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ORGANOTIN(IV) CHLORIDE COMPLEXES OF BIS-β-KETOPHOSPHONIUM YLIDES

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Summary

The coordination behaviour of bis-β-ketophosphonium ylides, ($Ph_3\overset{+}{P}-\bar{C}RCO(CH_2)_nCO\bar{C}R-\overset{+}{P}Ph_3$) towards some organotin(IV) chlorides has been studied. Accordingly, organotin(IV) chloride complexes of the general formula ($Ph_3\overset{+}{P}-\bar{C}RCO(CH_2)_nCO\bar{C}R-\overset{+}{P}Ph_3$)$R'_xSnCl_{4-x}$ ($n = 0$ or 2; $x = 1$–3) have been synthesized and characterized by analysis and IR, ^1H, ^{13}C, ^{31}P, ^{119}Sn NMR and Mössbauer spectroscopy. The complexes are 1 : 1 adducts, containing weak carbon–tin bonds and are non-electrolytes and monomeric in solution.

Introduction

The valuable biological properties of organotin compounds [2,3] have prompted studies of a wide variety of organotin compounds. Several studies have been made on the coordination ability of ylides, particularly those of phosphorus and sulfur, towards different metals [4], but there are only scanty reports on organotin complexes of the ylides [5–10]. In the first report [5], in 1961, a quaternary phosphonium salt type structure was suggested for the triphenylphosphonium methylide complexes of organotin(IV) chlorides. Later Harrison et al. [6,7] on the basis of IR and Mössbauer spectroscopic data suggested the coordination of ylide to the metal was through the carbonyl oxygen in the case of triorganotin chlorides and this was later confirmed by X-ray studies [6]. However, diorganotin halides form complex structures of various stoichiometries containing carbon–metal bonds [7]. Almost at the same time Kato and coworkers [8] reported the trialkyltin chloride complex of triphenylphosphoniumphenacylide, and suggested an ionic structure for the complex on the basis of elemental analysis and IR studies, but the mode of coordination could not be unambiguously assigned. These conclusions were later justifiably questioned by Brumeister et al. [9], who on the basis of the decrease in the ν(C=O) frequency on complexation of the ylide with triorganotin chloride, such as had been reported by earlier workers, suggested neutral coordination of ylides with tin through the oxygen atom.

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In 1982, Yamamoto [10] reported the preparation of Group IVA metals (Ge, Sn, Pb) complexes with alkylidenetriphenylphosphoranes and suggested the formation of carbon–metal bonds.

In view of these results, we decided to study the coordination behaviour of bis-β-ketophosphonium ylides towards some organotin(IV) chlorides. All the ylides form 1 : 1 non-electrolyte monomeric complexes. The structures and mode of coordination are discussed in the light of IR, ^{1}H, ^{13}C, ^{31}P, ^{119}Sn NMR and Mössbauer data.

Experimental

Preparation of starting materials

The bis-β-ketophosphonium ylides were prepared by transylidation of phosphonium alkylides with the appropriate thioesters. The synthesis of the ylides has been given earlier [1].

Preparation of organotin(IV) chloride complexes of the ylides

The following general method was used for the synthesis of all the complexes.

To the chloroform solution of the ylide (1 equiv.) the relevant organotin chloride (1 equiv.) was added. The reaction mixture was refluxed for about 10 h. In some cases, pale yellow complexes separated, and these were found to be insoluble in common organic solvents. In other cases the reaction mixture was concentrated to about 10 ml and a small amount of petroleum ether was added; an oil separated, and this was washed with ether and dried under vacuum to yield an amorphous powder.

