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Di- and tri-organotin(IV) diphenyldithiophosphinates

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Abstract

Di- and tri-organotin(IV) diphenyldithiophosphinates, $R_2Sn(S_2PPh_2)_2$ (R = Me, n-Bu, Bz, Ph) and $R_3SnS_2PPh_2$ (R = Me, Cy, Bz, Ph) were prepared by reaction of the corresponding organotin chlorides or oxides with diphenyldithiophosphinic acid or its ammonium salt. All the compounds were characterized by IR and ^1H NMR spectra. For $R_2Sn(S_2PPh_2)_2$ (R = Me, Ph) and $Ph_3SnS_2PPh_2$ mass spectra and tin-119m Mössbauer spectra were also recorded. Monodentate bonding of the dithiophosphinic ligand and tetrahedral structures are proposed for the triorganotin derivatives, while in diorganotin compounds there appears to be distorted octahedral geometry around tin, with anisobidentate dithiophosphinic ligands.

Introduction

Organotin dithiophosphates, $R_nSn[S_2P(OR')_2]_{4-n}$, have received considerable attention in recent years, and their investigation by X-ray diffraction and other physical methods has revealed interesting structural features [1–4]. These results have been reviewed in the context of the chemistry of organometallic dithiophosphates [5,6].

However, the dithiophosphinates, $R_nSn(S_2PR'_2)_{4-n}$, have received much less attention. A limited number of derivatives have been synthesized [7–13], and some tin-119m Mössbauer spectra [11,13] and the crystal structures of $Me_2Sn(S_2PMe_2)_2$

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[14] and $Me_2Sn(S_2PEt_2)_2$ [13] have been reported. The finding that derivatives of this type, e.g., $Ph_2Sn(S_2PPh_2)_2$, exhibit some antitumor activity [15], and the ability of dithiophosphorus ligands to exhibit various coordination patterns [16] are further reasons for the investigation of other members of this class of compounds. The literature data, for example those on melting points, are sometimes contradictory, and the list of possible organotin dithiophosphinates contains many gaps. We describe here the synthesis and characterization of some diphenyldithiophosphinates, $R_nSn(S_2PPh_2)_{4-n}$, in which R = Me, n-Bu, cyclo-C_6H_{11} (abbreviated Cy), Ph, CH_2Ph (abbreviated Bz) and $n = 2$ or 3.

Results and discussion

Preparation

The organotin diphenyldithiophosphinates were prepared by the following two routes:

(A) The reaction of organotin chlorides with diphenyldithiophosphinic acid or its ammonium salt:

$$R_nSnCl_{4-n} + (4-n)MS_2PPh_2 \rightarrow R_nSn(S_2PPh_2)_{4-n} + (4-n)HCl \qquad (1)$$

(M = H, NH_4; $n = 2$, R = Me, n-Bu, Bz, Ph; $n = 3$, R = Me, Cy, Bz, Ph)

(B) The reaction of organotin oxides or hydroxides with diphenyldithiophosphinic acid:

$$R_2SnO + 2HS_2PPh_2 \rightarrow R_2Sn(S_2PPh_2)_2 + H_2O \qquad (2)$$

(R = Me, Ph, n-Bu)

$$R_3SnOH + HS_2PPh_2 \rightarrow R_3SnS_2PPh_2 + H_2O \qquad (3)$$

(R = Cy, Ph)

The solvents used were anhydrous ethanol or benzene. Diphenyldithiophosphinates made by the two methods had identical melting points and spectra. All the compounds are white solids, except for tribenzyltin diphenyldithiophosphinate, which is an oil of high boiling point. They are all stable in air for a reasonable time. Yields, recrystallization solvents, and melting points are given in Table 1.

Spectra and structure

The compounds prepared were characterized by infrared, ^1H NMR, tin-119m Mössbauer, and mass spectra.

Infrared spectra. The infrared spectra of the compounds reported here are listed in Table 2. The assignments of the bands were made in accordance with literature data [17–20].

