ₄ C _{1d}	118.1(3)
Sn ₂ Sn ₁ Sn ₄	88.4(1)	C _{1c} Sn ₄ C _{1d}	105.9(5)
Sn ₂ Sn ₁ Sn ₅	88.2(1)	Sn ₁ Sn ₅ C _{1e}	115.7(3)
Sn ₄ Sn ₁ Sn ₅	89.9(1)	Sn ₃ Sn ₅ C _{1e}	123.8(3)
Sn ₂ Sn ₃ Sn ₄	88.6(1)	Sn ₁ Sn ₅ C _{1f}	125.6(3)
Sn ₂ Sn ₃ Sn ₅	88.5(1)	Sn ₃ Sn ₅ C _{1f}	114.8(3)
Sn ₄ Sn ₃ Sn ₅	89.3(1)	C _{1e} Sn ₅ C _{1f}	103.9(4)
C _{1g} Sn ₆ Sn ₆ , ^c	110.0(3)	C _{1g} Sn ₆ C _{1h}	108.2(4)
C _{1h} Sn ₆ Sn ₆ , ^c	110.4(3)	C _{1g} Sn ₆ C _{1i}	112.0(4)
C _{1i} Sn ₆ Sn ₆ , ^c	109.8(3)	C _{1h} Sn ₆ C _{1i}	106.4(4)

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.

^b Atoms are labeled in agreement with Figures 1 and 2.

^c Atoms labeled with a prime are related to nonprimed atoms by the crystallographic inversion center at 0,0,1/2 in the unit cell.

Table V. Bond Lengths for Ligand Nonhydrogen Atoms in Crystalline
 $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$ ^a

Type ^b	Length, Å								
	<u>Ligand</u>								
	<u>a</u>	<u>b</u>	<u>c</u>	<u>d</u>	<u>e</u>	<u>f</u>	<u>g</u>	<u>h</u>	<u>i</u>
C ₁ -C ₂	1.41(2)	1.40(2)	1.44(2)	1.41(2)	1.38(2)	1.39(2)	1.42(2)	1.38(2)	1.41(2)
C ₂ -C ₃	1.39(2)	1.41(2)	1.43(2)	1.45(2)	1.42(2)	1.42(2)	1.44(2)	1.38(2)	1.44(2)
C ₃ -C ₄	1.37(2)	1.40(2)	1.40(2)	1.36(2)	1.34(2)	1.35(3)	1.37(2)	1.40(2)	1.35(2)
C ₄ -C ₅	1.37(2)	1.33(2)	1.32(2)	1.38(2)	1.34(2)	1.38(2)	1.36(2)	1.39(2)	1.38(3)
C ₅ -C ₆	1.41(2)	1.39(2)	1.38(2)	1.41(2)	1.39(2)	1.38(2)	1.39(2)	1.37(2)	1.38(2)
C ₆ -C ₁	1.35(2)	1.45(2)	1.38(2)	1.39(2)	1.39(2)	1.40(2)	1.37(2)	1.40(2)	1.41(2)
C ₂ -C ₇	1.55(2)	1.50(2)	1.46(2)	1.51(2)	1.54(2)	1.52(2)	1.49(2)	1.53(2)	1.49(2)
C ₆ -C ₉	1.52(2)	1.48(2)	1.49(2)	1.51(2)	1.49(2)	1.54(2)	1.52(2)	1.52(2)	1.55(2)
C ₇ -C ₈	1.42(2)	1.41(3)	1.50(2)	1.34(3)	1.44(2)	1.45(3)	1.48(2)	1.50(2)	1.48(2)
C ₉ -C ₁₀	1.49(2)	1.51(2)	1.48(3)	1.44(2)	1.45(3)	1.49(3)	1.53(2)	1.47(3)	1.47(3)

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.

^b Atoms are labeled in agreement with Figures 1 and 2.

