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On the Reactivity of Metal and Organometallic Halides toward $R_3Sn{-}O{-}SnR_3$ Systems

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ABSTRACT: *Interaction of metallic salts (M = Hg, Sb, and Te) with bis(triorganotin)oxide, $(R_3Sn)_2O$, where (R = C_6H_5, p-$CH_3C_6H_4$, and cyclo-C_6H_{11}) at room temperature proceeded with the simultaneous cleavage of the Sn—C and Sn—O bonds, invariably yielding R_2SnO along with other products. Thus the treatment of HgX_2 (X = Cl, CN, SCN) with $(R_3Sn)_2O$ resulted in the formation of polymeric diorganotin oxide R_2SnO along with R_3SnX and RHgX derivatives. The reaction of $SbCl_3$ with $(R_3Sn)_2O$ was found to give R_2SnO, R_3SnCl, and $RSbCl_2$, whereas interaction with $SbCl_5$ provided R_2SnO, R_2SnCl_2, and R_2SbCl_3. Treatment of $TeCl_4$ with $(R_3Sn)_2O$ provided R_2SnO, R_3SnCl, and $RTeCl_3$ at room temperature. At reflux temperature, reaction of $PhTeCl_3$ with $(R_3Sn)_2O$ yielded R_2SnO, R_3SnCl, and mixed diorganotellurium dichloride, $RPhTeCl_2$. The course of reaction indicated the instability of Sn—O—Sn system proceeding via a four-centered mechanism, providing organometallic compounds in profitable yield.* © 2009 Wiley Periodicals, Inc. Heteroatom Chem 20:278–283, 2009; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20547

INTRODUCTION

In sharp contrast to well-documented reactions of metallic and organometallic halides with tetraorganotins and hexaorganoditins, those involving $R_3Sn{-}O{-}SnR_3$ compounds have been studied to a limited extent [1,2]. Action of silicon, tin, and phosphorus-halides on organotin oxides has been reported to produce organotin halides [3] whereas reaction of vinylmagnesium chloride with $(Bu_3Sn)_2O$ yielded tributylvinyltin [4]. Reactions of organometallic halides with diorganotin oxides have also been studied [3]. Datta et al. [5] reported the action of mercuric halides on bis(triphenyltin) oxides in an attempt to isolate $R_3Sn{-}O{-}HgCl$ compounds parallel to earlier reported reactions of AlX_3 (X = Cl, Br) with $R_3Si{-}O{-}SiR_3$, which produced stable and isolable $R_3SiOAlX_2$ compounds [6]. Interaction of $(R_3Sn)_2O$ with thionylchloride, $SOCl_2$ to give organotinhalides has also been investigated [7]. Despite such a variety of reactions of bis(triorganotin)oxide, reactions of the $R_3Sn{-}O{-}SnR_3$ system toward heavier metal and organometal halides and pseudohalides are yet to be investigated.

The present communication deals with an investigation into the reactivity of metallic and organometallic halides, namely HgX_2 (X = Cl, CN, SCN), $SbCl_3$, $SbCl_5$, $TeCl_4$, and $PhTeCl_3$ toward Sn—O—Sn bond in bis(triorganotin) oxides, $(R_3Sn)_2O$ (where R = C_6H_5, *p*-$CH_3C_6H_4$, and *cyclo*-C_6H_4).

Our interest in the present investigation has been twofold (i) to examine the stability of the $R_3Sn{-}O{-}M{-}$ system under the given set of condition, where M = Hg, Sb, and Te, and (ii) to study the pattern and ease of cleavage of Sn—O—Sn and Sn—C bond. Consequently, these reactions could be found useful to produce organotellurium and organoantimony compounds in moderate yields that are

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otherwise obtained by conventional longer routes and in some cases with difficulty, especially in the case of bulkier cyclohyexyl or α-naphthyl group(s) containing organometallic compounds.

