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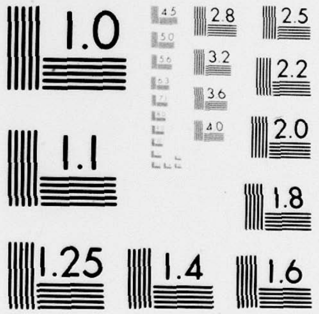

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Oxy- and Thio- Phosphorus Acid Derivatives of Tin. V. The X-ray Crystal and Molecular Structure of Bis-[O,O' Diisopropyldithiophosphate)diphenyltin(IV), a Monomeric, Molecular, Virtual Polymer.

by

K.C. Molloy, M.B. Hossain, D. van der Helm and J.J. Zuckerman
and I. Haiduc

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Norman, Oklahoma 73019

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Bis-(O,O'-diisopropopyldithiophosphato)diphenyltin(IV), $C_{24}H_{38}O_4S_4P_2Sn$, crystallizes in the monoclinic space $P2_1/n$ with a=17.517(7), b=14.212(7), c=6.341(8)Å, β=100.63(7)°, V=1551Å^3, Z=4, ρ_{calc}=1.50 gm cm^{-3} and μ=12.17cm^{-1}. The structure was determined by the heavy atom technique from 3195 reflections measured at 138±2K on an automatic diffractometer using monochromated Mo-K$_\alpha$ radiation to a final R value of 0.0522. The tightly packed, centro-symmetric structure consists of trans-diphenyltin(IV) units octahedrally coordinated

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by symmetrically chelating dithiophosphate ester ligands. There are short (3.30Å) intermolecular sulfur-sulfur atom contact distances bringing the tin atoms, which lie along the axis of propogation of the needle-like habit, to a distance of 6.34Å.

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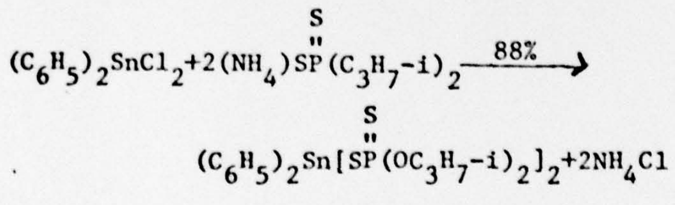
ABSTRACT

Bis-(0,0'-diisopropopyldithiophosphato)diphenyltin(IV), $C_{24}H_{38}O_4S_4P_2Sn$, crystallizes in the monoclinic space group $P2_1/n$ with a=17.517(7), b=14.212(7), c=6.341(8)Å, β=100.63(7)°, V=1551Å^3, Z=4, ρ_{calc}=1.50 gm cm^{-3} and μ=12.17cm^{-1}. The structure was determined by the heavy atom technique from 3195 reflections measured at 138±2K on an automatic diffractometer using monochromated Mo-K$_\alpha$ radiation to a final R value of 0.0522. The tightly packed, centrosymmetric structure consists of <u>trans</u>-diphenyltin(IV) units octahedrally coordinated by symmetrically chelating dithiophosphate ester ligands. There are short (3.30Å) intermolecular sulfur-sulfur atom contact distances bringing the tin atoms, which lie along the axis of propogation of the needle-like habit, to a distance of 6.34Å.

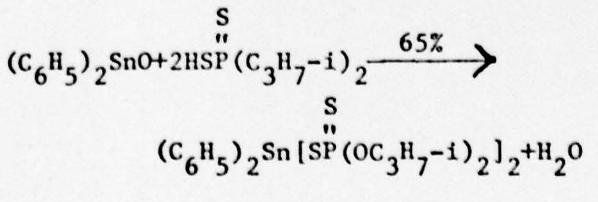
In the fourth part of this series we described the preparation of a series of diorganotin dithiophosphate esters, $R_2Sn\overset{S}{\overset{\shortparallel}{S}}P(OR')_2$, where $R=CH_3-$ and C_6H_5- and $R'=CH_3-$, C_2H_5-, $n-C_3H_7-$, $i-C_3H_7-$, $n-C_4H_9-$ and $i-C_4H_9-$, and their characterization by infrared, nmr and ^{119m}Sn Mössbauer spectroscopies.[1] Such systems are of interest structurally[2] since the dithiophosphate ester ligand can adopt a monodentate configuration, as we have found for 0,0'-diethyldithiophosphatotriphenyltin(IV), $(C_6H_5)_3SnS\overset{\overset{S}{\shortparallel}}{P}(OC_2H_5)_2$, which is described in Part II of this series[3], and is extremely rare.[4] Virtually all other known complexes of these ligands engage the metal in chelating or bridging bidentate behavior.