Table VI. Bond Angles for Ligand Nonhydrogen Atoms in Crystalline
 $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]^a$

Type ^b	Angle, deg.								
	<u>Ligand</u>								
	<u>a</u>	<u>b</u>	<u>c</u>	<u>d</u>	<u>e</u>	<u>f</u>	<u>g</u>	<u>h</u>	<u>i</u>
SnC_1C_2	122(1)	118(1)	120(1)	115(1)	123(1)	117(1)	121(1)	118(1)	123(1)
SnC_1C_6	118(1)	123(1)	119(1)	123(1)	116(1)	125(1)	119(1)	123(1)	118(1)
$\text{C}_2\text{C}_1\text{C}_6$	120(1)	119(1)	121(1)	122(1)	122(1)	118(1)	120(1)	120(1)	119(1)
$\text{C}_1\text{C}_2\text{C}_3$	120(1)	120(1)	116(1)	116(1)	117(1)	119(1)	117(1)	121(1)	117(1)
$\text{C}_1\text{C}_2\text{C}_7$	122(1)	121(1)	123(1)	124(1)	123(1)	124(1)	128(1)	124(1)	128(1)
$\text{C}_3\text{C}_2\text{C}_7$	118(1)	119(1)	122(1)	119(1)	121(1)	117(1)	114(1)	115(1)	115(1)
$\text{C}_2\text{C}_3\text{C}_4$	119(1)	121(2)	120(1)	123(2)	122(2)	122(1)	122(1)	121(1)	123(1)
$\text{C}_3\text{C}_4\text{C}_5$	121(1)	118(2)	122(2)	118(1)	121(2)	120(1)	119(1)	118(2)	118(2)
$\text{C}_4\text{C}_5\text{C}_6$	120(1)	126(1)	120(2)	123(1)	121(2)	120(2)	122(1)	122(1)	123(2)
$\text{C}_5\text{C}_6\text{C}_1$	120(1)	116(1)	121(1)	118(1)	118(1)	121(1)	120(1)	119(1)	120(1)
$\text{C}_5\text{C}_6\text{C}_9$	114(1)	123(1)	119(1)	117(1)	118(1)	120(1)	117(1)	115(1)	118(1)
$\text{C}_1\text{C}_6\text{C}_9$	126(1)	121(1)	121(1)	125(1)	124(1)	119(1)	123(1)	126(1)	122(1)
$\text{C}_2\text{C}_7\text{C}_8$	120(1)	116(1)	118(1)	117(1)	120(1)	115(1)	116(2)	121(1)	114(1)
$\text{C}_6\text{C}_9\text{C}_{10}$	113(1)	118(1)	118(2)	117(1)	116(2)	116(1)	118(1)	119(2)	120(1)

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.

^b Atoms are labeled in agreement with Figures 1 and 2.