Physical measurements

Solid state IR spectra in the range of 4000–200 cm^{-1} were recorded on a Perkin–Elmer model 577 grating spectrophotometer as Nujol mulls in CsI cells. The PMR spectra were usually recorded with $CDCl_3$ solutions on a Perkin–Elmer 12B spectrometer at 60 MHz, but some were recorded on a Bruker WM 400 (400 MHz) spectrometer with TMS as external standard. The ^{13}C, ^{31}P and ^{119}Sn NMR spectra were recorded on FX90Q JEOL spectrometer using external standards. The molecular weights were determined ebullioscopically on Gallenkamp ebulliometer and molar conductances were measured on a conductivity meter type bridge 302 Sr. No. 417. Melting points are uncorrected.

Results

Reaction of ylides (Y) with organotin(IV) chlorides (R'_xSnCl_{4-x}) in the appropriate solvent ($CHCl_3$ or $CHCl_3/CH_3OH$ mixture) under reflux gave the complexes (YR'_xSnCl_{4-x}).

$$Y + R'_xSnCl_{4-x} \rightarrow Y.R'_xSnCl_{4-x}$$

The complexes were obtained in 60–94% yields as light-coloured solids. All of them are stable in air, and most are soluble in common organic solvents, the exceptions being those derived from triphenyltin chloride and phenyltin trichloride.

The physical, analytical and spectral data of the complexes are given in Tables 1–5. Metal and chlorine analyses show the compounds to be 1 : 1 adducts. The molar conductance values (0.0–0.9 ohm^{-1} cm^{-2}) and molecular weights determination show them to be non-electrolytes and monomeric in chloroform solution.

Discussion

β-Ketoylides and their complexes usually exhibit two absorption bands in the carbonyl stretching region owing to partial delocalization of ylidic charge on to the carbonyl oxygen, but in some cases these two bands are not well resolved, and a broad band is observed. The complexes prepared during the present investigations have similar spectra in the carbonyl stretching region.

The shift in the ν(C=O) stretching frequency has been used as the main criterion in the previous studies [5–9] for elucidating the mode of coordination of ylides in their organotin(IV) complexes. The coordination of ylidic ligands through carbonyl oxygen (=C–O–Sn) should cause a substantial lowering in ν(C=O) stretching frequency. In the present studies there is instead, a blue shift (ca. 5–35 cm^{-1}) in all the complexes, except for those involving the $Ph_3\overset{+}{P}-\overline{C}(COOCH_3)COCO\overline{C}(COOCH_3)-\overset{+}{P}Ph_3$ ylide. These observations indicate that the coordination of the ylides in these complexes is not through carbonyl oxygen. The observed higher frequency shift can be explained on the basis of coordination through ylidic carbon and this is corroborated by ^{1}H and ^{13}C NMR spectral studies of some of the complexes.

The complexes formed by the ylide $Ph_3\overset{+}{P}-\overline{C}(COOCH_3)COCO(COOCH_3)\overline{C}-\overset{+}{P}Ph_3$ do not show any significant change in the carbonyl stretching vibrations. It appears that the presence of bulky carbomethoxy group on the ylidic carbon hinders coordination to the metal. But as there is no red shift either, it is difficult to decide the mode of coordination in these complexes on the basis of IR studies. A greater blue shift in ν(C=O) stretching vibrations of diorganotin dichloride and monoorganotin trichloride complexes is attributable to the stronger electrophilic character of tin in these species, resulting in the formation of stronger carbon–metal bonds. The appearance of two Sn–Cl stretching vibrations in diorganotin complexes in the range of 275 to 380 cm^{-1} suggests a *cis* arrangement of halogens around tin [11]. The triorganotin chloride complexes each show a single Sn–Cl stretching vibration in the range of 260–360 cm^{-1}.

