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Synthesis and Characterization of O-Alkyl, O-Cycloalkyl and O-Aryl Trithiophosphato Derivatives of Trimethyl Tin(IV) Chloride and Tributyl Tin(IV) Chloride

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Trimethyltin(IV)trithiophosphates $[(RO)P(S)S_2][Sn(Me)_3]_2$ and *tributyl tin(IV)trithiophosphates* $[(RO)P(S)S_2][Sn(Bu)_3]_2$ ($R=CH_3$, $-C_2H_5$, $-C_3H_7$, $-C_3H_7^{iso}$, $-C_4H_9$, $-C_4H_9^{iso}$, $-C_4H_5^{sc}$, $-C_5H_{11}^{iso}$, C_6H_{11} , $-C_6H_5$) were prepared in methanolic solution of R_3SnCl and dipotassium salt of trithiophosphates in 2:1 molar ratio. These newly synthesised derivatives have been characterized by elemental analysis, molecular weight measurements, IR, ^{13}C , ^{31}P , and ^{119}Sn , NMR spectral studies. Coordination number of four suggested for tin(IV) in these derivatives.

Keywords Phosphorotrithioate; tin(IV)

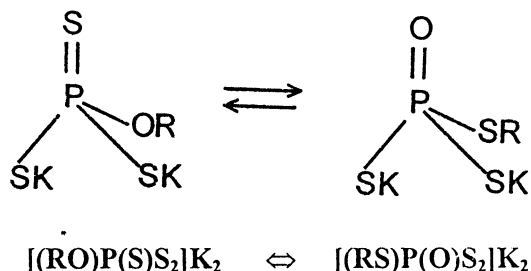
INTRODUCTION

In the recent years considerable interest has been evinced in the chemistry of metallic moieties bonded with sulfur ligands such as thiolates, dithiolates,¹ thio β -diketonates,² dithiocarbamates and O,O'-alkylene dithiophosphates.^{3–5} Some mono di- and tri-organo tin(IV) derivatives of dialkyl dithiophosphates have been synthesised and characterized by ^{119}Sn , ^{13}C , ^{31}P , and ^{119}Sn , NMR and mossbauer spectral studies. The ^{119}Sn , NMR chemical shifts and $[^1J(^{119}Sn-^{13}C)]$ and $[^2J(^{119}Sn-^1H)]$ of triorgano tin(IV) dialkyl dithiophosphates are consistent with tetrahedral geometry and four coordinated Sn, although mossbauer study indicates five coordinated tin in solid state. These studies also reveal that in mono and diorgano tin(IV) dithiophosphates Sn atom is weakly coordinated to ligand.^{6–7} Organic trithiophosphates esters have been

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**FIGURE 1**

used as defoliants,⁸ insecticides,⁸⁻⁹ nematodicidal,⁹ and inhibitors¹⁰ of steel corrosion. Potassium trithiophosphates exist in two isomeric form.

The perusal of literature revealed only two publications on the metallic ester of trithiophosphoric acids.^{11,12} Trithiophosphates of tin element have received no attention to best of our knowledge. Hence it was thought worthwhile to study the trithiophosphates of tri-methyl tin(IV) and tributyl tin(IV). The main bonding pattern of trithiophosphates ligands with metal are shown in Figure 2.

EXPERIMENTAL

Dipotassium salt of O-alkyl, O-cycloalkyl, and O-aryl trithiophosphates were prepared by reaction of the requisite anhydrous alcohol with P_2S_5 and triethylamine in 1:3:3 molar ratio respectively in anhydrous benzene. The reaction mixture was stirred for 1/2 an hour on water bath.