The title compound, 0,0'-diisopropyldithiophosphatodiphenyltin(IV), was synthesized by the action of diphenyltin dichloride on the ammonium salt of diisopropyldithiophosphoric acid:



$$(C_6H_5)_2SnCl_2+2(NH_4)S\overset{\overset{S}{\shortparallel}}{P}(C_3H_7-i)_2\xrightarrow{88\%}(C_6H_5)_2Sn[S\overset{\overset{S}{\shortparallel}}{P}(OC_3H_7-i)_2]_2+2NH_4Cl \qquad (1)$$

or by the action of diphenyltin oxide on the free acid:



$$(C_6H_5)_2SnO+2HS\overset{\overset{S}{\shortparallel}}{P}(C_3H_7-i)_2\xrightarrow{65\%}(C_6H_5)_2Sn[S\overset{\overset{S}{\shortparallel}}{P}(OC_3H_7-i)_2]_2+H_2O \qquad (2)$$

The white crystalline product which melts at 108°C was studied by tin-119m Mössbauer spectroscopy. The isomer shift (I.S.) ($=1.40\pm0.02$mm/s) and quadrupole splitting (Q.S.) ($=3.78\pm0.04$mm/s) values ($\rho=2.70$) specify a higher than four-coordinated situation at the tin atom.[5] The magnitude of the slope of the temperature dependence of the area under the Mössbauer resonance ($-1.06\times10^{-2}K^{-1}$) between 77 and 155K indicates that the tin atom is firmly held in the lattice,[6-8] more firmly so than in the corresponding bis-(O,O'-diethyldithiophosphato)-diphenyltin(IV), $(C_6H_5)_2Sn[S\overset{S}{\overset{\|}{P}}(OC_2H_5)_2]_2$, whose structure has recently been published. This latter material, which exhibits a slope of $-1.92\times10^{-2}K^{-1}$ between 77 and 150K, exists as monomeric, six-coordinated units with tin in a badly distorted octahedral environment with <u>trans</u>-diphenyl groups and anisobidentate dithiophosphate ligands about the girdle of the molecule. The two short Sn-S distances are <u>cis</u>-oriented, which allows an alternative view of the structure as a badly distorted tetrahedron about tin involving the two phenyl groups and two nearest sulfur atoms.[9] However, the rather wide C-Sn-C angle (135°) and acute S-Sn-S angles (69.2 and 84.5°), along with the tin-119m Mössbauer evidence make this interpretation seem less likely. Finally, a treatment of the Mössbauer and low-energy Raman data based upon the effective vibrating mass model developed by Herber[10] yields a unit molecularity for both compounds for the intermolecular, intra-unit cell, lattic mode observed at 28 and 34 cm^{-1} for the diethyl- and diisopropyl derivatives, respectively, in the Raman spectra.

We report in this paper the results of a single-crystal, X-ray diffraction investigation of the diisopropyl dithiophosphate ester to determine the manner in which this lattice is tightened with respect to its diethyl ester analogue.

Experimental Section

Crystal Data. Colorless, needle-like crystals of the title compound suitable for intensity measurements were obtained by slow evaporation of a benzene solution. A crystal of approximate dimensions 0.15mm x 0.15mm x 0.40mm was used in the initial diffractometry investigation and subsequent data collection. Data were obtained at 138 ± 2K with a CAD-4 counter diffractometer (Enraf-Nonius) controlled by a PDP8/e computer and fitted with a low temperature apparatus. Crystal data are listed in Table I.

Collection and Reduction of Intensity Data

All reflections with $2\theta \leq 53°$ were measured at 138 ± 2K on the above-mentioned diffractometer using θ-2θ scan techniques with variable scan rates (ν) using zirconium filtered, Mo-K_α radiation. The angular scan width was variable, and obtained from $(0.70 + 0.35 \tan\theta)°$. An aperture of variable width $(0.35 + 0.86 \tan\theta)$ mm and at constant height (6 mm) was located at 173mm from the crystal. Each reflection was scanned for a maximum of 60s, of which two-thirds was spend scanning the peak (P), the remaining time being divided equally between the left and right backgrounds (LB, RB respectively). Scaled intensities (I) were obtained from $I = [P - 2(LB + RB)]/\nu$.

Reflection $(\bar{6}\ 5\ 1)$ was used as standard, and its intensity monitored every 27 reflections. The maximum variation in the standard was ca. 7%, although for most of the data (80%), variation was less than 4%. Centering was based upon three reflections monitored every 100 data points; for angular change $>0.15°$, a new orientation matrix was automatically determined from a list of 15 reflections. In all, 3195 independent reflections were measured, of which 557 were considered indistinguishable from the background $[I \leq 2\sigma(I)]$ and were assigned an intensity of $T^{1/2}$ where $T = P + 2(LB + RB)$.

Structure factors for each reflection were assigned a weight $\omega_F=1/\sigma_F{}^2$, where $\sigma_F{}^2$ is given by

$$ {}_F{}^2=\frac{1}{2}\left[\frac{\sigma^2+(0.04I\nu)^2}{(Lp)(I\nu)}\right] $$

and $\sigma=T^{1/2}\nu$, and Lp is the Lorentz and polarization factor. Corrections for absorption were also made.

Structure Determination and Refinement

The position of the tin atom was obtianed from a Patterson map, and found to lie on the special position 0,0,0 (the crystallographic center of symmetry). The positions of the non-hydrogen light atoms were located by a succession of block-diagonal least squares refinements[11] followed by a difference Fourier synthesis. Subsequent cycles of least-squares refinement allowed the thermal parameters to vary isotropically, initially, and anisotropically in later stages, to yield an R-factor Of 0.054 for all data. At this point, correction was made for the anomalous dispersion of tin, and the structure further refined before all non-methyl hydrogen atoms were located from a final difference Fourier synthesis. Refinement was concluded when shifts in all parameters were less than one-third of their corresponding standard deviation. The final R factor was 0.033 for the 2599 reflections included in the least-squares calculations and 0.0522 over all data.