The PS_2 and SnC_n stretching vibrations provided useful structural information [18,21]. All the compounds show two strong absorptions in regions characteristic for P–S bands [22]: one, in the range 660–640 cm^{-1}, has been assigned to ν_{as}(P=S) stretching vibration of the P=S double bond, and the other, in the range 550–530 cm^{-1}, has been assigned to the P–S single bond stretching, ν_s(P–S). The difference, $\Delta\nu$, between these two frequencies, provides information about the coordination pattern of the dithiophosphinic ligand [21]. Thus, a value of $\Delta\nu$ of 50–70 cm^{-1},

Table 1

Preparation of $R_nSn(S_2PPh_2)_{4-n}$

	R	n	Method	M.p. (°C)	Yield (%)	Recryst. solvent	Analysis (Found (calcd.)(%))	
							C	H
1	Me	2	A	178–180 [a]	91	EtOH		
			B	178–180	98			
2	n-Bu	2	A	138–140 [b]	96	EtOH		
			B	138–140	97			
3	Bz	2	A	155–156	87	Me_2CO	56.59	4.01
							(57.09)	(4.26)
4	Ph	2	A	225 [c]	81	C_6H_6		
			B	223–224	98			
5	Me	3	A	42–44	91	–	43.15	4.24
							(43.62)	(4.60)
6	Cy	3	A	85–87	77	EtOH	57.93	6.58
			B	85–87	74		(58.38)	(6.97)
7	Bz	3	A	oil	84	–	61.73	4.64
							(61.81)	(4.84)
8	Ph	3	A	128–130 [d]	87	EtOH		
			B	128–130	100			

[a] M.p. 161–162 °C [8]. [b] M.p. 78 °C [12], 114 °C (dec.) [11], 138–140 °C [9,10]. [c] M.p. 215–217 °C [11]. [d] M.p. 46 °C [12], 128–130 °C [38].

Table 2

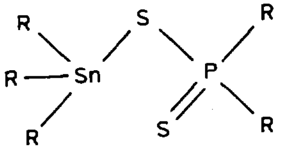
Infrared data for $R_nSn(S_2PPh_2)_{4-n}$ (in cm^{-1}) [a,b]

	R	n	$\nu_{as}(PS_2)$	$\nu_s(PS_2)$	$\Delta\nu$	$\nu_{as}(Sn–C)$	$\nu_s(Sn–C)$
1	Me	2	644s	542s	102	561w	517w
2	n-Bu	2	650s	549s	101	600w	510w
3	Bz	2	646s	544s	102	444w	426w
4	Ph	2	644s	546s	98		
5	Me	3	656s	539s	117		510m
6	Cy	3	655s	536s	119		
7	Bz [c]	3	651s	531s	120	448m	
8	Ph	3	659s	535s	124		

[a] s = strong, m = medium, w = weak. [b] In KBr pellets. [c] In CS_2 solution.

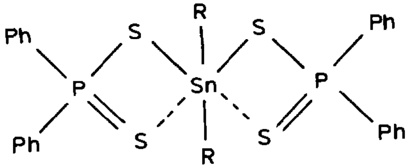
falling between the values for P–S asymmetric and P–S symmetric stretching vibrations, suggests a bidentate coordination of the dithio ligand, with equivalent P–S bonds, while values of $\Delta\nu$ larger than 95 cm^{-1} are diagnostic for a monodentate behavior of the ligand, with pure double and single phosphorus–sulfur bonds. Values of $\Delta\nu$ between 70–90 cm^{-1} are characteristic of anisobidentate coordination of the dithio ligand, with non-equivalent P–S bonds.

