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SPECTROSCOPIC PROPERTIES AND X-RAY CRYSTAL STRUCTURE OF A DINUCLEAR TIN ADDUCT: μ-BIS(DIPHENYLPHOSPHINYL)ETHANEBIS(CHLOROTRIPHENYLTIN)

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Summary

The crystal structure of a dinuclear adduct of formula $[(SnPh_3Cl)_2(OPPh_2CH_2)_2]$ has been determined by X-ray diffraction, using counter data. The compound crystallizes in the monoclinic space group $P2_1/n$, with a = 12.271(7), b = 25.690(13), c = 9.327(5) Å, β = 101.34(6)°, Z = 4. The final R value from 3832 observed reflections was 0.0367. The tin atom has an essentially trigonal bipyramidal coordination with the phenyl rings in the equatorial positions and the chlorine ion and the phosphorus ligand occupying the axial sites.

Introduction

During our previous work on the synthesis and the structural characterization of adducts of organotin derivatives with dioxo-diphosphines we were able to confirm the versatile ligand behaviour of the latter species towards metal ions. In particular, the adduct $[(SnPh_3NO_3)_2(OPPh_2CH_2)_2]$ was found to have a dinuclear nature with the phosphorus ligand bridging two tin atoms through the two oxygen atoms [1], whilst in $[SnPh_3Cl(OPPh_2CH)_2]$ the phosphorus ligand shows a unusual unidentate behaviour which is assumed to be a consequence of various factors including the stoicheiometry of the complex, the geometry of the ligand, and the tendency of the triphenyltin moiety to produce a trigonal-bipyramidal stereochemistry [2].

In this paper we report the synthesis and the structure of a dinuclear adduct of formula $[(SnPh_3Cl)_2(OPPh_2CH_2)_2]$, in which 1,2-bis(diphenylphosphinyl)-ethane again behaves as a bidentate agent using both of its oxygen atoms to bridge two tin atoms.

Experimental

Preparation

An acetone solution of triphenyltin chloride and 1,2-bis(diphenylphosphine)ethane (2 : 1 molar ratio) was stirred at room temperature for 1 h, and then set aside. After two days the title compound crystallized as colourless prisms. The compound can be also obtained from the same reactants by another procedure [3].

Crystal structure determination

Rotation and Weissenberg photographs taken with Cu-K_α radiation gave the preliminary unit-cell parameters and also indicated that the crystals were monoclinic, with systematic absences ($h0l$, $h + l = 2n + 1$; $0k0$, $k = 2n + 1$) consistent with the space group $P2_1/n$, a non standard setting of the space group $P2_1/c$. A crystal of approximate dimensions 0.32 × 0.21 × 0.29 mm was chosen for X-ray study and was mounted on an on-line Siemens AED single-crystal diffractometer with the c axis coincident with the φ axis of the instrument. The angular setting of 18 reflections with 2θ values in the range 24.3—44.6° were carefully determined to obtain the refined unit-cell dimensions, which together with other pertinent crystal data, are listed in Table 1.

Intensity data were taken at room temperature by use of niobium-filtered Mo-K_α radiation with the takeoff angle set to 4°. The moving-counter, moving-crystal scan technique was used, with a drive speed related to the number of counts on the peak. The pulse-height discriminator was set to accept 90% of the Mo-K_α peak. A total of 6502 independent reflections were collected over the range $6.0° < 2\theta < 53.0°$; symmetry-equivalent reflections were merged to produce a unique set of 5993 reflections, of which 3832 (64%) were considered observed at the $2\sigma(I)$ significance level and used in the structure refinement. The 4,4,2 reflection was chosen as standard and its intensity, monitored periodically throughout the data collection, showed only random deviations from its mean value. The intensities were corrected for Lorentz, polarization and absorption effects.