The scattering factors used were for neutral atoms, and were taken from Refs. 12 (Sn, C, P, S, O) and 13 (H).

Final positional and thermal parameters for non-hydrogen atoms are listed in Tables II and III, respectively. The corresponding parameters for the located hydrogen atoms, which are numbered according to the carbon to which they are bonded, are given in Table IV. Final intramolecular distances and angles are listed in Tables V and VI. Figures 1 and 2 show the asymmetric unit, with atomic numbering, and the contents of the unit cell, respectively.

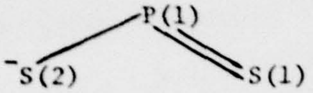
p-5

Description and Discussion of the Structure

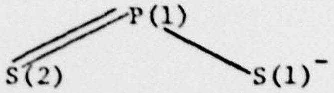
The tightening of the lattice in the title compound compared to its ethyl ester analogue[9], which was predicted by the tin-119m Mössbauer I.S. and Q.S. data, the slope of the plot of the area under the resonance curve vs. temperature, and the molecularity of the vibrating unit from the effective vibrating mass model using low-energy, lattice-mode Raman data, is seen in the more symmetrical, monomeric complex depicted in Figure 1, and shown packed into the asymmetric unit in Figure 2. The structure is centrosymmetric about the tin atom. The trans-diphenyltin octahedron with bidentate, 0,0'-diisopropyldithiophosphate ligands about the equatorial plane formed by the tin and four virtually equidistant sulfur atoms contrasts with the severely anisobidentate ligands in the diethyl ester analogue. The angles at tin between oppositely placed sulfur atoms are all 180°, and the tin-sulfur distances are 2.689(1) [Sn(1)-S(1)] and 2.678(1)Å[Sn(1)-S(2)]. The $Sn(SPS)_2$ moiety is essentially planar (equation of least-squares plane: $0.0012\,x - 0.096y - 0.115z = 0$) with deviations from this plane having a minimum of 0.00Å [for Sn(1)] and a maximum of ±0.027Å [for P(1) and P(1')].

The more tightly bound to the metal atom to which it is attached, then the longer is the bond that the sulfur atom in the dithiophosphate ester ligand makes with phosphorus.[4] All the complexes of this ligand but 0,0'-diethyldithiophosphatotriphenyltin(IV), whose structure we have recently solved,[3] and the adduct of $Ni[S_2P(OCH_3)_2]_2$ with 2,9-dimethyl-1,10-phenanthroline, which contain monodentate ligands,[14] are chelated and contain anisobidentate arrangements of the ligand sulfur atoms. The longer sulfur-metal internuclear distances in those complexes are ascribed to coordinate-covalent bonding, with the adjacent sulfur to phosphorus interaction written as a double, P=S bond. These shorter P=S distances are found to lie in the range 1.85-1.95 with the corresponding single P-S distances at 1.99-2.19Å.[4] The sulfur to phosphorus distances in the title compound [P(1)-S(1) = 1.998(2);

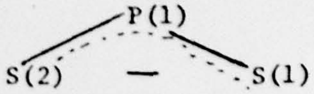
P(1)-S(2) = 2.006(2)Å] lie outside the P=S range, and at the low end of the single bond range. This is interpreted as reflecting extensive delocalizatin within the S-P-S moiety; that is, an almost equal contribution of canonical resonance form (B) to the overall electron distribution of the system (c).



(A)



(B)



(C)

Direct comparison may be made with the monomeric structure of the corresponding diethyl ester, bis-(0,0'-diethyldithiophosphato)diphenyltin(IV) torted octahedral with chelating, anisobidentate dithiophosphate ester ligands and where resonance form (A) dominates the electron distribution. The four sulfur atoms lie in an equatorial plane and the diphenyltin system makes an angle of 135°. The shorter sulfur-tin distances at 2.48 and 2.49 (shorter than ours) are on the same side of the plane, with the larger distances opposite at 3.20 and 3.23Å (longer than ours). This structure may be considered to be midway between tetrahedral and octahedral at tin. The sulfur-phosphorus distances found in the title compound [1.998(2) and 2.006(2)] are midway between those found for the P=S [1.92(3) and 1.94(1)] and P-S @2.03(1) and 2.04(2)Å] bonds in the ethyl ester analogue. The average of the two sets of sulfur-tin distances in the latter complex (2.85)^9 is somewhat larger than in the title compound (2.684Å).

It has been noted that in diorganotin systems, the magnitude of the Mössbauer Q.S. parameter is a function of the S-electron content of the Sn-C bonds, and, hence, the C-Sn-C angle. Moreover, the Q.S. value is found to increase from <u>ca</u>. 2.0mms^{-1} for the <u>cis</u>-isomer (C-Sn-C = 90°) to <u>ca</u>. 4.00 mms^{-1} for the <u>trans</u>-isomer (C-Sn-C = 180°)^5. We find that the Q.S. value increases from 3.12 to 3.67 mms in going from the diethyl for the iso-propylester, reflecting to the observed opening of the C-Sn-C angle from 135° to 180°.