The differences $\Delta\nu$ for the triorganotin diphenyldithiophosphinates lie in the range 117–124 cm^{-1}, consistent with a four-coordinated tin atom with the diphenyldithiophosphinic group acting as a monodentate ligand (**a**):



(a)

Although the difference, $\Delta\nu$, for $R_2Sn(S_2PPh_2)_2$ indicates a monocoordinated behavior of the two dithiophosphinic ligands, they are appreciably smaller (98–102 cm^{-1}) than those for $R_3SnS_2PPh_2$. This can be accounted for in terms of a weak interaction between the tin atom and the sulfur atom double bonded to phosphorus (**b**):



(b)

A similar structure, with anisobidentate dithiophosphinic ligands, was proposed by Mullins for a number of $R_2Sn(S_2PR'_2)_2$ derivatives (R = n-Bu, Ph; R′ = Me, Et, Ph), on the basis of dipole moments and tin-119m Mössbauer data [11]. The observation of a $\nu_{sym}(Sn–C)$ band in the infrared spectra of $R_2Sn(S_2PPh_2)_2$ (R = Me, n-Bu, Bz) and $Me_3SnS_2PPh_2$ is consistent with an unsymmetrical octahedral environment of the tin atom (probably with *trans*-SnR_2 units) for the former compounds and with a tetrahedral structure and angular Sn–C bonds for the latter.

The Sn–C stretching vibrations for phenyltin derivatives lie below 400 cm^{-1} [19] and were not recorded.

^1H NMR spectra. Chemical shifts and phosphorus–hydrogen coupling constants are listed in Table 3. The ^1H NMR spectra of the investigated compounds each contain two separate groups of signals: one for the phenyl groups attached to phosphorus atom, and the other for the organic groups bonded to tin atom. All the compounds exhibit similar signals for diphenyldithiophosphinic ligand: one multiplet for the *meta* and *para* protons of the phenyl groups and a doublet of multiplets for those in *ortho* positions, the latter arising from a $|^3J(^{31}P–C–C–^1H)|$ coupling with the ^{31}P nucleus and with the other phenyl protons.

From the value of $|^2J(^{119}Sn–C–^1H)|$ 81.6 Hz, measured for $Me_2Sn(S_2PPh_2)_2$, the relation given by Lockhart [23], viz: $\theta_{deg} = 0.0161|^2J|^2 - 1.32|^2J| + 133.4$ indicates that the Me–Sn–Me bond angle is 132.9°. This compares satisfactorily with the value calculated from Mössbauer data (see below).

Although the ^1H NMR spectra confirm the nature of the compounds investigated, they provide no detailed information about the coordinated ligands.

Tin-119m Mössbauer spectra. The tin-119m Mössbauer data are listed in Table 4. They are consistent with structures (**a**) and (**b**) suggested by the IR data. The isomer shift (*IS*) values confirm that the compounds investigated contain tin(IV) [24,25]. Using the correlation between the magnitude of the quadrupole splitting

Table 3

^{1}H NMR data for $R_nSn(S_2PPh_2)_{4-n}$ [a,b,c]

	R	*n*	Chemical shifts (ppm) and coupling constants (Hz)
1	Me	2	1.47s (6H), $^2J(^{117}SnH)$ 78, $^2J(^{119}SnH)$ 81.6, (Sn–CH_3) 7.34m (12H, *meta* + *para*), 7.83dm (8H, *ortho*), ^3J(PH) 19.2, (P–C_6H_5)
2	n-Bu	2	0.67–1.83m (18H), (Sn–CH_2–CH_2–CH_2–CH_3) 7.36m (12H, *meta* + *para*), 7.90dm (8H, *ortho*), ^3J(PH) 21.6, (P–C_6H_5)
3	Bz	2	3.66s (4H), (Sn–CH_2–C_6H_5) 6.85–7.89m (30H), (Sn–CH_2–C_6H_5, P–C_6H_5)
5	Me	3	0.48s (9H), $^2J(^{117}SnH)$ 56.4, $^2J(^{119}SnH)$ 58.8, (Sn–CH_3) 7.30m (6H, *meta* + *para*), 7.89dm (4H, *ortho*), ^3J(PH) 18.4, (P–C_6H_5)
6	Cy	3	1.6m (33H), (Sn–C_6H_{11}) 7.36m (6H, *meta* + *para*), 8.01dm (4H, *ortho*), ^3J(PH) 20, (P–C_6H_5)
7	Bz	3	2.61s (6H), (Sn–CH_2–C_6H_5) 6.71–7.99m (25H), (Sn–CH_2–C_6H_5, P–C_6H_5)
8	Ph	3	7.28–7.94m (30H), (Sn–C_6H_5, P–C_6H_5)