A three-dimensional Patterson map was used to obtain the coordinates of the tin atom. This was followed by a series of structure factor calculations and difference Fourier maps which revealed the positions of all non-hydrogen atoms. Two cycles of full-matrix least-squares refinement gave convergence at R 0.0693 with isotropic thermal parameters, and after two further cycles of

TABLE 1

CRYSTAL DATA

Empirical formula	$C_{62}H_{54}Cl_2O_2P_2Sn_2$
Formula weight	1201.34
Monoclinic. Space group	$P2_1/n$
$a = 12.271(7)$ A	$Z = 2$
$b = 25.690(13)$	$D_c = 1.384$ g cm^{-3}
$c = 9.327(5)$	λ(Mo-K_α) = 0.71069 A
$\beta = 101.34(6)°$	μ(Mo-K_α) = 10.56 cm^{-1}
$V = 2883(3)$ A^3	$F(000) = 1212$

TABLE 2

FRACTIONAL ATOMIC COORDINATES ($\times 10^5$ for Sn, $\times 10^4$ for Cl, P, O, and C) AND ANISOTROPIC THERMAL PARAMETERS ($\times 10^4$), WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES, FOR NON-HYDROGEN ATOMS

Atom	*x/a*	*y/b*	*z/c*	Atom	*x/a*	*y/b*	*z/c*
Sn	28 562(2)	11 646(1)	21 491(3)	C(15)	1073(8)	2653(4)	1551(17)
Cl	4 712(1)	1 381(1)	3 607(1)	C(16)	1364(9)	2892(4)	2855(19)
P	31(1)	852(0)	—181(1)	C(17)	2018(8)	2651(4)	3984(15)
O	1 022(2)	966(1)	974(3)	C(18)	2458(6)	2159(3)	3806(9)
C(1)	2 709(5)	470(2)	3 349(6)	C(19)	—1222(4)	983(2)	459(6)
C(2)	1 759(6)	345(3)	3 894(7)	C(20)	—1169(6)	1268(3)	1732(7)
C(3)	1 726(9)	—126(4)	4 631(9)	C(21)	—2162(10)	1366(4)	2229(11)
C(4)	2 590(11)	—465(4)	4 793(10)	C(22)	—3149(9)	1201(5)	1440(15)
C(5)	3 521(9)	—342(4)	4 255(10)	C(23)	—3198(6)	929(4)	204(14)
C(6)	3 580(6)	117(3)	3 552(8)	C(24)	—2239(4)	813(3)	—324(9)
C(7)	3 330(5)	1 045(3)	107(6)	C(25)	—19(4)	1228(2)	—1836(5)
C(8)	3 370(6)	546(4)	—449(8)	C(26)	727(5)	1134(3)	—2736(7)
C(9)	3 613(8)	461(6)	—1 812(11)	C(27)	744(6)	1445(3)	—3917(8)
C(10)	3 824(7)	858(7)	—2 656(11)	C(28)	62(7)	1862(3)	—4209(9)
C(11)	3 802(7)	1 355(6)	—2 160(10)	C(29)	—668(8)	1961(4)	—3311(11)
C(12)	3 561(6)	1 462(4)	—743(8)	C(30)	—698(7)	1649(3)	—2120(9)
C(13)	2 181(5)	1 904(2)	2 470(8)	C(31)	—12(4)	171(2)	—670(5)
C(14)	1 490(6)	2 148(3)	1 328(11)				