A low field shift in the methine proton resonance indicates that there is coordination of the ylide moieties to the metal [10,11]. In the present studies, the methine proton shows a doublet in the range of δ 4.5–6.2 ppm (^2J(P–C–H) 17–20 Hz) which is considerably downfield to that for the ylides. A decrease in the coupling between phosphorus and methine proton in complexes compared to that in the ylide provides additional evidence for coordination of the ylide to the metal. In the present investigation we observed a decrease of about 6–9 Hz in the coupling constant which is due to the increase of P–C bond length on coordination. The higher value of the ^{119}Sn–CH$_3$ coupling constant (64–88 Hz) for trimethyltin chloride complexes (I, IX, XII) than trimethyltin chloride itself reveals pentacoordination [12,13] of tin. The protons of the Sn–CH$_3$ groups give a singlet in all the complexes, indicating that all the methyl protons in these complexes are chemically equivalent. In the case of trimethyltin chloride complexes, this is possible only when all three methyl groups occupy the equatorial position in a trigonal bipyramidal geometry. This suggests that there is pentacoordination of tin in the triorganotin chloride complexes.

^{13}C NMR spectroscopy has also been employed previously to study the coordination behaviour of ylides in their complexes [14]. It was found that when coordination

TABLE 1

PHYSICAL AND ANALYTICAL DATA FOR THE COMPLEXES

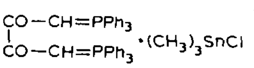
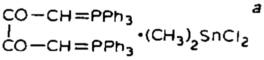
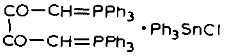
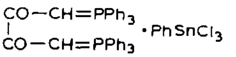
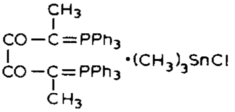
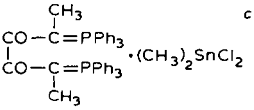
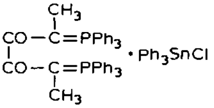
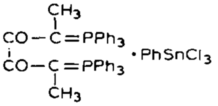
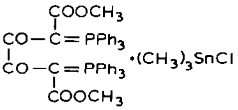
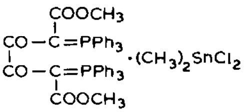
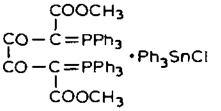
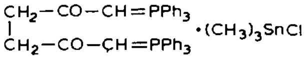
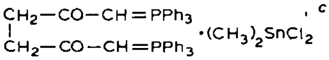
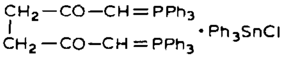
Compound	M.p. (°C)	Yield (%)	Analyses (Found (calcd.)(%))		MW (Found (calcd.))
			Metal	Halogen	
 $\begin{array}{l}\mathrm{CO-CH=PPh_3}\\ \mathrm{CO-CH=PPh_3}\end{array}\cdot\mathrm{(CH_3)_3SnCl}$ [a] (I)	142	96	14.88 (14.78)	4.26 (4.40)	824 (805.5)
 $\begin{array}{l}\mathrm{CO-CH=PPh_3}\\ \mathrm{CO-CH=PPh_3}\end{array}\cdot\mathrm{(CH_3)_2SnCl_2}$ [a] (II)	149	89	14.29 (14.40)	8.38 (8.59)	833 (826)
 $\begin{array}{l}\mathrm{CO-CH=PPh_3}\\ \mathrm{CO-CH=PPh_3}\end{array}\cdot\mathrm{Ph_3SnCl}$ [a] (III)	139	94	11.86 (12.02)	3.53 (3.58)	Insoluble
 $\begin{array}{l}\mathrm{CO-CH=PPh_3}\\ \mathrm{CO-CH=PPh_3}\end{array}\cdot\mathrm{PhSnCl_3}$ [b] (IV)	185	93	12.97 (13.09)	11.3 (11.7)	Insoluble
 $\begin{array}{l}\qquad\mathrm{CH_3}\\ \mathrm{CO-C=PPh_3}\\ \mathrm{CO-C=PPh_3}\\ \qquad\mathrm{CH_3}\end{array}\cdot\mathrm{(CH_3)_3SnCl}$ [c] (V)	192	87	14.09 (14.27)	4.16 (4.26)	Solubility poor
 $\begin{array}{l}\qquad\mathrm{CH_3}\\ \mathrm{CO-C=PPh_3}\\ \mathrm{CO-C=PPh_3}\\ \qquad\mathrm{CH_3}\end{array}\cdot\mathrm{(CH_3)_2SnCl_2}$ [c] (VI)	178	92	13.81 (13.93)	8.33 (8.31)	830 (854)
 $\begin{array}{l}\qquad\mathrm{CH_3}\\ \mathrm{CO-C=PPh_3}\\ \mathrm{CO-C=PPh_3}\\ \qquad\mathrm{CH_3}\end{array}\cdot\mathrm{Ph_3SnCl}$ [a] (VII)	175	92	11.60 (11.72)	3.37 (3.48)	Poor solubility
 $\begin{array}{l}\qquad\mathrm{CH_3}\\ \mathrm{CO-C=PPh_3}\\ \mathrm{CO-C=PPh_3}\\ \qquad\mathrm{CH_3}\end{array}\cdot\mathrm{PhSnCl_3}$ [c] (VIII)	240	80	12.34 (12.70)	11.77 (11.37)	Insoluble
 $\begin{array}{l}\qquad\mathrm{COOCH_3}\\ \mathrm{CO-C=PPh_3}\\ \mathrm{CO-C=PPh_3}\\ \qquad\mathrm{COOCH_3}\end{array}\cdot\mathrm{(CH_3)_3SnCl}$ [a] (IX)	171	97	12.83 (12.92)	3.90 (3.85)	937 (922)