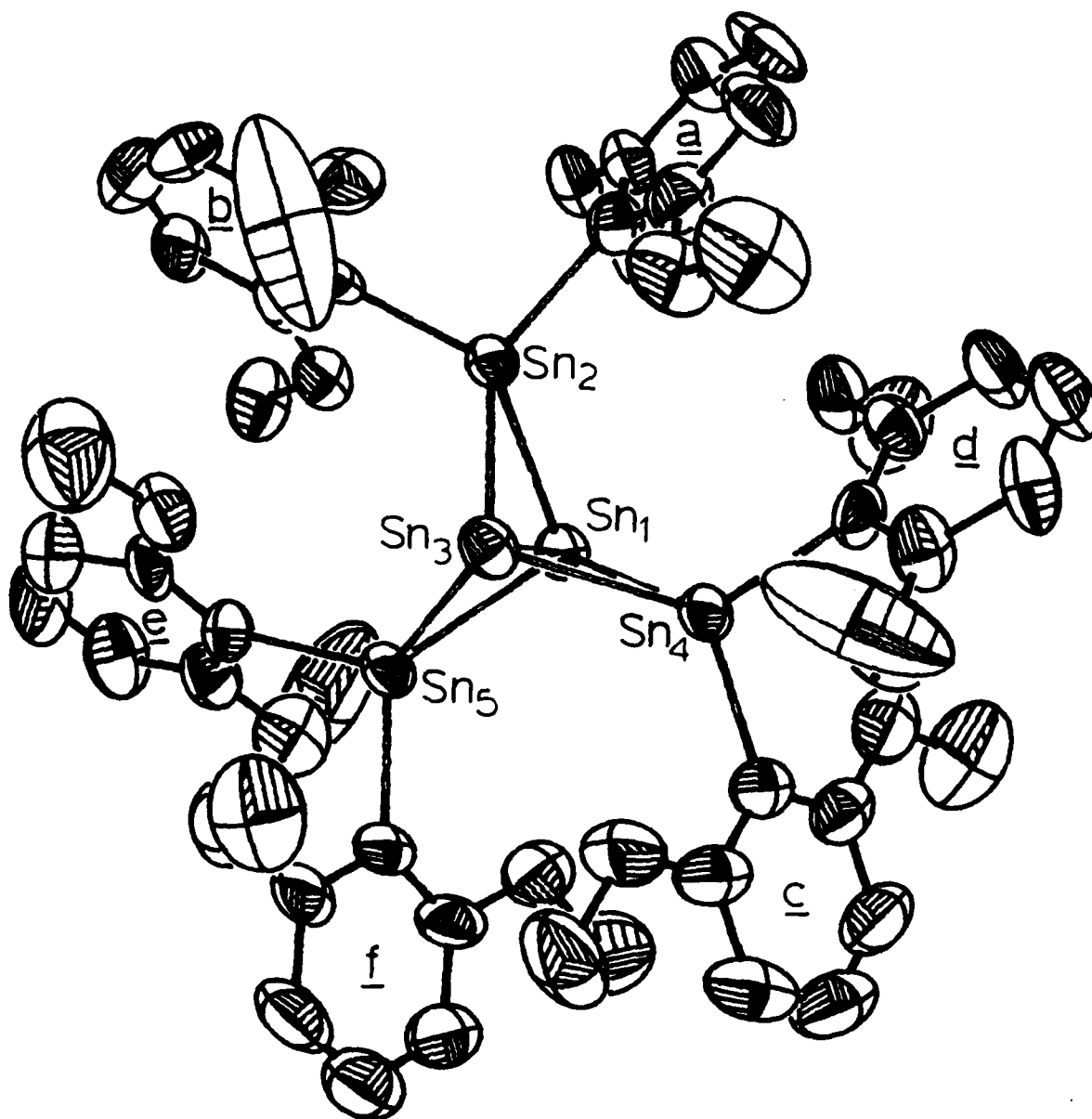


Figure 4. A perspective drawing of the nonhydrogen atoms for $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6]$ present in crystalline $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$; all atoms are represented by thermal vibration ellipsoids to encompass 50% of their electron density.