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sn	360(2)	409(2)	402(2)	—4(1)	48(1)	19(1)
Cl	374(6)	785(8)	665(8)	—72(6)	—21(5)	—43(6)
P	315(5)	406(6)	462(6)	21(5)	32(4)	8(4)
O	353(15)	521(17)	577(19)	10(15)	—46(14)	—37(13)
C(1)	485(32)	431(30)	393(28)	44(23)	14(25)	—30(24)
C(2)	665(45)	759(49)	475(34)	47(32)	97(31)	—471(57)
C(3)	936(65)	1198(73)	669(50)	272(48)	113(46)	—471(57)
C(4)	1614(103)	678(64)	727(55)	272(47)	—241(60)	—154(67)
C(5)	1037(76)	719(55)	930(60)	251(46)	—162(54)	168(53)
C(6)	651(49)	578(40)	642(41)	77(33)	0(37)	113(35)
C(7)	358(28)	734(47)	445(30)	—18(28)	72(23)	—71(27)
C(8)	677(44)	997(65)	639(43)	—309(42)	205(35)	—36(41)
C(9)	801(56)	1648(118)	748(59)	—423(70)	162(46)	138(66)
C(10)	643(47)	2364(160)	520(58)	—389(79)	169(40)	—9(68)
C(11)	642(45)	1831(126)	695(54)	494(72)	157(38)	—250(61)
C(12)	698(42)	1057(79)	603(43)	91(49)	145(33)	—181(36)
C(13)	431(30)	429(31)	777(43)	—25(28)	80(29)	13(24)
C(14)	674(44)	540(43)	1224(74)	82(46)	—230(46)	23(34)
C(15)	736(55)	525(56)	2311(127)	193(70)	—353(68)	126(42)
C(16)	681(58)	543(63)	2381(158)	—277(82)	134(78)	—24(49)
C(17)	834(57)	802(62)	1613(108)	—616(68)	505(64)	—201(47)
C(18)	538(41)	590(43)	871(58)	—187(41)	255(41)	63(34)
C(19)	408(27)	597(33)	544(33)	186(26)	66(24)	76(24)
C(20)	744(48)	876(51)	620(39)	168(35)	252(36)	260(39)
C(21)	1380(87)	1387(79)	983(64)	540(60)	779(66)	732(69)
C(22)	923(65)	1508(88)	1744(102)	968(79)	899(75)	685(65)
C(23)	452(41)	1168(71)	1776(99)	650(68)	363(57)	72(43)
C(24)	363(29)	806(34)	1053(58)	228(40)	71(32)	1(27)
C(25)	405(25)	507(30)	509(27)	80(24)	69(21)	23(22)
C(26)	599(33)	743(43)	642(35)	90(32)	190(27)	40(31)
C(27)	809(46)	991(53)	667(41)	119(39)	284(36)	13(40)
C(28)	1031(56)	1114(60)	824(54)	456(47)	316(46)	158(47)
C(29)	1387(72)	1182(72)	1480(79)	850(64)	743(63)	787(61)
C(30)	1091(56)	1002(53)	1143(58)	614(45)	662(49)	588(44)
C(31)	562(27)	410(24)	542(27)	2(20)	29(22)	—11(20)

TABLE 3

FRACTIONAL ATOMIC COORDINATES ($\times10^3$) AND THERMAL PARAMETERS ($\times10^3$), WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES, FOR HYDROGEN ATOMS

Atom	x/a	y/b	z/c	U	Atom	x/a	y/b	z/c	U
H(2)	119(5)	59(2)	384(6)	98(16)	H(18)	305(5)	196(2)	479(6)	80(16)
H(3)	112(5)	−20(2)	496(6)	91(16)	H(20)	−46(4)	137(2)	232(5)	80(14)
H(4)	265(5)	−82(2)	511(6)	96(16)	H(21)	−199(4)	157(2)	321(6)	117(14)
H(5)	423(5)	−61(2)	432(6)	102(16)	H(22)	−384(4)	130(2)	186(5)	103(14)
H(6)	418(5)	22(2)	311(6)	61(17)	H(23)	−392(4)	77(2)	−52(5)	117(14)
H(8)	312(5)	20(2)	13(6)	86(16)	H(24)	−231(4)	64(2)	−154(6)	94(14)
H(9)	383(4)	1(2)	−210(6)	118(16)	H(26)	130(4)	84(2)	−243(5)	81(14)
H(10)	392(5)	87(2)	−367(7)	96(16)	H(27)	129(4)	135(2)	−456(5)	100(14)
H(11)	371(5)	168(2)	−253(7)	100(17)	H(28)	7(4)	203(2)	−509(5)	72(14)
H(12)	341(5)	182(2)	−35(6)	86(16)	H(29)	−110(4)	225(2)	−338(5)	105(14)
H(14)	123(5)	197(2)	23(6)	53(17)	H(30)	−123(4)	168(2)	−146(5)	92(14)
H(15)	73(5)	278(2)	71(7)	87(17)	H(31A)	62(4)	12(2)	−115(5)	67(14)
H(16)	110(5)	328(2)	283(6)	112(16)	H(31B)	−60(4)	12(2)	−138(6)	63(14)
H(17)	216(5)	282(2)	484(6)	116(16)					