[a] In $CDCl_3$ and TMS as internal standard. [b] s = singlet, m = multiplet, dm = doublet of multiplets; [c] *ortho, meta* and *para* = *ortho, meta* and *para* protons of phenyl groups linked to phosphorus atom;

Table 4

Tin-119m Mössbauer data for $R_nSn(S_2PPh_2)_{4-n}$ (at 77K, in mm s^{-1})

Compound	*IS* ±0.03	*QS* ±0.06	Γ_1 ±0.03	Γ_2 ±0.03	$\rho = QS/IS$
$Me_2Sn(S_2PPh_2)_2$	1.49	2.96	1.69	1.58	1.99
$Ph_2Sn(S_2PPh_2)_2$ [a]	1.48	3.00	1.76	1.76	2.03
$Ph_3SnS_2PPh_2$	1.44	2.95	1.32	1.27	2.05

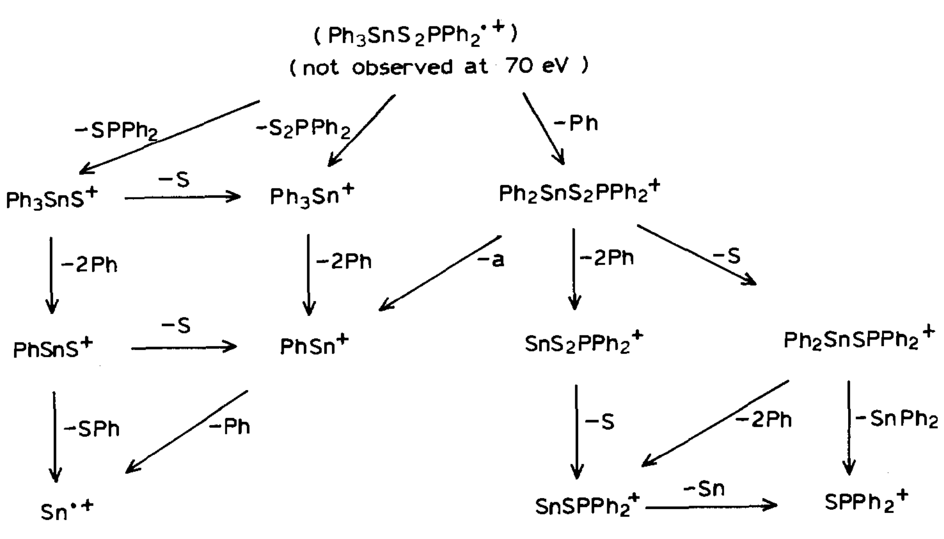
[a] *IS* = 1.46, iQ*S* = 2.98 [11];

Table 5

70 eV monoisotopic mass spectra of $R_nSn(S_2PPh_2)_{4-n}$ (*m/e*, (%)) [a]

	$R_2Sn(S_2PPh_2)_2$		$R_3SnS_2PPh_2$ R = Ph
	R = Me	R = Ph	
$RSn(S_2PPh_2)_2{}^+$	633 (8%)		
$Sn(S_2PPh_2)_2{}^{\cdot+}$	618 (2%)	618 (22%)	
$R_2SnS_2PPh_2{}^+$	399 (100%)	523 (20%)	523 (49%)
$R_2SnSPPh_2{}^+$		491 (8%)	491 (17%)
R_3SnS^+			383 (4%)
$SnS_2PPh_2{}^+$	369 (23%)	369 (76%)	369 (47%)
R_3Sn^+			351 (25%)
$SnSPPh_2{}^+$			337 (0.5%)
$SPPh_2{}^+$	305 (66%)	305 (100%)	305 (100%)
$RSnS^+$		229 (7%)	229 (11%)
RSn^+		197 (15%)	197 (24%)
$Sn^{\cdot+}$			120 (7%)

[a] The *m/e* values were computed according to H = 1, C = 12, P = 31, S = 32, Sn = 120;



a = PhS_2PPh_2

Fig. 1. Fragmentation pattern of $Ph_3SnS_2PPh_2$.