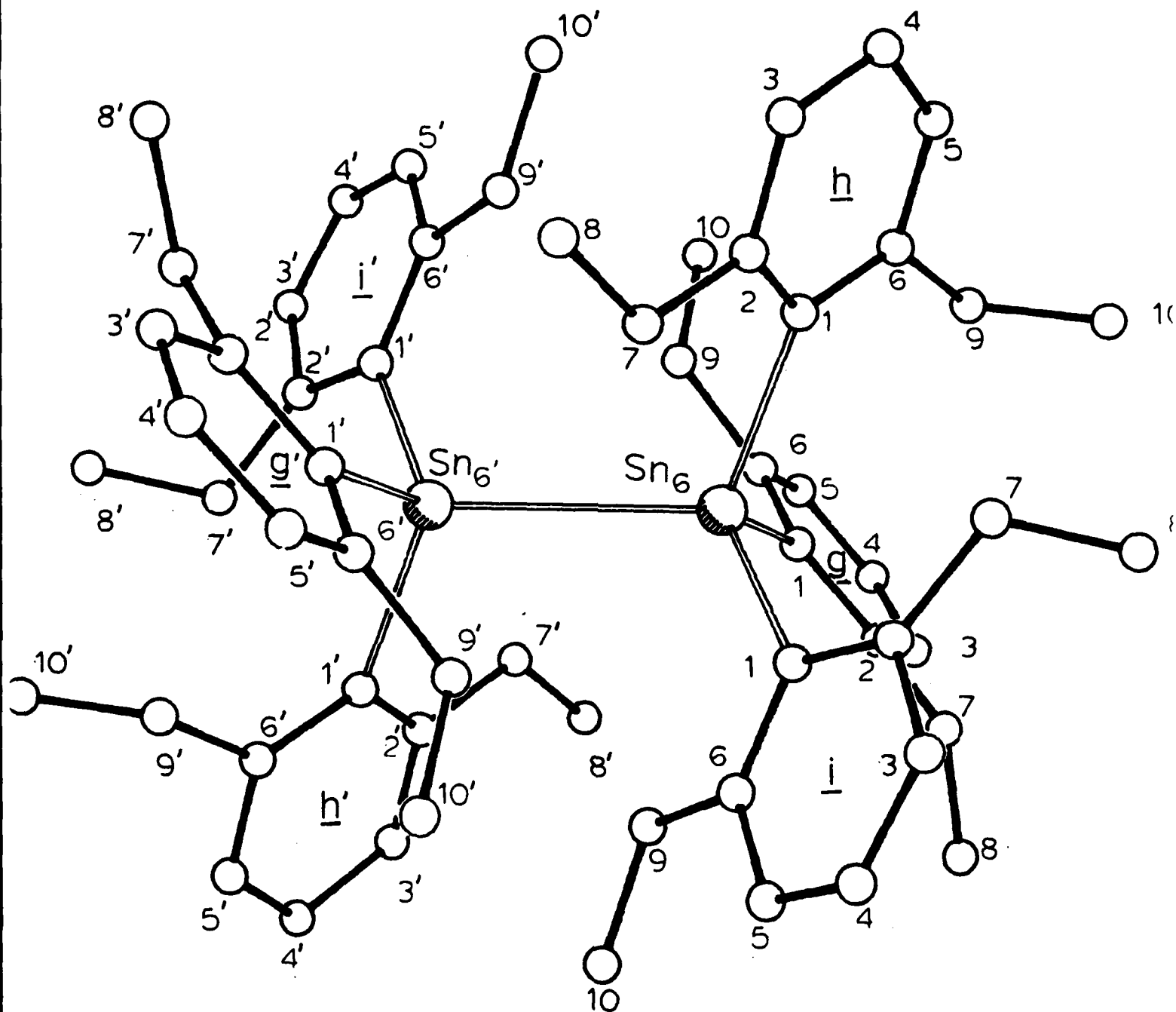


Figure 5. A perspective drawing of the nonhydrogen atoms for $[\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$ present in crystalline $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$ with metal atoms represented by large shaded spheres and carbon atoms represented by medium-sized open spheres. Atoms labeled with a prime (') are related to nonprimed atoms by the crystallographic inversion center at $0,0,1/2$ in the unit cell.