refinement with anisotropic thermal parameters R converged at 0.0434. At this point a ΔF map produced well defined electron densities for all the hydrogen atoms. Inclusion of these last atoms with isotropic thermal parameters in further least-squares cycles yielded, at convergence, $R = 0.0367$ and $R_w = 0.0391$ (3832 reflections, 424 parameters). A final ΔF map showed no regions of significant electron density.

Complex neutral-atom scattering factors corrected for anomalous dispersion were employed with the weighting scheme: $w = 1/[\sigma^2(F_0) + 0.005|F_0|^2]$ for the final stages of refinement. Final atomic coordinates and thermal parameters are listed in Tables 2 and 3. A listing of observed and calculated structure factors can be obtained from the author upon request.

All calculations subsequent to the data reduction were performed with the SHELX 76 system of programs [4]. The calculations were carried out on the Cyber 76 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale (Bologna), with financial support from the University of Parma.

Results and discussion

Figure 1 shows the structure of the title compound and also gives the atom-numbering scheme; bond lengths and bond angles are shown in Table 4. The overall stereochemistry of the complex is clearly five coordinate, with the tin atom bonded to three phenyl carbon atoms, a chlorine atom, and one of the oxygen atoms of the phosphorus ligand. The five coordinate atoms assume a distorted trigonal-bipyramidal arrangement with the carbon atoms in the equatorial positions, as usually observed in five coordinate triphenyltin compounds [1,3], and with the chlorine atom and the oxygen atom occupying the axial sites. The values of the bond angles at tin indicate a small, but significant, distortion from the idealized trigonal-bipyramidal stereochemistry, with the three in-plane angles (114.6(2), 116.2(3), 126.9(3)°) significantly distorted