RESULTS AND DISCUSSION

Action of Mercuric Salts on Bis(triorganotin) Oxide

The interaction of equimolar amounts of mercuric halides and pseudohalides, HgX_2 (X = Cl, CN, SCN) with bis(triorganotin) oxide in diethyl ether at room temperature proceeded with the formation of diorganotin oxide and the corresponding organo-tin and mercury halides and pseudohalides. The reactions were conducted at room temperature. In each case, polymeric diorganotinoxide as a white precipitate appeared immediately. The reaction may be represented as shown in Eq. (1):

$$(R_3Sn)_2O + HgX_2 \rightarrow R_3SnX + RHgX + R_2SnO \quad (1)$$

where $R = C_6H_5$, $p\text{-}CH_3C_6H_4$ and X = Cl, CN, SCN.

The reaction of mercuric chloride with bis(tri-*p*-tolyltin) oxide, in fact, was performed for the sake of comparison and resulted in the formation of bis(*p*-tolyltin)oxide, *p*-tolylmercuric chloride, and tri(*p*-tolyl)tin chloride. No traces of tetra(*p*-tolyl)tin, however, were obtained.

Action of Tellurium Tetrachloride and Organotellurium Trichloride with Bis(triorganotin) Oxides

Both tin-oxygen and tin-carbon bonds in bis(triorganotin) oxide were found to be prone to attack by $TeCl_4$ and $PhTeCl_3$. Cleavage of tin-carbon bond from $R_nSnR'_{4-n}$ compounds employing $TeCl_4$ and $PhTeCl_3$ is well established and has been used as a convenient method for the preparation of organotellurium compounds in high yield and high purity [8,9]. The reactions of bis(triorganotin) oxide with $TeCl_4$ and $PhTeCl_3$ were conducted at room temperature and in refluxing toluene, respectively, with the immediate formation of white solid, R_2SnO, in the both cases (Eqs. (2) and (3)).

$$R_3Sn—O—SnR_3 + TeCl_4 \xrightarrow[\text{Toluene}]{\text{Room temp.}} R_2SnO + R_3SnCl + RTeCl_3 \quad (2)$$

$$R_3Sn—O—SnR_3 + PhTeCl_3 \xrightarrow[\text{Toluene}]{\text{Reflux temp.}} R_2SnO + R_3SnCl + RPhTeCl_2 \quad (3)$$

where $R = C_6H_5$, $p\text{-}CH_3C_6H_4$, and *cyclo*-C_6H_{11}.

The separation of organotin halides from organotellurium compounds does not pose much difficulty due to a significant difference in solubilities. Organotin chlorides are highly soluble in organic solvents, whereas organotellurium halides are insoluble at room temperature. The reaction of cyclohexyl derivatives seems to be more advantageous because cyclohexyltin or cyclohexyltellurium derivatives are not easily obtained. Furthermore, the reactions also provide unsymmetrical organotellurium derivatives of the type Ph(*cyclo*-C_6H_{11})$TeCl_2$, which are now being used to obtain unsymmetric organotellurium compounds by the successive replacement of the halide group by other anion such as pseudohalide or carboxylate as has been reported in the past for obtaining unsymmetric organotellurium compounds [10].

Action of Antimony Halides on Bis(triorganotin) Oxides

Metallic halides, namely antimony(V) chloride and antimony(III) chloride, have profitably been used to cleave metal-carbon bond(s) to varying extent and in the process themselves get arylated to give Ar_2SbCl_3 or $ArSbCl_2$, respectively [1,2,11]. $SbCl_5$ readily cleaves into two aryl groups from tetraaryltin even at room temperature [12]. On the other hand, $SbCl_3$ being mild electrophile is arylated to give $ArSbCl_2$ under ordinary condition although complete arylation of antimony has also been reported [1,2,11].

It is, therefore, not surprising that the reaction of antimony pentachloride with $(R_3Sn)_2O$ proceeded not only with the specific cleavage of Sn—O—Sn bond but also with the removal of an organic group from the tin atom taking place simultaneously (Eq. (4)).

$$R_3Sn—O—SnR_3 + SbCl_5 \rightarrow R_2SbCl_3 + R_2SnO + R_2SnCl_2 \quad (4)$$

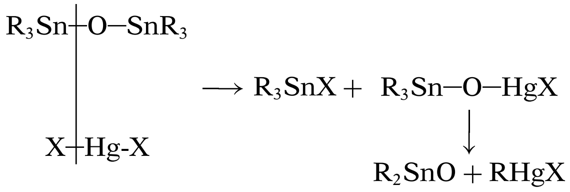
where $R = C_6H_5$ and $p\text{-}CH_3C_6H_4$.