The prismatic crystals of the ethyl ester analogue contrast with the needle-like appearance of the crystals of the title compound, and the much less severe dependence of the area of the tin-119m Mössbauer resonances with temperature of the latter would lead to the expectation of a polymeric structure. Indeed, in the lead(II) system the bis-(0,0'-diethyldithiophosphate) derivative is a chelated monomer,[15] while its diisopropyl analogue is a polymer whose structure involves anisobentate chelation from two dithiophosphate ester ligands which lie in a plane containing the lead(II) lone pair along with short, out-of-plane interactions with neighboring sulfur atoms to create pentagonal bypyramidal geometry about the lead atom.[16] However, in the zinc(II) series the diethyl ester forms a polymer,[17] while its diisopropyl analogue contains dimeric units in the solid.[18] Anisobidentate bridging is the rule in these and other related structures involvind the dithiophosphate ester group.[4]

The dithiocarbamate is also of interest. Here the dimethyltin(IV) derivatives are trans-octahedral,[19-21] but the structure of bis-(N-diethyldithiocarbamate)diphenyltin(IV) contains a cis-diphenyltin system with a C-Sn-C angle of 101.4°.[22] This change has been discussed in terms of the effect of the size of the phenyl group upon the minimization of the interligand repulsions and the of the tin-carbon and -sulfur bonding. In the present study, on the other hand, the change in the structure at tin from the ethyl analogues is produced by differences on the periphery of the molecule.

The molecules stack along the c-axis to give rise to the observed, needle-like habit. Figure 2 shows the arrangement of the ligands which produces the linear tin-tin array along c. In Figure 3 this arrangement is viewed from the side to show that the -S-P-S-units are not perpendicular to this axis (that is, the P---Sn---P vector is not normal to c), but rather that the ligand system is somewhat rotated about the tin atom to bring pairs of sulfur atoms on the opposite sides of alternating molecules together. The van der Waals radius of sulfur is given as 1.85[23]; the covalent radius as 1.04Å.[24] The shortest intermolecular sulfur-sulfur contact in this crystal at 3.30Å [S(2°)-S(2')] is thus intermediate in distance.

We note a remarkable fact. The relatively short tin-tin distance along the c-axis [6.341(8)Å] is within the bridging range for dithiophosphate ligands. For example, the distance between dithiophosphate-bridged zinc atoms in the $Zn[S_2P(OC_2H_5)_5]_2$ one-dimensional polymer is 6.82Å. That is, the four sulfur and two phosphorus atoms that chelate each tin in our structure could be rearranged to lie between instead of about the tin atoms, thereby linking together diphenyltin units at an equivalent distance into a polymer of equal dimensions and density. The anisotropic thermal parameters from the X-ray and the slope of the plot of Mössbauer resonance area *vs*. temperature would then not be expected to differ in the resulting polymer from those in the array of monomers actually found. The factors which determine how tightly the tin atoms are held include, *inter alia*, (1) the number of nearest neighbor atoms and their distance; (ii) the strength of the interactions arising from the packing of molecules in the lattice which may be bonding (as in a true polymer) or non-bonding (van der Waals forces); (iii) the mass of these neighboring units. None would be expected to change in the polymer corresponding to our structure. Our strcture may best be described then as a "virtual polymer" created by the packing forces which can operate more powerfully in this isopropyl ester derivative than in its ethyl analogue. These packing factors are reflected in the anomalously small slope of the Mössbauer resonance area *vs*. temperature.[1] Interpretation of variable temperature Mössbauer data should take this possibility into account.
So far as we are aware, the structure of the title compound is the first

example of a monomeric tin species with the same dimensions as its corresponding pooymeric form, although information concerning the location of the next nearest molecule and the shortest contact distances are surprisingly difficult to obtain from published structure reports.[2]

Acknowledgement. Our work is supported by the Office of Naval Research (J.J.Z.) and the National Cancer Institute, DHEW through grant CA-17562 (D.v.d.H.). We thank the University of Oklahoma for providing computer time, and the M&T Chemicals, Inc. for the donation of organotin starting materials.

Table I. Crystal Data

Formula	$C_{24}H_{38}O_4P_2S_4Sn$
Formula weight	699.49
Crystal system	Monoclinic
a,Å	17.517(7)
b,Å	14.212(7)
c,Å	6.341(8)
β,deg	100.63(7)
V,Å^3	1551
Space group	$P2_1/n$
Z	4
D_{calc} gm cm^{-3}	1.50
μ, cm^{-1}	12.17

Table II. Final Positional ($\times 10^4$) Parameters for the Non-Hydrogen Atoms in $(C_6H_5)_2Sn[S_2P(OC_3H_7)_2]_2$(a)

Atom	*x*	*y*	*z*
Sn(1)	0	0	0
P(1)	1225(1)	1537(1)	-1173(2)
S(1)	1030(1)	1305(1)	1789(2)
S(2)	533(1)	737(1)	-3335(2)
O(1)	2113(2)	1415(2)	-1313(5)
O(2)	1158(2)	2616(2)	-1808(5)
C(1)	-909(2)	1030(3)	-331(7)
C(2)	-1037(3)	1519(3)	1478(8)
C(3)	-1678(3)	2119(4)	1341(9)
C(4)	-2180(3)	2235(4)	-608(9)
C(5)	-2041(3)	1768(4)	-2412(9)
C(6)	-1409(3)	1168(3)	-2271(8)
C(7)	2474(3)	478(3)	-881(7)
C(8)	2769(3)	197(4)	-2883(8)
C(9)	3103(3)	575(4)	1093(9)
C(10)	385(3)	3064(3)	-2258(8)
C(11)	277(3)	3441(4)	-4531(9)
C(12)	369(4)	3819(4)	-584(9)

(a) Estimated standard deviations in parentheses.