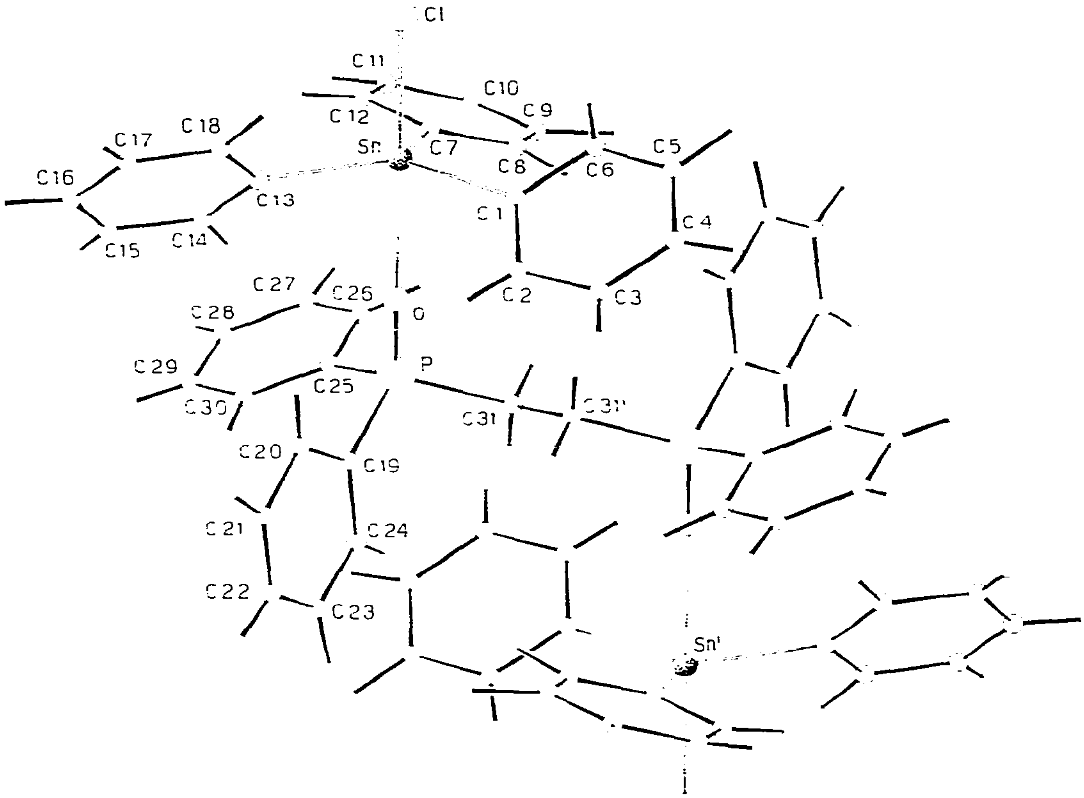

Fig. 1. Projection of the structure.

from a regular 120°, with the axial to axial angle of 174.4(1)° instead of 180°, and with the angles between equatorial and axial sites in the range 83.7—98.1°. The tin atom lies 0.18 Å away from the C_3 equatorial plane towards the chlorine atom. The three phenyl rings are perfectly planar, with tin 0.07—0.12 Å out of the ring planes. The rings are inclined at angles of 44.4° (ring C(1)—C(6)), 16.8° (ring C(7)—C(12)), and 133.0° (ring C(13)—C(18)) to the SnC_3 equatorial plane.

The arrangement of donor atoms of the ligands around tin is identical to that found earlier by us in other triphenyltin adducts, namely $[(SnPh_3NO_3)_2(OPPh_2CH_2)_2]$ [1], $[SnPh_3Cl(OPPh_2CH)_2]$ [2], $[SnPh_3NO_3Ph_3PO]$ [5], $[SnPh_3NO_3Ph_3AsO]$ [6], and $[SnPh_3NO_3(C_5H_5NO)]$ [7,8], all of which have trigonal-bipyramidally coordinated tin atoms with the phenyl rings occupying the equatorial positions. The three Sn—C bonds found here are nearly equivalent at 2.134(5), 2.120(6) and 2.116(6) Å. These distances lie in the middle of the range (2.075—2.159 Å) of lengths observed in the five tin compounds mentioned above.

As illustrated in Fig. 1, the complex has a dinuclear structure, with the phosphorus ligand forming a bridge, through its oxygen atoms, between two tin atoms related by a crystallographic center of symmetry lying in the middle of

TABLE 4

BOND DISTANCES (Å) AND ANGLES (°)

(i) in the coordination polyhedron

Sn—Cl	2.475(2)	Sn—O	2.357(3)	Sn—C(1)	2.134(5)
Sn—C(7)	2.120(6)	Sn—C(13)	2.116(6)		

Cl—Sn—O	174.4(1)	Cl—Sn—C(1)	93.6(2)
Cl—Sn—C(7)	98.1(2)	Cl—Sn—C(13)	93.7(2)
O—Sn—C[1)	83.7(2)	O—Sn—C(7)	87.5(2)
O—Sn—C(13)	84.0(2)	C(1)—Sn—C(7)	114.6(2)
C(1)—Sn—C(13)	126.9(3)	C(7)—Sn—C(13)	116.2(3)