Similarly, the reaction of $SbCl_3$ with bis(triaryltin)oxide yielded $RSbCl_2$, R_2SnO, and R_3SnCl (Eq. (5)).

$$R_3Sn—O—SnR_3 + SbCl_3 \rightarrow R_2SnO + RSbCl_2 + R_3SnCl \quad (5)$$

where $R = C_6H_5$ and $p\text{-}CH_3C_6H_4$.

As has been reported earlier for the reactions of bis(triorganotin) oxides with mercuric halides, the reactions of mercuric cyanide, proceeded in the same fashion. The intermediate $R_3Sn—O—Hg—$ is unstable, rearranges and split itself to give diorganotin oxide and the corresponding organomercuric compounds as shown below:



The mechanisms of the reaction with tellurium and antimony derivatives may or may not be the same, but on the basis of the nature of products obtained it is most reasonable to assume that the reactions proceeded in the same manner as that of mercuric salts [5].

EXPERIMENTAL

Bis(triorganotin) oxides were prepared by the reported procedures [13]. Metallic halides and pseudohalides, HgX_2 (X = Cl, CN, NCS), $TeCl_4$, $SbCl_3$, and $SbCl_5$ were used without further purification. $PhTeCl_3$ was prepared by the cleavage of tetraphenyl tin and had reported melting point and superimposable IR spectra with that an authentic sample. The reactions were performed in an atmosphere of nitrogen under anhydrous conditions. A few representative experiments are described later. Further analytical data are given in Tables 1 and 2.

Reaction of Bis(tri-p-tolyltin) Oxide with $HgCl_2$

To a stirring solution of bis(tri-*p*-tolyltin) oxide (3.0 g, 0.005 mol) in diethyl ether (30 mL), a suspension of mercuric chloride (1.36 g, 0.005 mol) in

TABLE 1 Reaction of Metal and Organometal Halides/Pseudohalides with Bis(triorganotin) Oxides