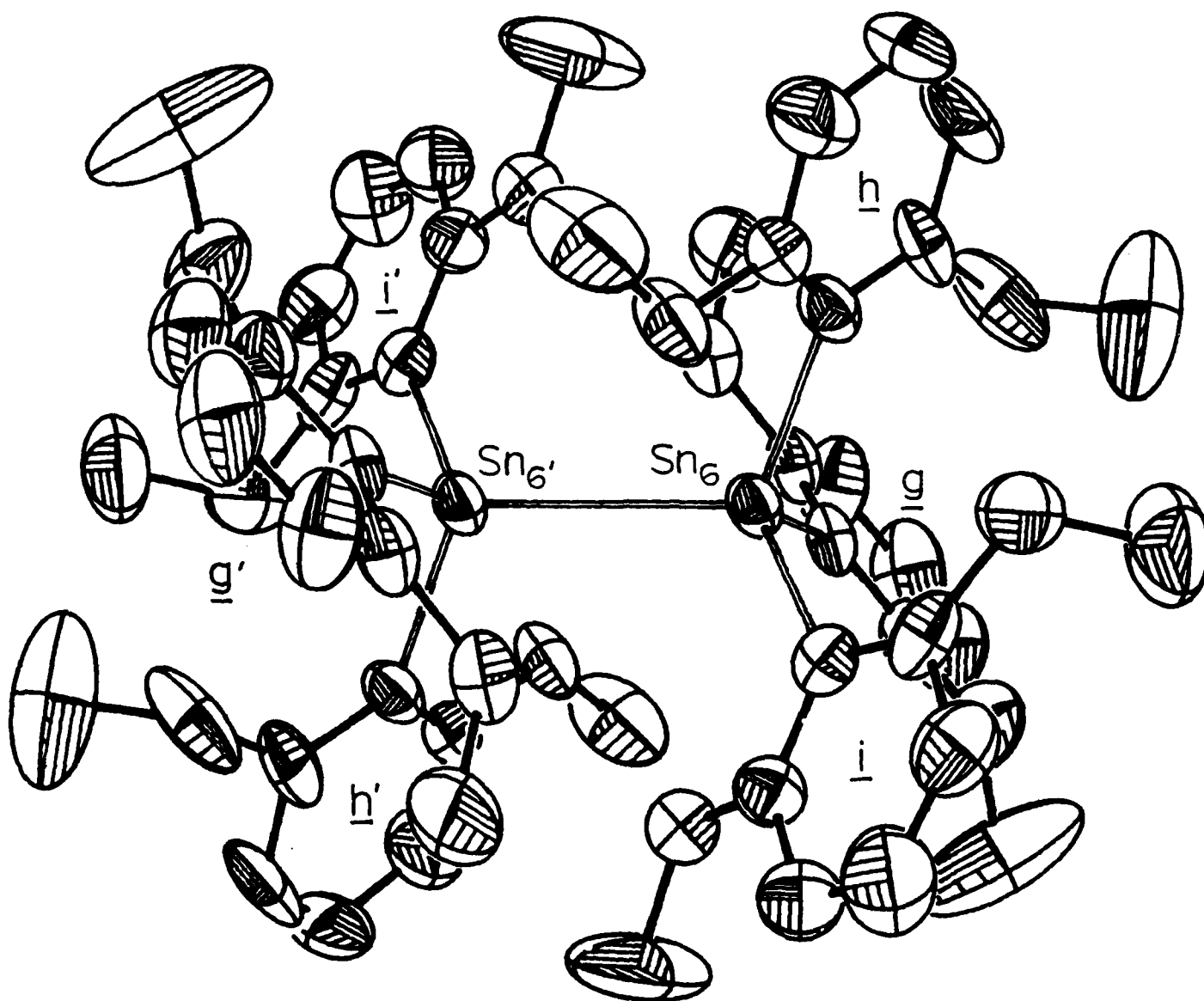


Figure 6. A perspective drawing of the nonhydrogen atoms for $[\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$ present in crystalline $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] - 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$; all atoms are represented by thermal vibration ellipsoids to encompass 50% of their electron density. Atoms labeled with a prime (') are related to nonprimed atoms by the crystallographic inversion center at $0,0,1/2$ in the unit cell.