TABLE 1 (continued)

Compound	M.p. (°C)	Yield (%)	Analyses (Found (calcd.)(%))		MW (Found (calcd.))
			Metal	Halogen	
 $\begin{array}{l}\mathrm{COOCH_3}\\ \mathrm{CO{-}C{=}PPh_3}\\ \mathrm{CO{-}C{=}PPh_3}\\ \mathrm{COOCH_3}\end{array}\cdot(\mathrm{CH_3})_2\mathrm{SnCl_2}$ [a] (X)	159	73	12.51 (12.63)	7.62 (7.53)	934 (942)
 $\begin{array}{l}\mathrm{COOCH_3}\\ \mathrm{CO{-}C{=}PPh_3}\\ \mathrm{CO{-}C{=}PPh_3}\\ \mathrm{COOCH_3}\end{array}\cdot\mathrm{Ph_3SnCl}$ [d] (XI)	148	82	10.60 (10.74)	3.17 (3.20)	1086 (1107)
 $\begin{array}{l}\mathrm{CH_2{-}CO{-}CH{=}PPh_3}\\ \mathrm{CH_2{-}CO{-}CH{=}PPh_3}\end{array}\cdot(\mathrm{CH_3})_3\mathrm{SnCl}$ [b] (XII)	96	50	13.88 (14.24)	4.41 (4.26)	939 (933.5)
 $\begin{array}{l}\mathrm{CH_2{-}CO{-}CH{=}PPh_3}\\ \mathrm{CH_2{-}CO{-}CH{=}PPh_3}\end{array}\cdot(\mathrm{CH_3})_2\mathrm{SnCl_2}$ [c] (XIII)	85	91	13.80 (13.93)	8.70 (8.31)	842 (854)
 $\begin{array}{l}\mathrm{CH_2{-}CO{-}CH{=}PPh_3}\\ \mathrm{CH_2{-}CO{-}CH{=}PPh_3}\end{array}\cdot\mathrm{Ph_3SnCl}$ [c] (XIV)	167	77	11.54 (11.70)	3.6 (3.5)	Insoluble

[a] Pale yellow. [b] Yellow. [c] Cream. [d] White.