(*QS*) values and the C–Sn–C bond angle in diorganotin compounds [26], we estimate this angle as 126.6° and 134.9°, for the dimethyltin- and diphenyltin-bis(diphenyldithiophosphinates), respectively. The magnitudes of the *QS* indicate a *trans*-configuration of the organic groups bonded to tin atom in the diorganotin(IV) derivatives [24,25]. The ρ (ratio between *QS* and *IS*) values (1.99–2.05) are at the limit between a four-coordinated and a higher than four-coordinated configuration of the tin atom [3,24,25]. Thus a weak interaction between the sulfur atom double bonded to phosphorus and the tin atom may have to be considered.

Mass spectra. The 70 eV monoisotopic mass spectra of $R_2Sn(S_2PPh_2)_2$ (R = Me and Ph) and $Ph_3SnS_2PPh_2$ are listed in Table 5.

For the triphenyltin derivative the spectrum is easily interpreted in terms of the fragmentation pattern shown in Fig. 1. This pattern shows that the cleavage of the tin–carbon or tin–sulfur bonds are not the only possible ones, as they are for other organotin compounds containing Sn–S bonds [27,28], the presence of other hetero-elements (P, S) are responsible for this peculiar behavior.

The presence of a Ph_3SnS^+ ion could be explained in terms of loss of Ph_2PS group from the molecular ion which was not detected at 70 eV, i.e., by the cleavage of a P–S bond. The other ions from first fragmentation, $Ph_2SnS_2PPh_2{}^+$ and Ph_3Sn^+, are those obtained by cleavage of a carbon–tin and sulfur–tin bond, respectively. Fig. 1. also shows clearly that only tin fragments with tin in oxidation state II or IV are observed in the mass spectra, as for other organotin compounds [27,28]. The base peak is Ph_2PS^+. Three other rather intense ions are also seen: $PhPSH^+$ (*m/e* 141, 54%), Ph_3PS^+ (*m/e* 414, 9%) and $Ph_3PS_2{}^+$ (*m/e* 446, 3%).

For $R_2Sn(S_2PPh_2)_2$ (R = Me, Ph), the situation is more complex. There are four common ions in both compounds and others are present only for dimethyl- or

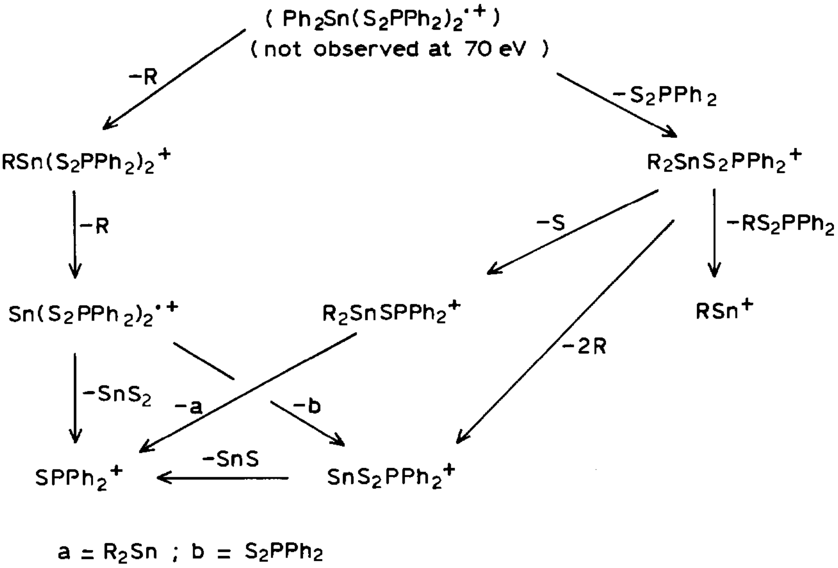


Fig. 2. Fragmentation pattern of $R_2Sn(S_2PPh_2)_2$.

diphenyltin derivative (Table 5), so that the fragmentation pattern shown in Fig. 2. does not necessarily apply to a specific case.