(ii) in the phenyl rings

C(1)—C(2)	1.398(10)	C(7)—C(8)	1.387(12)	C(13)—C(14)	1.375(10)
C(2)—C(3)	1.396(12)	C(8)—C(9)	1.379(13)	C(14)—C(15)	1.425(13)
C(3)—C(4)	1.357(16)	C(9)—C(10)	1.344(20)	C(15)—C(16)	1.346(21)
C(4)—C(5)	1.372(17)	C(10)—C(11)	1.360(23)	C(16)—C(17)	1.343(18)
C(5)—C(6)	1.358(13)	C(11)—C(12)	1.437(13)	C(17)—C(18)	1.397(13)
C(6)—C(1)	1.386(9)	C(12)—C(7)	1.395(12)	C(18)—C(13)	1.390(10)

Sn—C(1)—C(2)	123.1(5)	C(8)—C(9)—C(10)	121.4(12)
Sn—C(1)—C(6)	118.5(5)	C(9)—C(10)—C(11)	119.6(10)
C(2)—C(1)—C(6)	118.4(6)	C(10)—C(11)—C(12)	120.9(12)
C(1)—C(2)—C(3)	119.0(7)	C(11)—C(12)—C(7)	118.5(9)
C(2)—C(3)—C(4)	120.9(9)	Sn—C(13)—C(14)	119.9(5)
C(3)—C(4)—C(5)	120.0(10)	Sn—C(13)—C(18)	121.1(5)
C(4)—C(5)—C(6)	120.3(10)	C(14)—C(13)—C(18)	119.0(6)
C(5)—C(6)—C(1)	121.4(8)	C(13)—C(14)—C(15)	119.1(9)
Sn—C(7)—C(8)	120.4(5)	C(14)—C(15)—C(16)	120.5(10)
Sn—C(7)—C(12)	121.4(6)	C(15)—C(16)—C(17)	120.6(11)
C(8)—C(7)—C(12)	118.1(6)	C(16)—C(17)—C(18)	120.6(11)
C(7)—C(8)—C(9)	121.4(9)	C(17)—C(18—C(13)	120.1(8)

(iii) in the bis(diphenylphosphinyl) ethane molecule

P—O	1.487(3)	C(21)—C(22)	1.356(15)	C(27)—C(28)	1.354(10)
P—C(19)	1.787(6)	C(22)—C(23)	1.339(18)	C(28)—C(29)	1.365(14)
P—C(25)	1.812(5)	C(23)—C(24)	1.394(12)	C(29)—C(30)	1.376(13)
P—C(31)	1.806(5)	C(24)—C(19)	1.387(7)	C(30)—C(25)	1.359(9)
C(19)—C(20)	1.386(9)	C(25)—C(26)	1.380(9)	C(31)—C(31')[a]	1.523(8)
C(20)—C(21)	1.409(15)	C(26)—C(27)	1.364(10)		

O—P—C(19)	110.8(2)	C(21)—C(22)—C(23)	121.0(10)
O—P—C(25)	113.5(2)	C(22)—C(23)—C(24)	121.2(10)
O—P—C(31)	110.7(2)	C(23)—C(24)—C(19)	118.8(8)
C(19)—P—C(25)	107.3(3)	P—C(25)—C(26)	120.6(4)
C(25)—P—C(31)	107.9(2)	P—C(25)—C(30)	120.5(5)
C(19)—P—C(31)	106.4(3)	C(26)—C(25)—C(30)	118.4(6)
Sn—O—P	161.8(2)	C(25)—C(26)—C(27)	120.4(6)
P—C(19)—C(20)	119.2(5)	C(26)—C(27)—C(28)	121.3(7)
P—C(19)—C(24)	120.6(5)	C(27)—C(28)—C(29)	118.6(8)
C(20)—C(19)—C(24)	120.2(6)	C(28)—C(29)—C(30)	120.8(9)
C(19)—C(20)—C(21)	118.6(7)	C(29)—C(30)—C(25)	120.5(8)
C(20)—C(21)—C(22)	120.2(10)	P—C(31)—C(31')[a]	110.9(4)

[a] ' = $\bar{x}, \bar{y}, \bar{z}$.

the C(sp^3)—C(sp^3) bond. A quite analogous situation was recently observed in [$(SnPh_3NO_3)_2(OPPh_2CH_2)_2$], in which the chlorine atom is replaced by the nitrate group. The Sn—O bond distance determined in the present study, 2.357(3) Å, is somewhat longer than that in the nitrate derivative, 2.238(5) Å.