occurs through ylidic carbon, this carbon tends to become sp^3-hybridized and there is an upfield shift in its resonance. On the other hand, the carbon of the β-carbonyl group of the ylide shows a downfield shift because of electron-withdrawal from it [14]. The ^{13}C NMR spectra of two of the ylides and four complexes (I, II, XII, XIII, Table 3) from them were recorded, and revealed some interesting differences in the nature of two types of complexes. The ylidic carbon of trimethyltin chloride complexes (I, XII) unexpectedly shifts downfield by about 9–11 ppm (J(P–C) 99–100 Hz), indicating that the hybridization state of coordinated ylidic carbon does not fully change to sp^3, but that is deshielded because of the electron-withdrawal. The carbonyl group shows a downfield shift of about 7–16 ppm indicating that there is an electron withdrawal from the ylide molecule. It appears that only one ylidic carbon of the bis-ylide is coordinated to tin thereby giving rise to pentacoordinated tin species. However, the appearance of only one signal for the ylidic carbon as well as for the methine protons (Tables 2 and 3) in these complexes indicates that there is a fluxional type of coordination between the ylidic carbons and tin, i.e., there is fast interchange of the tin moiety between the two ylidic carbons. This will not allow to change the hybridization state of the ylidic carbons but should simply make them a little more deshielded. For an effective fluxional mechanism, the ylide could be expected to acquire a type of conformation which brings the two ylidic carbons close enough to facilitate the oscillation of tin between them. The fluxional type of coordination has been supported by scanning PMR spectra of a representative

TABLE 2

IR (cm^{-1}) AND ^1H NMR DATA (δ (ppm), J(Hz)) FOR THE SOLUBLE COMPLEXES

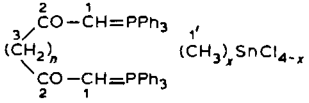
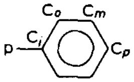
Compound	IR		^1H NMR
	ν(C=O)	ν(M–Cl)	
I	1535 1505	340	0.7 (s, 9H, CH_3, $^2J(^{119}Sn-C-H)$ 64, $^2J(^{117}Sn-C-H)$ 62), 6.2 (d, 2H, CH, $^2J(P-C-H)$ 20, 7.3–7.8 (m, 30H, Ar)
II	1540 1515	290 390	0.3 (s, 6H, CH_3, $^2J(^{119}Sn-C-H)$ 80), 6.1 (d, 2H, CH, $^2J(P-C-H)$ 19
III	1535br	340	
IV	1545br	300 325	
V	1520br	[a]	
VI	1520br	275 335	1.3 (s, 6H, CH_3, $^2J(^{119}Sn-C-H)$ 144), 1.9 (d, 6H, CH_3, $^3J(P-C-H)$ 17), 7.4–7.8 (m, 30H, Ar)
VII	1520br	260	
VIII	1520br	270 335	
IX	1720 1660 1620	 310	1.1 (s, 9H, CH_3, $^2J(^{119}Sn-C-H)$ 88), 3.8 (s, 6H, OCH_3), 7.4–8.4 (m, 30H, Ar)
X	1720 1660 1620	340 380	1.2 (s, 6H, CH_3, $^2J(^{119}Sn-C-H)$ 84), $^2J(^{117}Sn-C-H)$ 80), 3.75 (s, 6H, OCH_3), 7.5–8.1 (m, 30H, Ar)
XI	1720 1660 1620	360	3.6 (s, 6H, OCH_3), 7.2–8.0 (m, 30H, Ar)
XII	1550 1520	350	0.64 (s, 9H, CH_3, $^2J(^{119}Sn-C-H)$ 64, $^2J(^{117}Sn-C-H)$ 62), 2.4 (b, 4H, CH_2) 4.5 (b, 2H, CH), 7.4–7.8 (m, 30H, Ar)
XIII	1700 1610	290 310	1.2 (s, 6H, CH_3, $^2J(^{119}Sn-C-H)$ 90), 2.5 (b, 4H, CH_2), 5.9 (d, 2H, CH, $^2J(P-C-H)$ 17), 7.4–7.8 (m, 30H, Ar)
XIV	1540br	290	

[a] Not recorded.