Table III. Final Anisotropic Thermal Parameters$^{(a)}$ for Non-Hydrogen Atoms in $(C_6H_5)_2Sn[S_2P(OC_3H_7)_2]_2$$^{(b)}$

	U11	U22	U33	U12	U13	U23
n(1)	93(2)	120(2)	125(2)	-2(2)	23(1)	4(2)
(1)	119(5)	143(5)	140(5)	-22(4)	35(4)	-8(4)
(1)	152(5)	200(5)	111(5)	-39(4)	19(4)	-32(4)
(2)	162(5)	167(5)	95(5)	-22(4)	23(4)	-6(4)
(1)	131(15)	168(15)	279(18)	-16(12)	86(13)	28(13)
(2)	194(17)	137(15)	284(18)	-15(12)	69(13)	16(13)
(1)	150(21)	128(20)	213(23)	0(17)	52(17)	40(18)
(2)	183(23)	180(24)	261(25)	4(18)	42(18)	20(19)
(3)	315(27)	212(26)	380(31)	47(22)	95(23)	15(22)
(4)	267(27)	298(29)	489(35)	133(23)	60(24)	90(25)
(5)	239(27)	342(30)	347(30)	68(22)	-49(22)	85(24)
(6)	188(23)	230(25)	259(26)	15(19)	7(19)	32(20)
(7)	171(23)	193(25)	273(25)	25(18)	77(18)	2(20)
8)	305(27)	419(35)	272(27)	43(23)	86(21)	-50(23)
)	264(27)	535(39)	285(29)	126(25)	-26(22)	15(26)
10)	228(24)	149(24)	340(28)	21(15)	115(20)	-6(20)
1)	399(30)	385(33)	334(30)	43(23)	33(24)	132(26)
2)	560(38)	268(30)	412(33)	51(26)	219(29)	-124(25)

Anisotropic thermal parameters are of the form:-

$\exp\{-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2\underline{b}^{*2}+U_{33}l^2\underline{c}^{*2}+2U_{12}hk\underline{a}^*\underline{b}^*+2U_{13}hl\underline{a}^*\underline{c}^*+2U_{23}klb^*c^*)\}$.

Estimated standard deviations in parentheses.

Table III. Final Anisotropic Thermal Parameters[(a)] for Non-Hydrogen Atoms in $(C_6H_5)_2Sn[S_2P(OC_3H_7)_2]_2$[(b)]

	U11	U22	U33	U12	U13	U23
Sn(1)	93(2)	120(2)	125(2)	-2(2)	23(1)	4(2)
P(1)	119(5)	143(5)	140(5)	-22(4)	35(4)	-8(4)
S(1)	152(5)	200(5)	111(5)	-39(4)	19(4)	-32(4)
S(2)	162(5)	167(5)	95(5)	-22(4)	23(4)	-6(4)
O(1)	131(15)	168(15)	279(18)	-16(12)	86(13)	28(13)
O(2)	194(17)	137(15)	284(18)	-15(12)	69(13)	16(13)
C(1)	150(21)	128(20)	213(23)	0(17)	52(17)	40(18)
C(2)	183(23)	180(24)	261(25)	4(18)	42(18)	20(19)
C(3)	315(27)	212(26)	380(31)	47(22)	95(23)	15(22)
C(4)	267(27)	298(29)	489(35)	133(23)	60(24)	90(25)
C(5)	239(27)	342(30)	347(30)	68(22)	-49(22)	85(24)
C(6)	188(23)	230(25)	259(26)	15(19)	7(19)	32(20)
C(7)	171(23)	193(25)	273(25)	25(18)	77(18)	2(20)
C(8)	305(27)	419(35)	272(27)	43(23)	86(21)	-50(23)
C(9)	264(27)	535(39)	285(29)	126(25)	-26(22)	15(26)
C(10)	228(24)	149(24)	340(28)	21(15)	115(20)	-6(20)
C(11)	399(30)	385(33)	334(30)	43(23)	33(24)	132(26)
C(12)	560(38)	268(30)	412(33)	51(26)	219(29)	-124(25)

(a) Anisotropic thermal parameters are of the form:-

$$10^4 \exp\{-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2\underline{b}^{*2}+U_{33}l^2\underline{c}^{*2}+2U_{12}hk\underline{a}^*\underline{b}^*+2U_{13}hl\underline{a}^*\underline{c}^*+2U_{23}klb^*c^*)\}.$$

(b) Estimated standard deviations in parentheses.