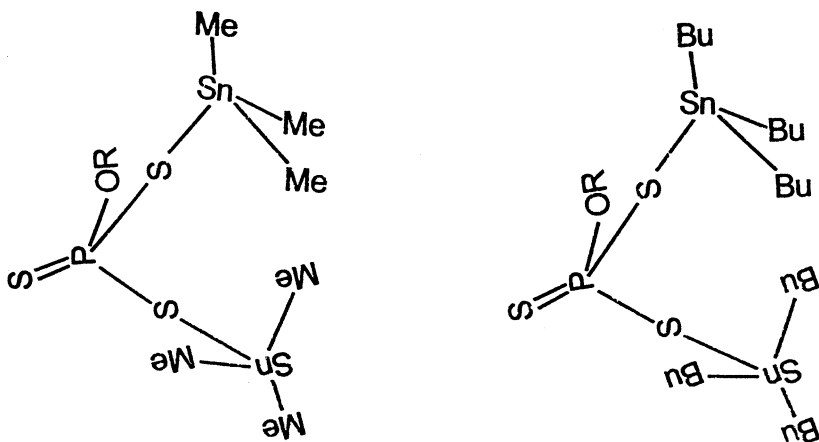
**FIGURE 2**

TABLE I Synthetic, Physical, and Analytical Data for Trimethyltin(IV) Trithiophosphates

S. no.	Reactants (in g.)		Molar ratio	Product (in g.)	Yield (%)	Mol. Wt. Found/(calcd.)	Analysis (%) Found/(Calcd.)			
	Me ₃ SnCl	[(RO)P(S)S ₂]K ₂					C	H	S	Sn
1.	2.14	[MeO)P(S)S ₂]K ₂ 1.26	2:1	[(MeO)P(S)S ₂][Me ₃ Sn] ₂ 2.45	95	482.67 (485.77)	17.12 (17.30)	4.18 (4.35)	19.59 (19.80)	48.62 (48.86)
2.	2.14	[(EtO)P(S)S ₂]K ₂ 1.34	2:1	[(EtO)P(S)S ₂][Me ₃ Sn] ₂ 2.40	90	497.35 (499.80)	19.09 (19.22)	4.40 (4.63)	19.14 (19.24)	47.24 (47.49)
3.	2.14	[(Pr ⁿ O)P(S)S ₂]K ₂ 1.42	2:1	[(Pr ⁿ O)P(S)S ₂][Me ₃ Sn] ₂ 2.58	94	511.53 (513.83)	20.84 (21.03)	4.76 (4.90)	18.56 (18.72)	46.02 (46.19)
4.	2.14	[(Pr ⁱ O)P(S)S ₂]K ₂ 1.42	2:1	[(Pr ⁱ O)P(S)S ₂][Me ₃ Sn] ₂ 2.42	95	511.30 (513.83)	20.82 (21.03)	4.78 (4.90)	18.54 (18.72)	46.02 (46.19)
5.	2.14	[(Bu ⁿ O)P(S)S ₂]K ₂ 1.49	2:1	[(Bu ⁿ O)P(S)S ₂][Me ₃ Sn] ₂ 2.53	85	524.88 (527.85)	20.86 (22.75)	5.02 (5.15)	18.02 (18.22)	44.72 (44.97)
6.	2.14	[(Bu ^s O)P(S)S ₂]K ₂ 1.49	2:1	[(Bu ^s O)P(S)S ₂][Me ₃ Sn] ₂ 2.50	87	524.92 (527.85)	22.48 (22.75)	4.98 (5.15)	18.14 (18.22)	44.68 (44.97)
7.	2.14	[(Bu ^t O)P(S)S ₂]K ₂ 1.49	2:1	[(Bu ^t O)P(S)S ₂][Me ₃ Sn] ₂ 2.65	92	524.84 (527.85)	22.40 (22.75)	4.96 (5.15)	18.10 (18.22)	44.78 (44.97)
8.	2.14	[(Am ⁱ O)P(S)S ₂]K ₂ 1.57	2:1	[(Am ⁱ O)P(S)S ₂][Me ₃ Sn] ₂ 2.75	90	539.13 (541.81)	22.46 (24.38)	5.16 (5.39)	17.56 (17.75)	43.58 (43.81)
9.	2.14	[(C.h.O)P(S)S ₂]K ₂ 1.63	2:1	[(C.h.O)P(S)S ₂][Me ₃ Sn] ₂ 2.66	87	551.49 (553.89)	25.88 (26.02)	5.04 (5.27)	17.18 (17.36)	42.62 (42.83)
10.	2.14	[(Ph.O)P(S)S ₂]K ₂ 1.60	2:1	[(Ph.O)P(S)S ₂][Me ₃ Sn] ₂ 2.49	85	545.75 (547.84)	26.16 (26.30)	4.02 (4.23)	17.36 (17.55)	43.12 (43.32)