complex (No. 1 in Table 1) at low temperatures. We observed that at −30°C, an additional doublet corresponding to the methine protons appears at δ 5.6 ppm ($^2J(P-C-H)$ 24 Hz). On the basis of these results, the following two structures (Fig. 1) seem plausible for the complexes formed by trimethyltin chloride; the fluxional types of bonds have been shown by broken lines. It is obvious that the isomer having both the phosphonium moieties in equatorial positions (**A**) will be more stable than the other isomer (**B**) which has one phosphonium moiety in equatorial and the other in axial position. This is in accord with the relative intensities (2/1/1) of the three signals in the ^{31}P NMR spectra. The most intense signal observed at δ 24.5–28.6 ppm correspond to the phosphorus atoms of the two equatorial phos-

TABLE 3

^{13}C NMR DATA (δ (ppm)) FOR COMPLEXES I, II, XII AND XIII AND THE FREE LIGANDS [a]

Compound									$J(^{119}Sn-^{13}C)$ (Hz)
	C(1)	C(2)	C(3)	C(1')	C_i	C_o	C_m	C_p	
Ligand Y_1	54.5	176.0	-		124.9	132.8	128.4	133.8	
($n = 0$)	(105)				(91)	(8)	(12)	(0)	
I	65.1	195.0	-	6.5	122.7	133.3	129.9	132.1	394
	(99)				(88)	(11)	(12)	(0)	
II	[b]	192.0	-	3.8	120.4	133.2	130.6	135.4	535
					(82)	(9)	(12)	(0)	
Ligand Y_2	50.8	193.0	34.4		127.3	132.9	128.6	131.7	
($n = 2$)	(107)		(14)		(89)	(10)	(11)	(0)	
XII	59.0	197.0	43.0	14.1	142.0	147.5	144.0	148.0	[b]
	(100)				(100)	(10)	(10)	(0)	
XIII	28.6	207.0	58.2	18.4	129.4	130.7	134.0	135.6	[b]
	(24)		(0)		(89)	(13)	(11)	(0)	

[a] The values of J(P–C) are given in parentheses. [b] Not observed.

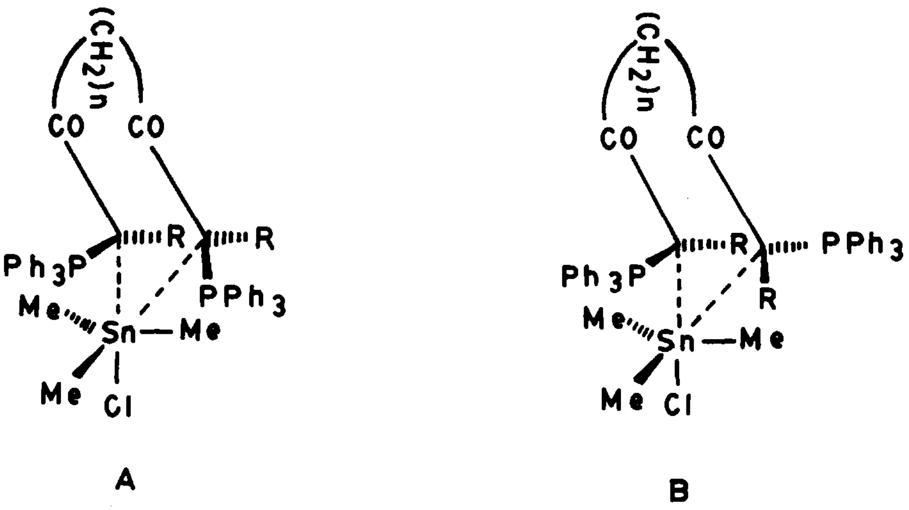


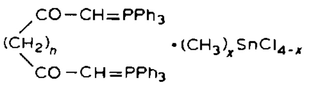
Fig. 1. Structures of the complexes of bis-β-ketophosphonium ylides with trimethyltin chloride.

phonium moieties of the isomer **A**, while the other two signals at δ 20.3–20.5 and 11.5–15.5 ppm, each one of which has almost half the intensity of the previous signal, result from the isomer **B**.