Table V. Final Intramolecular Distances (Å) in $(C_6H_5)_2Sn[S_2P(OC_3H_7)_2]_2$[a]

Sn(1) --- S(1)	2.689(1)	C(1) --- C(2)	1.394(6)
Sn(1) --- S(2)	2.678(1)	C(2) --- C(3)	1.399(7)
Sn(1) --- C(1)	2.145(4)	C(3) --- C(4)	1.388(8)
		C(4) --- C(5)	1.383(8)
P(1) --- S(1)	1.998(2)	C(5) --- C(6)	1.387(7)
P(1) --- S(2)	2.006(2)	C(6) --- C(1)	1.386(6)
P(1) --- O(1)	1.584(3)		
P(1) --- O(2)	1.584(3)	C(2) --- H(2)	1.04(5)
		C(3) --- H(3)	1.07(2)
O(1) --- C(7)	1.476(6)	C(4) --- H(4)	1.03(6)
O(2) --- C(10)	1.478(6)	C(5) --- H(5)	1.01(6)
		C(6) --- H(6)	1.00(4)
C(7) --- C(8)	1.510(7)		
C(7) --- C(9)	1.514(7)	C(7) --- H(7)	1.00(5)
C(10) -- C(11)	1.517(8)	C(10) -- H(10)	1.02(4)
C(10) -- C(12)	1.514(8)		

[a] Estimated standard deviations in parentheses.

Table VI. Final Intramolecular Bond Angles (deg.) in $(C_6H_5)_2Sn[S_2P(OC_3H_7)_2]_2$(a)

C(1) --- Sn(1) --- C(1')	180
S(1) --- Sn(1) --- S(1')	180
S(2) --- Sn(1) --- S(2')	180
S(1) --- Sn(1) --- S(2)	76.03(3)
S(1) --- Sn(1) --- C(1)	89.29(11)
S(2) --- Sn(1) --- C(1)	90.94(12)
Sn(1) --- S(1) --- P(1)	86.26(5)
Sn(1) -- S(2) --- P(1)	85.38(5)
S(1) --- P(1) --- S(2)	111.26(7)
S(1) --- P(1) --- O(1)	111.98(12)
S(1) --- P(1) --- O(2)	112.38(13)
S(2) --- P(1) --- O(1)	112.04(13)
S(2) --- P(1) --- O(2)	111.57(13)
O(1) --- P(1) --- O(2)	96.88(17)
P(1) --- O(1) --- C(7)	118.65(27)
P(1) --- O(2) --- C(10)	119.28(27)

O(1) --- C(7) --- C(8)	106.52(37)
O(1) --- C(7) --- C(9)	107.01(38
C(8) --- C(7) --- C(9)	114.16(42)
O(2) --- C(10) --- C(11)	106.57(39)
O(2) --- C(10) --- C(12)	107.73(39)
C(11) --- C(10) --- C(12)	113.61(44)
Sn(1) --- C(1) 1-- C(2)	119.27(32)
Sn(1) --- C(1) --- C(6)	121.29(32)
C(2) --- C(1) --- C(6)	119.24(41)
C(1) --- C(2) --- C(3)	120.11(43)
C(2) --- C(3) --- C(4)	119.78(48)
C(3) --- C(4) --- C(5)	120.03(51)
C(4) --- C(5) --- C(6)	120.14(49)
C(5) --- C(6) --- C(1)	120.67(44)

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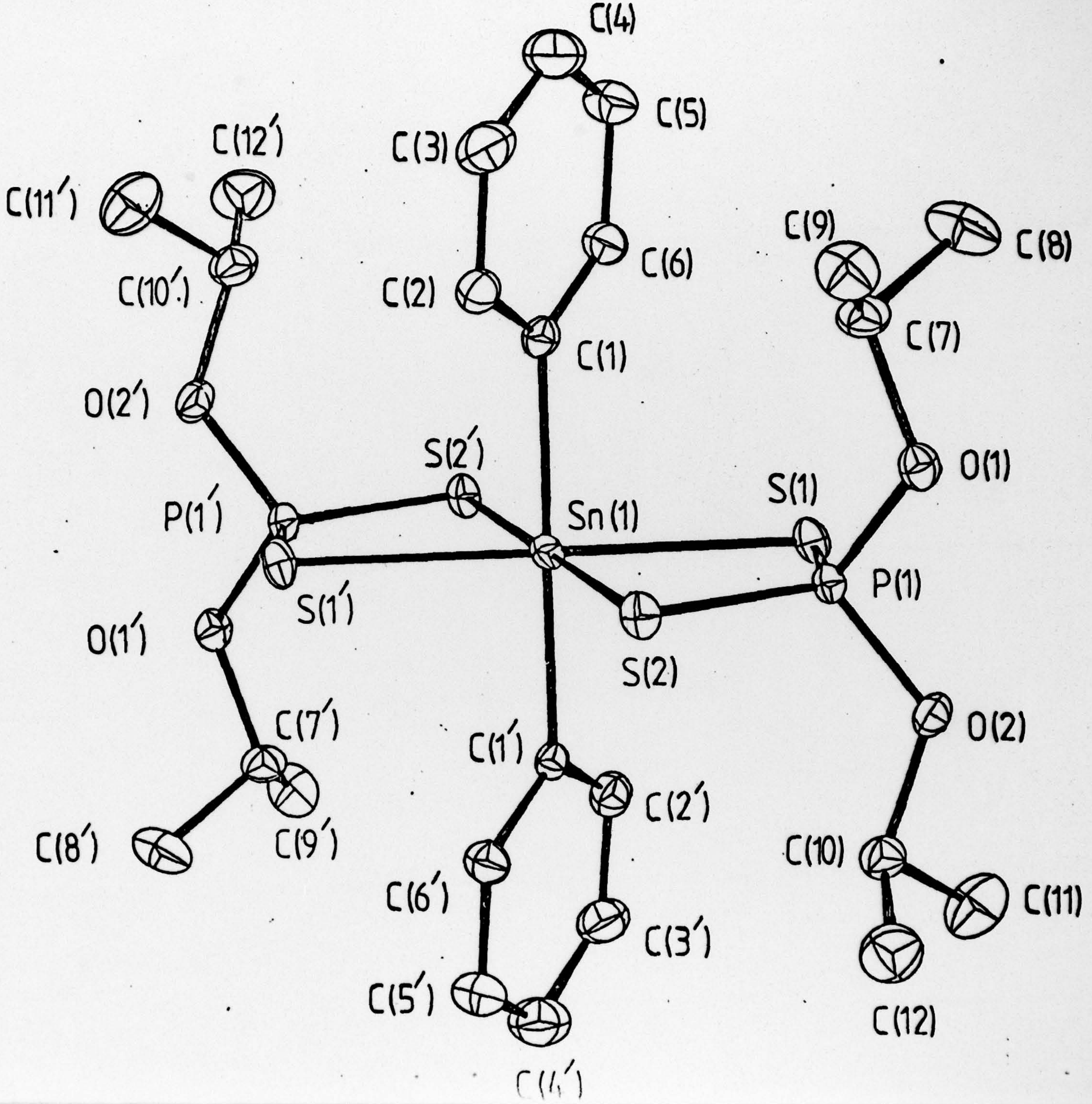
[a] Estimated standard deviations in parentheses.