TABLE II Synthetic, Physical, and Analytical Data for Tributyltin(IV) Trithiophosphates

S. no.	Reactants (in g.)		Molar ratio	Product (in g.)	Yield (%)	Mol. Wt. Found/(Calcd.)	Analysis (%) Found/(Calcd.)			
	Bu ₃ SnC	[(RO)P(S) ₂] ₂ K ₂					C	H	S	Sn
1.	2.16	[MeO)P(S) ₂] ₂ K ₂ 1.17	2:1	[(MeO)P(S) ₂] ₂ [Bu ₃ Sn] ₂ 2.45	95	520.05 (522.06)	57.32 (57.51)	10.78 (11.00)	18.28 (18.42)	22.52 (22.73)
2.	2.16	[(EtO)P(S) ₂] ₂ K ₂	2:1	[(EtO)P(S) ₂] ₂ [Bu ₃ Sn] ₂	90	533.73 (536.08)	58.10 (58.25)	10.86 (11.09)	17.72 (17.94)	21.90 (22.14)
3.	2.16	[(Pr ⁿ O)P(S) ₂] ₂ K ₂ 1.31	2:1	[(Pr ⁿ O)P(S) ₂] ₂ [Bu ₃ Sn] ₂ 2.55	94	548.41 (550.11)	58.68 (58.95)	10.88 (11.17)	17.28 (17.48)	21.33 (21.57)
4.	2.16	[Pr ⁱ O)P(S) ₂] ₂ K ₂ 1.31	2:1	[Pr ⁱ O)P(S) ₂] ₂ [Bu ₃ Sn] ₂ 2.58	88	548.81 (550.11)	58.72 (58.95)	10.84 (11.17)	17.32 (17.48)	21.35 (21.57)
5.	2.16	[Bu ⁿ O)P(S) ₂] ₂ K ₂	2:1	[Bu ⁿ O)P(S) ₂] ₂ [Bu ₃ Sn] ₂	90	562.90 (564.14)	58.46 (59.61)	11.02 (11.24)	16.92 (17.05)	20.86 (21.03)
6.	2.16	[(Bu ^s O)P(S) ₂] ₂ K ₂	2:1	[(Bu ^s O)P(S) ₂] ₂ [Bu ₃ Sn] ₂	89	561.98 (564.14)	58.48 (59.61)	11.06 (11.24)	16.88 (17.05)	20.92 (21.03)
7.	2.16	[(Bu ^t O)P(S) ₂] ₂ K ₂ 1.38	2:1	[(Bu ^t O)P(S) ₂] ₂ [Bu ₃ Sn] ₂ 2.56	95	561.88 (564.14)	58.51 (59.61)	11.08 (11.24)	16.86 (17.05)	20.94 (21.03)
8.	2.16	[(Am ⁱ O)P(S) ₂] ₂ K ₂ 1.45	2:1	[(Am ⁱ O)P(S) ₂] ₂ [Bu ₃ Sn] ₂ 2.57	90	575.57 (578.17)	60.08 (60.25)	11.18 (11.38)	16.42 (16.64)	20.36 (20.53)
9.	2.16	[(C.h.O)P(S) ₂] ₂ K ₂ 1.51	2:1	[(C.h.O)P(S) ₂] ₂ [Bu ₃ Sn] ₂ 2.54	85	588.87 (590.17)	60.92 (61.05)	10.92 (11.10)	16.12 (16.29)	19.86 (20.11)
10.	2.16	[(Ph.O)P(S) ₂] ₂ K ₂ 1.48	2:1	[(Ph.O)P(S) ₂] ₂ [Bu ₃ Sn] ₂ 2.45	90	582.02 (584.14)	61.42 (61.68)	10.94 (10.18)	16.32 (16.46)	20.18 (20.31)