When the vibrational bands of bis(diphenylphosphinyl)ethane in its free state are compared with those in the two tin compounds (Table 5), it is seen that the

TABLE 5

COMPARISON OF THE MAIN VIBRATIONAL BANDS (cm^{-1}) IN $(OPPh_2CH_2)_2$, $[(SnPh_3Cl)_2(OPPh_2CH_2)_2]$, AND $[(SnPh_3NO_3)_2(OPPh_2CH_2)_2]$

$(OPPh_2CH_2)_2$	$[(SnPh_3Cl)_2(OPPh_2CH_2)_2]$	$[(SnPh_3NO_3)_2(OPPh_2CH_2)_2]$	Assignment
3060m	3060m	3070w	$\nu(CH)_{aryl}$
—	3020(sh)	3050w	
2950w	2960w	2960w	$\nu(CH)_{alkyl}$
2920w	2910w	2920w	
1590mw	1590m	1590m	$\nu(CC)$
—	1575(sh)	1580(sh)	
1485m	1480s	1480s	ring
1435s	1435vs	1435m	ring
1420m	1428vs	1430m	
1188vs	1180s	1180m	$\nu(PO) + \beta(CH)$
1175vs	1155vs	1145s	$\nu(PO)$
1120s	1120s	1120m	$\nu(PC)$
725vs	730vs	730vs	$\nu(PC)$
530vs	530vs	530m	$\delta(PC)$
510vs	495(sh)	500m	
450m	450s	455ms	$\nu(PC)$
410m	415m	415w	
	375mw	375w	$\nu(SnO)$
—	—	280m	
—	263s(br)	265m	$\nu(SnC)$