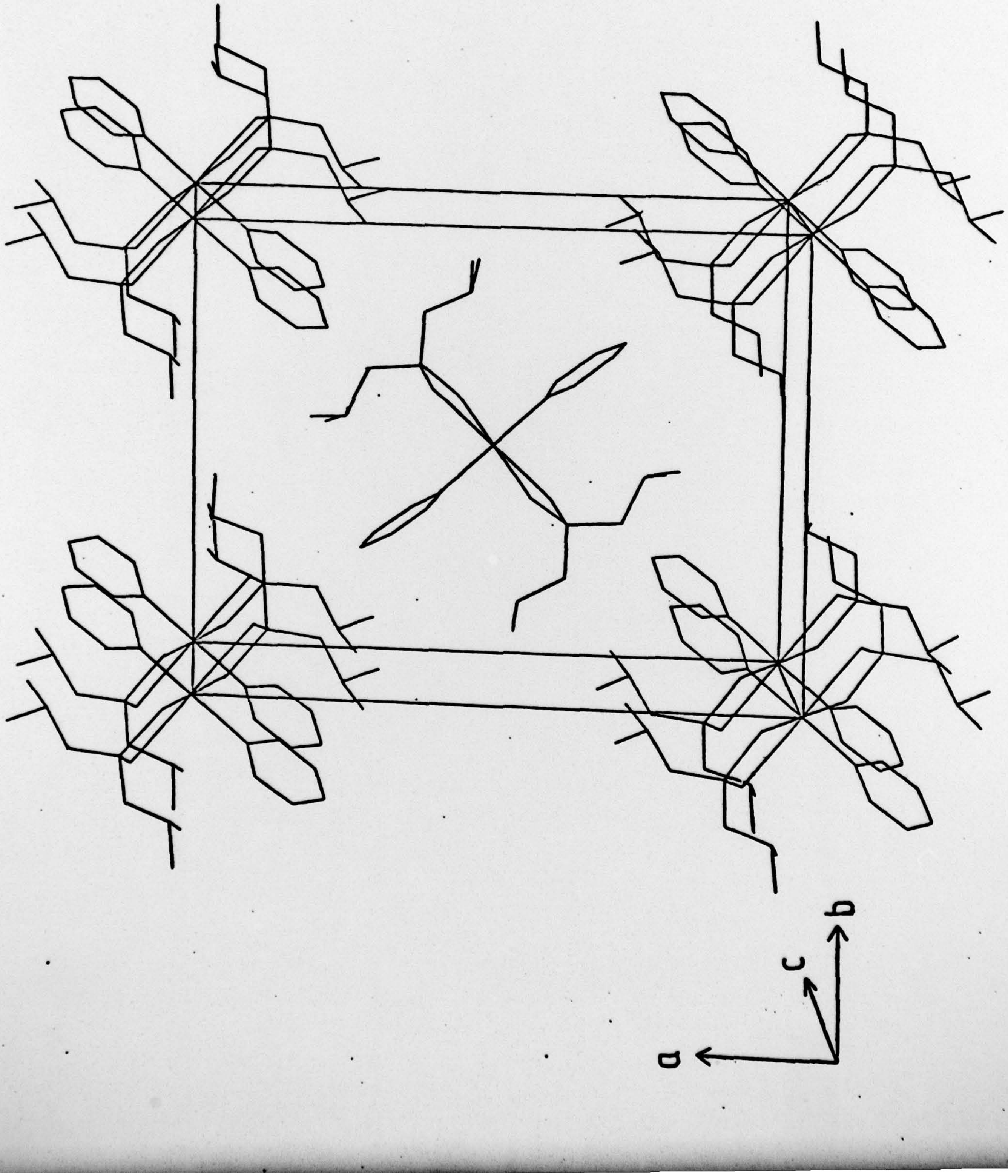
Figure Captions

Figure 1. The molecular structure of bis-(0,0'-diisopropyldithiophosphato)diphenyltin(IV) showing the atomic labeling. Primed atoms are related to the corresponding unprimed atoms by inversion center at Sn(1).