After stirring salt was precipitated out. All chemicals were of A.R. grade and were used after drying process. The derivatives described in the present paper were synthesised by the following general routes.

SYNTHESIS OF $[(RO)P(S)S_2][Sn(R)_3]_2$

A methanolic (2 mL) solution of anhydrous trimethyl tin(IV) chloride and methanolic solution of $[(RO)P(S)S_2]K_2$ were mixed and refluxed for 8 hrs. The reaction were carried out in 2:1 molar ratio, after refluxing the solid were precipitated. Insolubles were filtered off and the product was obtained from the filtrate by removal of volatiles under reduced pressure. The complex number 1–20 were prepared by this same procedure (where $R' = -CH_3, -C_2H_5, -C_3H_7^n, -C_3H_7^{iso}, -C_4H_9^n, -C_4H_9^{sec}, -C_4H_9^{iso}, -C_5H_{11}^{iso}, -C_6H_{11}, -C_6H_5$ and $R = \text{methyl and butyl}$).

MEASUREMENTS

IR spectra were recorded in nujol mulls using Csl cells on a Perkin Elmer 577 spectrometer. In the range $4000\text{--}200\text{ cm}^{-1}$. ^{13}C NMR, ^{31}P NMR, and ^{119}Sn NMR spectra of these derivatives have been recorded in $CDCl_3$ on a Bruker DRX-300 spectrometer using TMS, H_3PO_4 and tetramethyltin standards, respectively. Molecular weight were measured on a Knauer Vapour pressure osmometer in $CHCl_3$ at $45^\circ C$. Elemental analysis for Sn and S were carried out by standards procedure.¹³ Carbon, hydrogen, and nitrogen were estimated by Coleman C.H.N. analyzers.

RESULT AND DISCUSSION

$[(RO)P(S)S_2][(Me)_3Sn]_2$

All 10 compounds are white, solid, and soluble in common organic solvents (benzene, dichloromethane, chloroform, etc.) and coordinating solvents. The complex are quite stable at room temperature. The stoichiometry of the compound is fixed irrespective of the concentration of ligand used. The molecular weight measurements indicate monomeric nature of these compounds in dilute chloroform solution at $45^\circ C$.

The IR spectra of the complexes have been recorded in the $4000\text{--}200\text{ cm}^{-1}$ region. The bands observed in the region $945\text{--}965$ and $870\text{--}840\text{ cm}^{-1}$, have been assigned to $\nu[P-O-(C)]$ and $\nu[(P)-O-C]$, respectively. The $\nu[P=S]$ mode may be characterized by the presence of a band in the $700\text{--}660\text{ cm}^{-1}$ region, indicating the bidentate nature of trithiophosphate ligand. The band present in the $690\text{--}625\text{ cm}^{-1}$ region may be

ascribed to $\nu[\text{P}-\text{S}]$ stretching mode. Appearance of a new band (in comparison to free ligand) in the $400\text{--}360\text{ cm}^{-1}$ region indicates the formation of a Sn–S bond. The Sn–C bond band were found in an overlapping region $635\text{--}595\text{ cm}^{-1}$. The ^{13}C NMR spectra of these compounds show characteristic resonance due to the alkoxy and phenoxy groups. The ^{13}C resonance for the carbon atoms of Me_3Sn group are as expected. The ^{13}C resonance for the carbon atom of P–O–C group appears as doublet due to coupling with the ^{31}P nuclei.