Pentacoordination in the trimethyltin chloride complexes is further supported by the ^{119}Sn NMR spectra of the complexes I and XII when ^{119}Sn signals lie well within

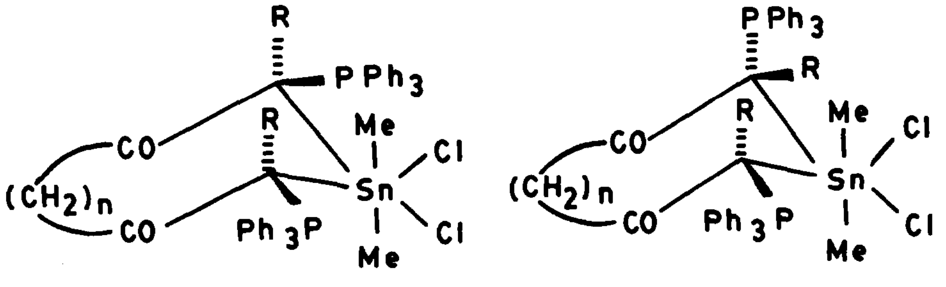
TABLE 4

^{31}P AND ^{119}Sn NMR DATA (δ(ppm)) FOR COMPLEXES I, II, XII AND XIII AND THE FREE LIGANDS



$$(CH_2)_n\begin{cases}CO-CH{=}PPh_3\\CO-CH{=}PPh_3\end{cases}\cdot(CH_3)_xSnCl_{4-x}$$

Compound	δ(^{31}P)	δ(^{119}Sn)
Ligand Y_1 ($n = 0$)	15.4	
I	15.5 20.3 24.5	55.3
II	20.2 29.5	−6.7
Ligand Y_2 ($n = 2$)	11.7	
XII	11.1 20.5 28.6	22.1
XIII	21.2 29.7	−8.4



C

D

Fig. 2. Structures of the complexes of bis-β-ketophosphonium ylides with dimethyltin dichloride.

the range for pentacoordinated tin [17]. Further, it was possible to observe coupling between ^{13}C–^{119}Sn in the case of complex I, and the value of the coupling constant (394 Hz) also indicates pentacoordination [18].

In the dimethyltin dichloride complexes II, VI and XIII, the ^{119}Sn–C–H coupling constant values (80–144 Hz) in PMR lie in the range associated with hexacoordination [12,13], and the latter is indicated also by the substantial upfield shift of ^{13}C resonance of the ylidic carbon on coordination, indicating a change of hybridization to sp^3 (Table 3). Hexacoordination of tin in these complexes is also indicated by the value of ^{13}C–^{119}Sn coupling constant (535 Hz) observed for complex II. Besides this, a single ^{119}Sn resonance (Table 4) is observed in each of the two complexes (II, XIII) at higher field than is the case for dimethyltin dichloride and pentacoordinated complexes. These complexes show two ^{31}P signals in the range of δ 20–30 ppm (Table 4), confirming the coordination of both the ylidic carbons and also indicating the presence of two isomers (**C** and **D**), as shown in Fig. 2.

Mössbauer spectroscopy has been used extensively for studying and elucidating the structure of tin complexes [19], and so the Mössbauer spectra of a few of the present complexes (I–III and V) were recorded. The quadrupole splitting and isomer shift values are in the range of 3.31–3.46 and 1.35–1.45 mm s^{-1} (Table 5), respectively, in the triorganotin chloride complexes (I, III and V) supporting pentacoordination of tin in these complexes. The quadrupole splitting and isomer shift value for dimethyltin dichloride complex II are 3.08 and 1.27 mm s^{-1}, respectively, indicating octahedral geometry for the dimethyltin dichloride complexes with chlorines in *cis* positions.

TABLE 5

^{119}Sn MÖSSBAUER DATA FOR COMPLEXES I, II, III AND V

Compound	*IS* (mm s^{-1})	*QS* (mm s^{-1})
I	1.45	3.46
II	1.27	3.08
III	1.35	3.31
V	1.38	3.43

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