One of the expected ions [27,28] of the first fragmentation, $R_2SnS_2PPh_2^+$, is the base peak for the dimethyltin derivative, while that for the diphenyltin compound is again Ph_2PS^+, as for $Ph_3SnS_2PPh_2$. The second expected ion of first fragmentation, $RSn(S_2PPh_2)_2^+$, is much less abundant in the dimethyltin and absent in the diphenyltin derivative.

There is now at least one exception to the general rule that only ions in which the tin atom is in oxidation state II or IV are observed: the $Sn(S_2PPh_2)_2^{\cdot+}$ ion is present in both compounds, and its abundance is rather high for the diphenyltin derivative.

For $Me_2Sn(S_2PPh_2)_2$ an unusual ion is present: $Ph_2PS_2SnMe_2S^+$ (*m/e* 431, 3.5% of the base peak), which may be $Ph_2PS_2SnMe(SMe)^+$ or $(Ph_2PS_2)Me_2SnS^+$. The second possibility is attractive because it could easily be formed from the molecular ion by cleavage of a sulfur–phosphorus bond. Two other ions, $PhMe_2Sn^+$ (*m/e* 227, 7%) and $PhSn^+$ (*m/e* 197, 3%) are also present, showing that a rearrangement of a phenyl group from phosphorus to tin takes place. A very weak MeS_2Sn^+ ion (*m/e* 199, 0.2%) is also present. For $Ph_2Sn(S_2PPh_2)_2$, a Ph_3Sn^+ ion (*m/e* 351, 12%) is present, and very weak $Ph_3P_2SSn^+$ (*m/e* 545) and $Ph_5P_2SSn^+$ (*m/e* 599) ions can also be recognized, and these ions are also present for $Ph_3SnS_2PPh_2$. Another common ion in the spectra of these two compounds is $PhPSH^+$ ion (*m/e* 141), which is rather abundant: 54% for $Ph_2Sn(S_2PPh_2)_2$ and 43% for $Ph_3SnS_2PPh_2$.

Experimental

Organotin chlorides, R_2SnCl_2 (R = Me, n-Bu) and R_3SnCl (R = Me, Ph), were commercial products. Bz_2SnCl_2 and Bz_3SnCl were prepared as described by

Shishido, from tin and benzyl chlorides [29], and Cy_3SnCl from $SnCl_4$ and Cy_4Sn [30,31]. Ph_2SnCl_2 was obtained from Ph_2SnO and concentrated hydrochloric acid, the oxide being a by-product of the preparation of Ph_2SbCl_3 [32]. The organotin oxides and hydroxides were obtained by hydrolysis of the corresponding chlorides with aqueous NaOH or aqueous ammonia [33–36].

The diphenyldithiophosphinic acid was synthesized by a published method from benzene and phosphorus pentasulfide in the presence of anhydrous $AlCl_3$ [37], and its ammonium salt was made by bubbling anhydrous ammonia through a benzene solution of the acid.

The IR spectra were recorded for KBr pellets or CS_2 solutions on a SPECORD 75 IR C. Zeiss-Jena (DDR) instrument, in the range 4000–400 cm^{-1} and the ^1H NMR spectra on a Varian EM 360-L spectrometer. Tin-119m Mössbauer spectra were recorded at 77 K on a Ranger Engineering constant-acceleration spectrometer using $Ca^{119m}SnO_3$ (New England Nuclear Corp.) as the source and as standard reference material for zero velocity. The mass spectra were recorded on an AEI MS-902 S instrument.