	Reactants				
S. No.	*R_3Sn—O—SnR_3*	*Metal/Organometal Moiety*	*Compound No.*	*Reaction Products*	*Reference No.*
1.	$(p\text{-CH}_3\text{C}_6\text{H}_4)\text{Sn—O—Sn(p-CH}_3\text{C}_6\text{H}_4)_3$	$HgCl_2$	**1a**	$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{SnO}$	[13]
			1b	$(p\text{-CH}_3\text{C}_6\text{H}_4)\text{HgCl}$	[14]
			1c	$(p\text{-CH}_3\text{C}_6\text{H}_4)_3\text{SnCl}$	[15]
2.	$(C_6H_5)_3Sn$—O—$Sn(C_6H_5)_3$	$Hg(CN)_2$	**2a**	$(C_6H_5)_2SnO$	[13]
			2b	$(C_6H_5)HgCN$	[14]
			2c	$(C_6H_5)_3SnCN$	[15]
3.	$(C_6H_5)_3Sn$—O—$Sn(C_6H_5)_3$	$Hg(SCN)_2$	**3a**	$(C_6H_5)_2SnO$	[13]
			3b	$(C_6H_5)HgNCS$	[16]
			3c	$(C_6H_5)_3SnNCS$	[16]
4.	$(C_6H_5)_3Sn$—O—$Sn(C_6H_5)_3$	$TeCl_4$	**4a**	$(C_6H_5)_2SnO$	[13]
			4b	$(C_6H_5)TeCl_3$	[8,9]
			4c	$(C_6H_5)_3SnCl$	[13]
5.	$(\text{p-CH}_3\text{C}_6\text{H}_4)_3\text{Sn—O—Sn}(p\text{-CH}_3\text{C}_6\text{H}_4)_3$	$TeCl_4$	**5a**	$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{SnO}$	[13]
			5b	$(p\text{-CH}_3\text{C}_6\text{H}_4)\text{TeCl}_3$	[9]
			5c	$(p\text{-CH}_3\text{C}_6\text{H}_4)_3\text{SnCl}$	[13]
6.	$(C_6H_5)_3Sn$—O—$Sn(C_6H_5)_3$	$C_6H_5TeCl_3$	**6a**	$(C_6H_5)_2SnO$	[13]
			6b	$(C_6H_5)_2TeCl_2$	[9]
			6c	$(C_6H_5)_3SnCl$	[13]
7.	$(cyclo\text{-C}_6\text{H}_{11})_3\text{Sn—O—Sn}(cyclo\text{-C}_6\text{H}_{11})_3$	$C_6H_5TeCl_3$	**7a**	$(cyclo\text{-C}_6\text{H}_{11})_2\text{SnO}$	[13]
			7b	$(cyclo\text{-C}_6\text{H}_{11})(\text{C}_6\text{H}_5)\text{TeCl}_2$	[17]
			7c	$(\text{cyclo-C}_6\text{H}_{11})_3\text{SnCl}$	[13]
8.	$(C_6H_5)_3Sn$—O—$Sn(C_6H_5)_3$	$SbCl_5$	**8a**	$(C_6H_5)_2SnO$	[13]
			8b	$(C_6H_5)_2SbCl_3$	[12]
			8c	$(C_6H_5)_2SnCl_2$	[13]
9.	$(p\text{-CH}_3\text{C}_6\text{H}_4)\text{Sn—O—Sn}(p\text{-CH}_3\text{C}_6\text{H}_4)_3$	$SbCl_5$	**9a**	$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{SnO}$	[13]
			9b	$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{SbCl}_3$	[12]
			9c	$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{SnCl}_2$	[15]
10.	$(C_6H_5)_3Sn$—O—$Sn(C_6H_5)_3$	$SbCl_3$	**10a**	$(C_6H_5)_2SnO$	[13]
			10b	$(C_6H_5)\ SbCl_2$	[18]
			10c	$(C_6H_5)_3SnCl$	[12]
11.	$(p\text{-CH}_3\text{C}_6\text{H}_4)\text{Sn—O—Sn}(p\text{-CH}_3\text{C}_6\text{H}_4)_3$	$SbCl_3$	**11a**	$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{SnO}$	[13]
			11b	$(p\text{-CH}_3\text{C}_6\text{H}_4)_3\text{SnCl}$	[13]
			11c	$(p\text{-CH}_3\text{C}_6\text{H}_4)\text{SbCl}_2$	[19]