TABLE III ^{13}C NMR Spectral Data for Trimethyltin(IV) Trithiophosphates

S. no.	Compound	Chemical shift (δ , ppm)	
		Me_3Sn carbons	$\text{S}_2(\text{S})\text{P}(\text{OR})$ carbons
1.	$[(\text{MeO})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.76, d, $^1J_{\text{Sn-c}} = 208\text{ cps}$	52.56, d, C; $^2J_{\text{P-C}} = 24\text{ cps}$
2.	$[(\text{EtO})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.90, d, $^1J_{\text{Sn-c}} = 205\text{ cps}$	53.62, d, C; $^2J_{\text{P-C}} = 26\text{ cps}$ 15.43, C ₂
3.	$[(\text{Pr}^n\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.82, d, $^1J_{\text{Sn-c}} = 212\text{ cps}$	68.98, d, C; $^2J_{\text{P-C}} = 88\text{ cps}$ 23.62, C ₂ 12.44, C ₃
4.	$[(\text{Pr}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.64, d, $^1J_{\text{Sn-c}} = 209\text{ cps}$	66.64, d, C; $^2J_{\text{P-C}} = 68\text{ cps}$ 26.54, C ₂ 25.80, C ₃
5.	$[(\text{Bu}^n\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.54, d, $^1J_{\text{Sn-c}} = 211\text{ cps}$	70.14, d, C; $^2J_{\text{P-C}} = 56\text{ cps}$ 31.12, C ₂ 13.12, C ₃ 15.12, C ₄
6.	$[(\text{Bu}^s\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.42, d, $^1J_{\text{Sn-c}} = 213\text{ cps}$	19.8, d, C; $^2J_{\text{P-C}} = 10\text{ cps}$ 74.26, C ₂ 31.08, C ₃ 9.16, C ₄
7.	$[(\text{Bu}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.56, d, $^1J_{\text{Sn-c}} = 209\text{ cps}$	74.04, d, C; $^2J_{\text{P-C}} = 12\text{ cps}$ 29.40, C ₂ 17.52, C ₃
8.	$[(\text{Am}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.68, d, $^1J_{\text{Sn-c}} = 214\text{ cps}$	63.96, d, C; $^2J_{\text{P-C}} = 24\text{ cps}$ 29.52, C ₂ 23.68, C ₃ 23.69, C ₄
9.	$[(\text{C.h.O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.34, d, $^1J_{\text{Sn-c}} = 210\text{ cps}$	75.84, d, C; $^2J_{\text{P-C}} = 344\text{ cps}$ 32.99, C _{2,6} 22.60, C _{3,5} 24.52, C ₄
10.	$[(\text{PhO})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	24.18, d, $^1J_{\text{Sn-c}} = 206\text{ cps}$	152.8, d, C; $^2J_{\text{P-C}} = 436\text{ cps}$ 110.52, C _{2,6} 120.40, C _{3,5} 108.62, C ₄

In the proton decoupled ^{31}P NMR spectra, only one sharp resonance for each compound in the range 95.29–99.38 ppm is obtained. In ^{119}Sn NMR spectra the coupling constant provides valuable information about coordination number of tin(IV). For the four coordinated trimethyl tin(IV) compounds $^1J_{\text{Sn-C}}$ values have been reported in the range 205–214 cps.

The ^{119}Sn NMR chemical shifts of all the compounds have been observed in the range at δ –211.23 to δ –238.4 ppm. These ^{119}Sn NMR chemical shifts suggest that in compound number 1–10 the tin(IV) is four coordinated.^{6,7} All the spectral data are summarized in Tables III and IV.

$[(\text{RO})\text{P}(\text{S})\text{S}_2][(\text{Bu}_3\text{Sn})_2]$

All 10 compounds are white solid, soluble in common organic (benzene, dichloromethane, chloroform etc.) and coordinating solvents. The complexes are quite stable at room temperature. The stoichiometry of the compounds is fixed in respective of the concentration of ligand used. The molecular weight measurement indicate monomeric nature of these compounds in dilute chloroform solution at 45°C.