All compounds gave correct elemental (C, H) analyses determined with a Perkin–Elmer C–H–N Analyzer).

1. *Dimethyltin bis(diphenyldithiophosphinate)*

A solution of 1.25 g (5 mmol) of diphenyldithiophosphinic acid in 20 ml ethanol was added to a solution of 0.55 g (2.5 mmol) of dimethyltin dichloride in 20 ml ethanol. The mixture was stirred under reflux for 2 h. White cyrstals of $Me_2Sn(S_2PPh_2)_2$ separated from the cooled solution. Yield 1.46 g (91%).

Similarly prepared were di-n-butyltin bis(diphenyldithiophosphinate), n-$Bu_2Sn(S_2PPh_2)_2$ (yield 88%), diphenyltin bis(diphenyldithiophosphinate), $Ph_2Sn(S_2PPh_2)_2$ (yield 81%), and triphenyltin diphenyldithiophosphinate, $Ph_3SnS_2PPh_2$ (yield 87%).

2. *Trimethyltin diphenyldithiophosphinate*

A solution of 0.996 g (5 mmol) of trimethyltin chloride in 20 ml anhydrous benzene was added to a suspension of 1.335 g (5 mmol) of ammonium diphenyldithiophosphinate, in 20 ml anhydrous benzene. The mixture was stirred under reflux for 1 h then ammonium chloride was filtered and the clear filtrate evaporated in vacuum. The greenish oily residue crystallized on standing. Yield 1.87 g (91%).

Similarly prepared were dimethyltin bis(diphenyldithiophosphinate), $Me_2Sn(S_2PPh_2)_2$ (yield 91%), di-n-butyltin bis(diphenyldithiophosphinate), n-$Bu_2Sn(S_2PPh_2)_2$ (yield 96%), dibenzyltin bis(diphenyldithiophosphinate), $Bz_2Sn(S_2PPh_2)_2$ (yield 87%), tricyclohexyltin diphenyldithiophosphinate, $Cy_3SnS_2PPh_2$ (yield 77%), tribenzyltin diphenyldithiophosphinate, $Bz_3SnS_2PPh_2$ (yield 84%), and triphenyltin diphenyldithiophosphinate, $Ph_3SnS_2PPh_2$ (yield 87%).

3. *Diphenyltin bis(diphenyldithiophosphinate)*

White crystals of $Ph_2Sn(S_2PPh_2)_2$ separated from the product solution obtained from ethanolic solutions of diphenyltin dichloride (1.72 g, 5 mmol, in 20 ml ethanol) and diphenyldithiophosphinic acid (2.5 g, 10 mmol, in 20 ml ethanol) after 2 h reflux with stirring. The crystals were filtered off and washed with diethyl ether. Yield 81%.

4. *Triphenyltin diphenyldithiophosphinate*

Diphenyldithiophosphinic acid (1.25 g, 5 mmol) in 20 ml anhydrous benzene was refluxed with 1.83 g (5 mmol) triphenyltin hydroxide in 20 ml benzene until the hydroxide had completely dissolved. The solution was evaporated and the residue recrystallized from absolute ethanol. White crystals of $Ph_3SnS_2PPh_2$ were obtained in practically quantitative yield.

Similarly obtained were dimethyltin bis(diphenyldithiophosphinate), $Me_2Sn(S_2PPh_2)_2$, from dimethyltin oxide (yield 98%), di-n-butyltin bis(diphenyldithiophosphinate), n-$Bu_2Sn(S_2PPh_2)_2$, from di-n-butyltin oxide (yield 97%), diphenyltin bis(diphenyldithiophosphinate), $Ph_2Sn(S_2PPh_2)_2$, from diphenyltin oxide (yield 98%), and tricyclohexyltin diphenyldithiophosphinate, $Cy_3SnS_2PPh_2$, from tricyclohexyltin hydroxide (yield 74%).

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