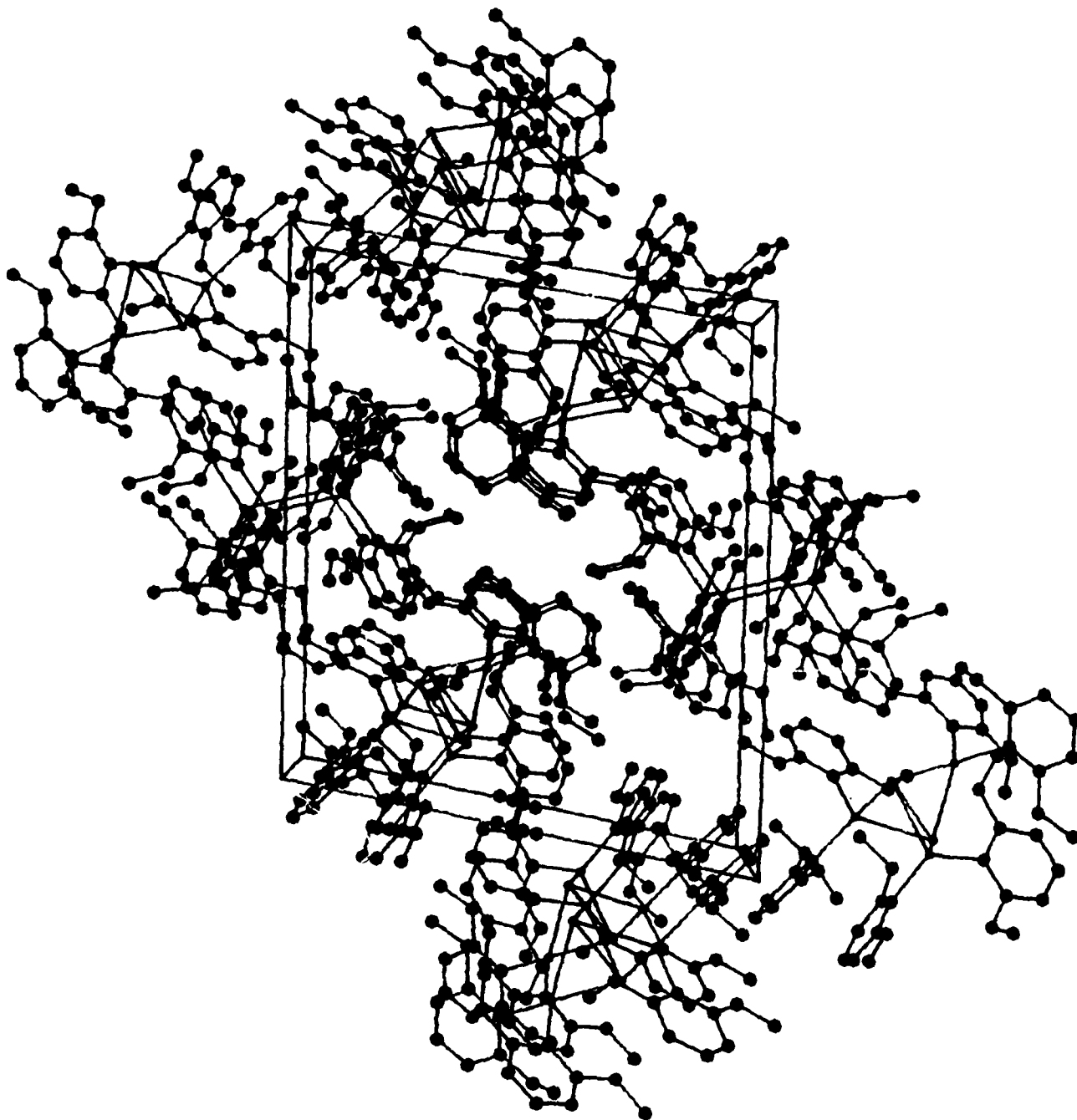


Figure 7. A perspective drawing of the crystal packing diagram for $[\text{Sn}_5(\text{C}_{10}\text{H}_{13})_6] \cdot 0.5 [\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6]$.