The IR spectra of the complexes have been recorded in the 4000–200 cm^{-1} region. The bands observed in the 1035–660 and 865–835 cm^{-1} regions have been assigned to $\nu[(\text{P})\text{—O—C}]$ and $\nu[\text{P—O—(C)}]$, respectively. The $\nu[\text{P=O}]$ mode may be characterized by the presence of a band in the 695–650 cm^{-1} region indicating the bidentate nature of trithiophosphate ligand. The band present in the 685–615 cm^{-1} region

TABLE IV ^{31}P and ^{119}Sn NMR Spectral Data for Trimethyltin(IV) Trithiophosphates

S. no.	Compound	Chemical shift (δ , ppm)	
		^{31}P NMR	^{119}Sn NMR
1.	$[(\text{MeO})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	99.02	–236.32
2.	$[(\text{EtO})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	97.34	–230.36
3.	$[(\text{Pr}^n\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	97.46	–226.32
4.	$[(\text{Pr}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	97.02	–224.36
5.	$[(\text{Bu}^n\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	95.08	–232.48
6.	$[(\text{Bu}^s\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	95.59	–230.28
7.	$[(\text{Bu}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	97.92	–228.14
8.	$[(\text{Am}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	96.48	–216.18
9.	$[(\text{C.h.O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	98.02	–211.23
10.	$[(\text{Ph O})\text{P}(\text{S})\text{S}_2][\text{Me}_3\text{Sn}]_2$	99.38	–238.40

TABLE V ¹³C NMR Spectral Data for Tributyltin(IV) Trithiophosphates

S. no.	Compound	Chemical shift (δ, ppm)	
		Bu ₃ Sn carbons	S ₂ (S)P(OR) carbons
1.	[(MeO)P(S)S ₂][Me ₃ Sn] ₂	29.32, d, ¹ J _{Sn-c} = 222 cps; C ₁ 26.89, C ₂ 17.31, C ₃ 13.19, C ₄	52.54, d, C; ² J _{P-C} = 24 CPS
2.	[(EtO)P(S)S ₂][Me ₃ Sn] ₂	29.24, d, ¹ J _{Sn-c} = 220 cps; C ₁ 26.84, C ₂ 17.30, C ₃ 13.14, C ₄	53.60, d, C; ² J _{P-C} = 26 CPS 15.48, C ₂
3.	[(Pr ⁿ O)P(S)S ₂][Me ₃ Sn] ₂	29.30, d, ¹ J _{Sn-c} = 224 cps; C ₁ 26.64, C ₂ 17.28, C ₃ 13.16, C ₄	68.96, d, C; ² J _{P-C} = 88 CPS 23.66, C ₂ 12.40, C ₃
4.	[Pr ⁱ O)P(S)S ₂][Me ₃ Sn] ₂	29.28, d, ¹ J _{Sn-c} = 224 cps; C ₁ 26.46, C ₂ 17.82, C ₃ 13.61, C ₄	66.58, d, C; ² J _{P-C} = 70 CPS 26.56, C ₂ 25.82, C ₃
5.	[Bu ⁿ O)P(S)S ₂][Me ₃ Sn] ₂	29.26, d, ¹ J _{Sn-c} = 226 cps; C ₁ 26.42, C ₂ 17.52, C ₃ 13.58, C ₄	70.16, d, C; ² J _{P-C} = 56 CPS 31.14, C ₂ 13.10, C ₃ 15.14, C ₄
6.	[(Bu ^s O)P(S)S ₂][Me ₃ Sn] ₂	29.38, d, ¹ J _{Sn-c} = 224 cps; C ₁ 26.36, C ₂ 17.90, C ₃ 13.68, C ₄	19.6, d, C; ² J _{P-C} = 12 CPS 74.28, C ₂ 31.18, C ₃ 9.18, C ₄
7.	[(Bu ⁱ O)P(S)S ₂][Me ₃ Sn] ₂	29.36, d, ¹ J _{Sn-c} = 222 cps; C ₁ 26.32, C ₂ 17.92, C ₃ 13.64, C ₄	74.02, d, C; ² J _{P-C} = 14 CPS 29.48, C ₂ 17.50, C ₃
8.	[(Am ⁱ O)P(S)S ₂][Me ₃ Sn] ₂	29.32, d, ¹ J _{Sn-c} = 226 cps; C ₁ 26.34, C ₂ 17.96, C ₃ 13.50, C ₄	63.98, d, C; ² J _{P-C} = 24 CPS 39.54, C ₂ 23.86, C ₃ 23.96, C ₄
9.	[(C.h.O)P(S)S ₂][Me ₃ Sn] ₂	29.42, d, ¹ J _{Sn-c} = 224 cps; C ₁ 26.48, C ₂ 17.94, C ₃ 13.52, C ₄	75.88, d, C; ² J _{P-C} = 340 CPS 32.98, C _{2,6} 22.40 C _{3,5} 24.58, C ₄
10.	[(Ph O)P(S)S ₂][Me ₃ Sn] ₂	29.48, d, ¹ J _{Sn-c} = 228 cps; C ₁ 26.52, C ₂ 17.98, C ₃ 13.58, C ₄	152.6, d, C; ² J _{P-C} = 432 CPS 110.50, C _{2,6} 122.42, C _{3,5} 108.58, C ₄