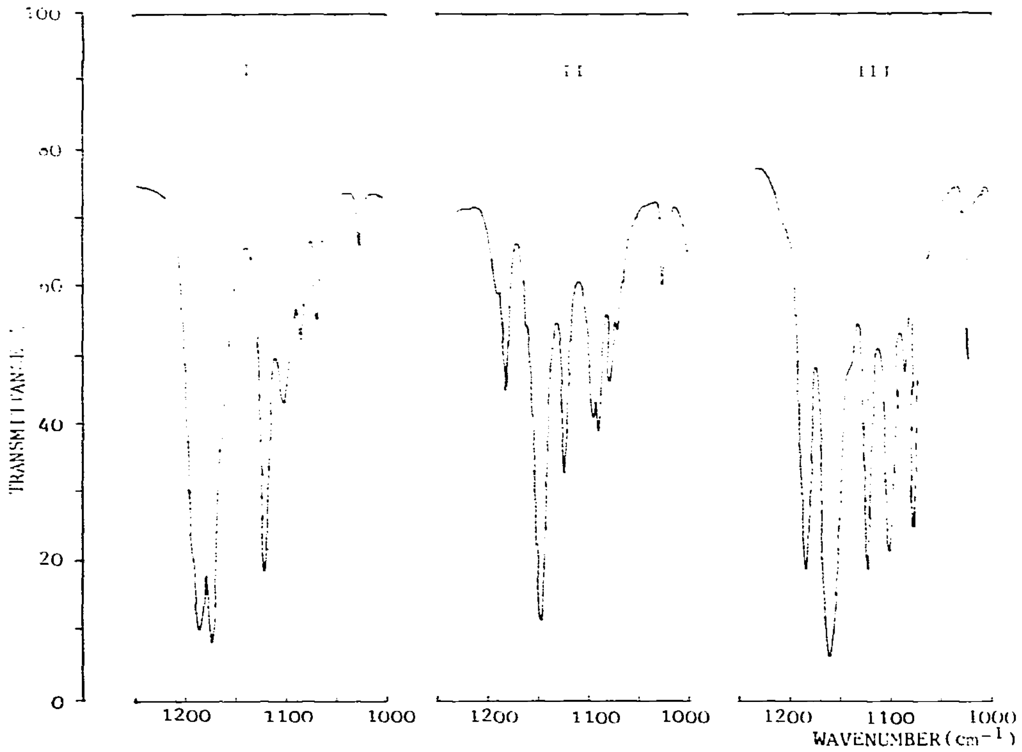

Fig. 2. Vibrational absorptions of the P=O bond in $(OPPh_2CH_2)_2$ (I), in $[SnPh_3NO_3)_2(OPPh_2CH_2)_2]$ (II), and in $[(SnPh_3Cl)_2(OPPh_2CH_2)_2]$ (III).

TABLE 6

SIGNIFICANT PACKING INTERACTIONS (Å)

Cl...C(22) [a]	3.64(1)	Cl...C(5) [b]	3.76(1)
Cl...C(15) [c]	3.83(1)	C(5)...C(6) [b]	3.78(1)
C(16)...C(23) [c]	3.71(2)	C(17)...C(30) [c]	3.64(1)
C(16)...C(11) [d]	3.69(2)	C(4)...C(21) [e]	3.73(1)
C(18)...C(27) [f]	3.75(1)		

[a] $1 + x, y, z$; [b] $1 - x, \bar{y}, 1 - z$; [c] $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; [d] $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$; [e] $\bar{x}, \bar{y}, 1 - z$; [f] $x, y, z + 1$.

IR absorptions associated with the P=O group (Fig. 2) are the only ones influenced by the coordination; the ν(PO) bands shift to lower frequency values compared to those at 1188, 1175 cm^{-1} in the free ligand. The assignment of the 375 cm^{-1} band to ν(Sn—O) [3] is not certain because of the weak intensity of the bond and the presence of a rather similar absorption in the spectrum of the free ligand. Comparison of the tabulated data reveals that no significant differences are noted for the vibrations of the P—C bonds or the phenyl rings.

Other than the compound studied in this work and the nitrate derivative mentioned above, there are only two additional bis(diphenylphosphinyl)ethane (L) complexes, for which the crystal structures have been determined, namely $CuCl_2 \cdot L$ [9] and $CoCl_2 \cdot L$ [10]. The most interesting comparison between these compounds involves the P—O—M linkage, which is distinctly bent to about the same extent in $(SnPh_3NO_3)_2L$, 148.3(8)°, $CuCl_2 \cdot L$, 148.9(1)°, and $CoCl_2 \cdot L$, 138.7(4) and 147.8(4)°, while in the present compound the bending is substantially smaller (161.8(2)°). Other dimensions within the L ligand are normal, with the geometry at the phosphorus atom slightly distorted from tetrahedral. The phosphorus atom is practically coplanar (0.02 Å) with the C(19)—C(24) ring plane, and lies only by a small amount (0.16 Å) out of the C(25)—C(30) ring plane. The two phenyl rings are characterized by different dihedral angles of 13.0° (ring C(19)—C(24)) and 73.9° (ring C(25)—C(30)) with the planes through C(19)PO and C(25)PO, respectively.

The Sn—Cl distance at 2.475(2) Å is short by comparison with those observed for axial bonds in most five coordinate triorganotin compounds, e.g.: 2.572(4), 2.696(3) Å in $[SnMe_3Cl_2]^-$ [11]; 2.565(2) Å in $SnMe_3Cl[Ph_3P{=}CH{=}C(O)Me]$ [12]; 2.573(7), 2.689(6) Å in $[SnBu_3Cl_2]^-$ [13], but agrees well with the 2.42(4) Å in $SnMeCl \cdot C_5H_5N$ [14] and the 2.489(4) Å in $[SnPh_3Cl(OPPh_2CH)_2]$ [2].

The molecular packing is determined by normal Van der Waals interactions, as indicated by Table 6, which shows the most significant packing contacts.

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