TABLE 2 Some Physical and Analytical Data for Compounds

Compound No.	*Molecular Formula*	*Molecular Weight*	*M.P. (Literature) (°C)*	*Yield (%)*	*Elemental Analysis: Found (Calculated) (%)*		
					C	*H*	*N*
1a	$C_{14}H_{14}OSn$	316.97	>300	90	52.80 (53.05)	4.30 (4.45)	
1b	C_7H_7ClHg	327.17	232 (233)	86	25.48 (25.70)	2.01 (2.16)	
1c	$C_{21}H_{21}ClSn$	427.55	97 (98.5)	84	58.89 (58.99)	4.90 (4.95)	
2a	$C_{12}H_{10}OSn$	288.92	>300	86	49.52 (49.89)	3.42 (3.49)	
2b	C_7H_5HgN	303.71	202 (204–206)	73	27.68 (27.68)	1.58 (1.66)	4.52 (4.61)
2c	$C_{19}H_{15}NSn$	376.04	254 (355–256)	80	60.60 (60.69)	3.92 (4.02)	3.72 (3.72)
3a	$C_{12}H_{10}OSn$	288.92	>300	90	49.20 (49.89)	3.33 (3.49)	
3b	C_7H_5HgNS	335.78	230 (231–232)	71	25.60 (25.04)	1.46 (1.50)	4.10 (4.17)
3c	$C_{19}H_{15}NSSn$	408.11	170 (172–173)	83	54.81 (55.92)	3.63 (3.70)	3.32 (3.43)
4a	$C_{12}H_{10}OSn$	288.92	>300	90	49.62 (49.89)	3.44 (3.49)	
4b	$C_6H_5Cl_3Te$	311.06	214 (214–216)	75	23.08 (23.17)	1.56 (1.62)	
4c	$C_{18}H_{15}ClSn$	385.47	105 (106–107)	76	55.98 (56.08)	3.83 (3.92)	
5a	$C_{14}H_{14}OSn$	316.97	>300	82	52.84 (53,05)	4.38 (4.45)	
5b	$C_7H_7Cl_3Te$	325.09	218 (217–219)	75	26.72 (25.86)	2.10 (2.17)	
5c	$C_{21}H_{21}ClSn$	427.55	97 (98.5)	72	58.87 (58.99)	4.93 (4.95)	
6a	$C_{12}H_{10}OSn$	288.92	>300	87	49.74 (49.89)	3.28 (3.49)	
6b	$C_{12}H_{10}C_2Te$	352.71	158 (160)	80	40.82 (40.86)	2.76 (2.86)	
6c	$C_{18}H_{15}ClSn$	385.47	105 (105–107)	88	55.96 (56.08)	3.78 (3.92)	
7a	$C_{12}H_{22}OSn$	301.01	>300	90	47.80 (47.88)	7.18 (7.37)	
7b	$C_{12}H_{16}Cl_2Te$	358.76	61 (61–62)	80	40.02 (40.17)	4.48 (4.50)	
7c	$C_{18}H_{33}ClSn$	403.62	126 (127–128)	85	53.52 (53.56)	8.08 (8.24)	
8a	$C_{12}H_{10}OSn$	288.92	>300	90	49.80 (49.89)	3.38 (3.49)	
8b	$C_{12}H_{10}Cl_3Sb$	382.33	170 (171–172)	80	37.56 (37.70)	2.56 (2.64)	
8c	$C_{12}H_{10}Cl_2Sn$	343.82	41 (42–45)	78	41.62 (41.92)	2.88 (2.93)	
9a	$C_{14}H_{14}OSn$	316.97	>300	90	52.79 (53.05)	4.36 (4.45)	
9b	$C_{14}H_{14}Cl_3Sb$	410.38	156 (152–155)	85	40.82 (40.97)	3.33 (3.44)	
9c	$C_{14}H_{14}Cl_2Sn$	371.88	47 (48–49)	70	45.12 (45.22)	3.62 (3.79)	
10a	$C_{12}H_{10}OSn$	288.92	>300	90	49.74 (49.89)	3.32 (3.49)	
10b	$C_6H_5Cl_2Sb$	269.77	60 (62)	71	26.51 (26.71)	1.81 (1.87)	
10c	$C_{18}H_{15}ClSn$	385.47	105 (105–107)	75	55.92 (56.08)	3.79 (3.92)	
11a	$C_{14}H_{14}OSn$	316.97	>300	89	52.75 (53.05)	4.36 (4.45)	
11b	$C_{21}H_{21}ClSn$	427.55	97 (98.5)	82	58.78 (58.99)	4.87 (4.95)	
11c	$C_7H_7Cl_2Sb$	283.80	154 (155)	75	29.37 (29.63)	2.32 (2.49)	

the same solvent (30 mL) was added slowly. A white precipitate appeared immediately. The mixture was further stirred for 2 h and then filtered. The residue was digested with benzene (50 mL) for 10 h in a solvent extractor to get a white residue, which was identified as polymeric di-*p*-tolyltin oxide (1.3 g) **1a** [13].

The benzene extract on evaporation yielded 0.90 g of *p*-tolylmercuric chloride **1b** [14]. The original filtrate on fractional crystallization from petroleum ether (40–60°C) afforded 0.5 g of *p*-tolylmercuric chloride **1b** (total yield of *p*-tolylmercuric chloride 1.40 g (86%)) and tri-*p*-tolytin chloride (1.80 g) **1c** [15].

Reaction of Bis(triphenyltin) Oxide with Mercuric Cyanide

To a stirring solution of bis(triphenyltin)oxide (0.71 g, 1.0 mmol) in diethyl ether (30 mL), a suspension of mercuric cyanide (0.27 g, 1.0 mmol) in the same solvent (30 mL) was added slowly. The white precipitate thus obtained was refluxed with benzene (70 mL) for 12 h and then filtered to leave an insoluble diphenyltin oxide (0.24 g) **2a** [13]. The benzene and diethyl ether solution on work up as discussed earlier yielded phenylmercuric cyanide (0.22 g) **2b** [14] and triphenyltin cyanide (0.30 g) **2c** [15].