TABLE VI ^{31}P and ^{119}Sn NMR Spectral Data for Tributyltin(IV) Trithiophosphates

S. No.	Compound	Chemical shift (δ , ppm)	
		^{31}P NMR	^{119}Sn NMR
1.	$[(\text{MeO})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	94.18	-132.42
2.	$[(\text{EtO})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	93.26	-128.60
3.	$[(\text{Pr}^n\text{O})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	95.12	-134.56
4.	$[(\text{Pr}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	95.10	-136.38
5.	$[(\text{Bu}^n\text{O})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	94.86	-135.48
6.	$[(\text{Bu}^o\text{O})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	94.92	-138.56
7.	$[(\text{Bu}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	94.96	-140.52
8.	$[(\text{Am}^i\text{O})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	95.86	-142.50
9.	$[(\text{C.h.O})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	95.98	-140.18
10.	$[(\text{Ph O})\text{P}(\text{S})\text{S}_2][\text{Bu}_3\text{Sn}]_2$	96.28	-148.02

may be ascribed to $\nu[\text{P}-\text{S}]$ stretching mode. Appearance of a new band (in comparison to free ligand) in the $395\text{--}368\text{ cm}^{-1}$ region, indicates the formation of a Sn-S bond. The Sn-C bond band were found in an overlapping region $625\text{--}585\text{ cm}^{-1}$. The ^{13}C NMR spectra of these compounds show characteristic resonance due to the alkoxy and phenoxy groups. The ^{13}C resonance for carbon atoms of Bu_3Sn groups are as expected. The ^{13}C resonance for the carbon atom of P-O-C group appears as a doublet due to coupling with the ^{31}P nuclei.

In the proton decoupled ^{31}P NMR spectra, only one sharp resonance for each compound in the range $92.19\text{--}96.28\text{ ppm}$ is obtained. In ^{119}Sn NMR spectra the coupling constant provides valuable information about coordination number of tin(IV). For the four coordinated $\text{Bu}_3\text{Sn(IV)}$ compounds, $^1J_{\text{Sn-C}}$ values have been reported in the range $220\text{--}228\text{ cps}$. The ^{119}Sn NMR chemical shifts of all the compounds observed in the range $\delta\text{--}128.60$ to $\delta\text{--}148.02\text{ ppm}$. These ^{119}Sn NMR chemical shifts suggest that in compound number 11-20 the tin(IV) is four coordinated.^{6,7} All the spectral data are summarized in Tables V and VI.

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