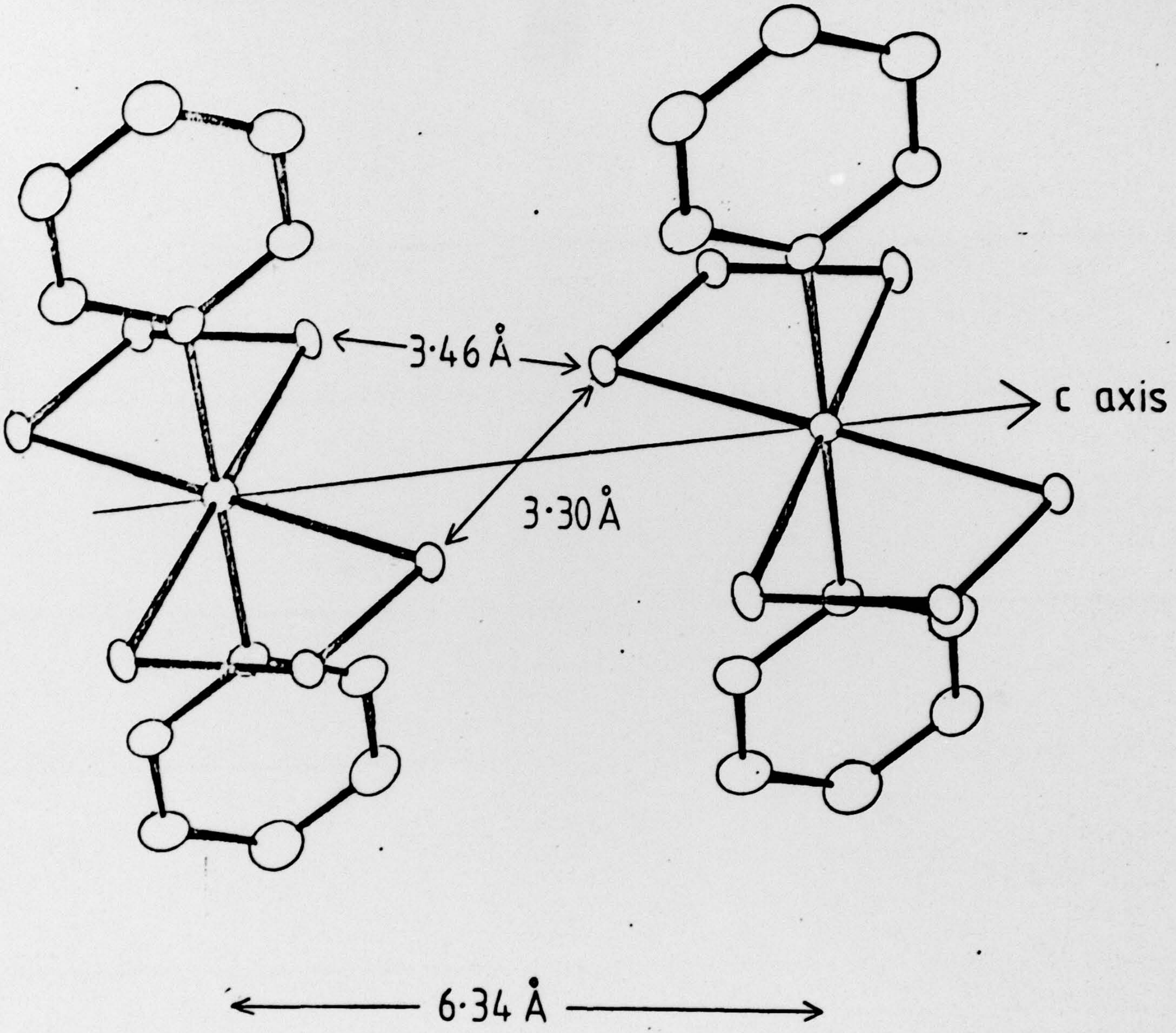
Figure 2. The contents of the unit cell showing the molecular packing.

Figure 3. A view of the *c*-axis of propogation showing the nearest intermolecular sulfur-sulfur atoms contacts and the tin-tin distance. Singly primed atoms are related to the corresponding unprimed atoms by the inverstion center at Sn(1). Doubly primed atoms are related to the corresponding unprimed atoms by a translation of one unit cell length along the *c*-axis.


C(4)
C(5)
C(12´)
C(3)
C(11´)
C(9)
C(8)
C(6)
C(10´.)
C(2)
C(7)
C(1)
O(2´)
S(2´)
O(1)
S(1)
P(1´)
Sn(1)
P(1)
S(1´)
O(1´)
S(2)
C(7´)
O(2)
C(1´)
C(2´)
C(8´)
C(9´)
C(10)
C(6´)
C(11)
C(3´)
C(5´)
C(12)
C(4´)



a
b
c


3·46 Å
c axis
3·30 Å
6·34 Å