Reaction of Bis(triphenyltin) Oxide with Mercuric Thiocyanate

To a stirring solution of bis(triphenyltin) oxide (3.60 g, 0.005 mol) in diethyl ether (300 ml), mercuric thiocyanate (1.58 g, 0.005 mol) in the same solvent (30 mL) was added slowly. The white precipitate thus obtained was refluxed with benzene (100 mL) for 12 h and then filtered to leave an insoluble diphenyltin oxide (1.29 g) **3a** [13]. The benzene and diethyl ether solution on work up as discussed earlier yielded phenylmercuric isothiocyanate (1.20 g) **3b** [16] and triphenyltin isothiocyanate (1.7 g) **3c** [16].

Reaction of Bis(tri-p-tolyltin) Oxide with Tellurium Tetrachloride

To a stirring solution of bis(tri-*p*-tolyltin) oxide (3.0 g, 0.005 mol) in diethyl ether (50 mL), a solution of tellurium tetrachloride (1.34 g, 0.005 mol) in the same solvent (30 mL) was added slowly. A white precipitate appeared immediately. The residue was digested with benzene (100 mL) for 10 h in a soxlet extractor to leave off white residue of di(*p*-tolyl)tin oxide (0.97 g) **5a** [13]. The benzene extract on evaporation yielded (*p*-tolyl)tellurium trichloride (0.92 g) **5b** [9] and tri(*p*-tolyl)tin chloride (1.15) **5c** [13].

Reaction of Bis(tricyclohexyltin) Oxide with Phenyltellurium Trichloride

To a stirring solution of bis(tricyclohexyltin) oxide (0.75 g, 1.0 mmol) in toluene (40 mL), phenyltellurium trichloride (0.31 g, 1.0 mmol) in toluene (20 mL) was added slowly and then refluxed for 6 h at reflux temperature (110°C).

A white precipitate was formed immediately, which was filtered and dried to afford di(cyclohexyl)tin oxide (0.27 g) **7a** [13]. The filtrate was further reduced to about 10 mL. A white solid started to appear at room temperature. The solution was kept under refrigeration overnight. The white solid was filtered and dried to yield (cyclohexyl)phenyltellurium dichloride (0.28 g) **7b** [17]. Afterward, solvent was completely removed and residue obtained was dissolved in petroleum ether (60–80°C). It was then refrigerated to give white crystalline solid, which was filtered and dried under vacuum to give tri(cyclohexyl)tin chloride (0.34 g) **7c** [13].

Reaction of Bis(triphenyltin) Oxide with Antimony Pentachloride

Antimony pentachloride (0.3 g, 1.0 mmol) in solvent diethyl ether (20 mL) was added slowly to a stirring solution of bis(triphenyltin) oxide (0.71 g, 1.0 mmol) in diethyl ether (30 mL). A white solid appeared immediately. The contents were stirred for 2 h at reflux temperature, and then filtered hot. The white residue was characterized as polymeric diphenyltin oxide (0.26 g) **8a** [13]. The filtrate was completely evaporated to give a viscous solid. It was then treated with petroleum ether/*n*-hexane mixture (1:3) (40 mL), concentrated and then cooled. On fractional crystallization, diphenylantimony trichloride (0.30 g) **8b** [12] and diphenyltindichloride (0.26 g) **8c** [13] were obtained.

Reaction of Bis(triphenyltin) Oxide with Antimony Trichloride

To a stirring solution of bis(triphenyltin) oxide (0.71 g, 1.0 mmol) in diethyl ether (100 mL), a solution of $SbCl_3$ (0.22 g, 1.0 mmol) in the same solvent (50 mL) was added slowly. A white solid appeared immediately. The reactants were further stirred for 6 h and then filtered hot. The white solid obtained was identified as polymeric diphenyltin oxide (0.26 g) **10a** [13]. The filtrate was completely evaporated to give a viscous solid. It was then treated with petroleum ether (60–80°C)/*n*-hexane mixture (1:3) (40 mL), concentrated and then cooled. On fractional crystallization, phenylantimony dichloride (0.19 g) **10b** [18] and triphenyltin chloride (0.28 g) **10c** [12] were isolated.

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