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Preparation and properties of unsymmetrical tetraorganotin compounds

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Abstract

Unsymmetrical tetraorganotins, $R_2R^1R^2Sn$ (R = Me, R^1 = Bu, R^2 = Pe (Pe = pentyl) or Ph; R = Bu, R^1 = Pe, R^2 = Ph or Me; R = Pe, R^1 = Bu, R^2 = Me or Ph), $Bu_nR_{4-n}Sn$ ($n = 1$–3, R = Me or Pe) and Pe_2R_2Sn (R = Me or Ph) have been synthesised. Various physical properties, including mass spectra, ^{1}H and ^{13}C NMR spectra, are reported.

Introduction

As part of a study on organotin compounds in the environment, we had need to prepare tetraorganotin compounds to use as standards and/or derivatives for analyses by GLC. In this paper, we report on the syntheses and some physical data for unsymmetric tetraorganotin compounds of the types, $R_nR^1_{4-n}Sn$ and $R_2R^1R^2Sn$.

Experimental

Apparatus. The gas chromatographs used were a Perkin–Elmer Model F-33 GC, equipped with a flame ionization detector (F.I.D.) and a 7 m × 0.33 mm ID BP-1 glass capillary column, and a Shimadzu Model GC-8A, equipped with a flame photometric detector (F.P.D.) (610 nm filter) and a 12 m × 0.53 mm ID BP-1 glass capillary column. The tin analyses were carried out on a Pye Unicam Model SP-9 Atomic Absorption Spectrometer using a published procedure [1].

^{1}H NMR spectra were obtained on a Perkin–Elmer R34 spectrometer operating at 220 MHz while a Varian CFT-20 Spectrometer was used for ^{13}C NMR spectra. Mass spectra were recorded on an AEI-Kratos M30 instrument.

Column chromatography was conducted on a 60 cm × 4 cm ID column of 5% water-deactivated Florisil (Aldrich).

Materials. Tetramethyltin (99% purity), tetrabutyltin (98%), tributyltin chloride (95%), dibutyltin dichloride (97%), butyltin trichloride (95%), dimethyltin dichloride

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(95%), and diphenyltin dichloride (96%) were purchased from Aldrich. Tributyltin chloride and butyltin trichloride were fractional distilled under reduced pressure and dimethyltin dichloride was recrystallised from hexane before use.

Diethyl ether was dried over sodium and distilled prior to use.

Synthesis of tetraorganotin compounds

The compounds, $MeBu_3Sn$, Me_2Bu_2Sn, Me_3BuSn, Bu_3PeSn, Bu_2Pe_2Sn and $BuPe_3Sn$ (Pe = pentyl) were obtained from the appropriate Bu_nSnCl_{4-n} and an excess of the alkyl-Grignard reagent in Et_2O. Me_2Pe_2Sn and Pe_4Sn were prepared by the reaction of the pentyl-Grignard reagent (from PeBr) with Me_2SnCl_2 and SnI_4 respectively.

General Grignard procedure. The solution of the alkyl halide in Et_2O was added dropwise with stirring to a slight excess of Mg. After the addition was complete, the mixture was stirred for 15 min, before the solution of the organotin halide in Et_2O was added. The mixture was stirred for 1 h, and then treated with 10% aq. NH_4Cl. The organic layer was separated and the aqueous layer extracted twice with Et_2O. The combined organic layers were collected and dried over $MgSO_4$, and the solvent was removed to leave a residue. The products were purified by passage through the Fluorisil column with hexane as eluant; appropriate fractions (10 ml) were pooled and the solvent removed. Further purification of $BuPe_3Sn$, Me_2Pe_2Sn, and Pe_4Sn was achieved by fractional distillation under reduced pressure.

Preparation of butyldimethylpentyltin from dimethyltin dichloride

Treatment of Me_2SnCl_2 (0.09 mol) in Et_2O (50 ml) with the Grignard reagent made from PhI (0.20 mol) provided Me_2Ph_2Sn; the crude product was chromatographed on the Florisil column and then distilled under reduced pressure. Iodine was added in small portions with stirring to a solution of Me_2Ph_2Sn (0.05 mol) in hexane at room temperature, the reaction being followed by GLC-FID. Addition of iodine was stopped when less than 0.5% of the Me_2Ph_2Sn remained; at this stage Me_2SnI_2 was beginning to be formed. The solution was filtered through anhydrous Na_2SO_4, the solvent removed, and the Me_2PhSnI purified by distillation under reduced pressure (B.p. 144–146 °C/12 torr; D_4^{20} 1.73; n_D^{25} 1.6240). (Anal. Found: Sn, 34.0%; calc: 33.7%).

A solution of Me_2PhSnI (0.025 mol) in Et_2O was treated with the Grignard reagent made from BuBr (0.03 mol). The product, $Me_2BuPhSn$, was purified by column chromatography and then by distillation. Treatment of $Me_2BuPhSn$ (0.015 mol) in hexane with iodine (0.015 mol) gave Me_2BuSnI (b.p. 96–100 °C/12 torr). Distilled Me_2BuSnI (0.010 mol) was treated with the Grignard reagent made from PeBr (0.015 mol) to give $Me_2BuPeSn$, which was purified by column chromatography.

Preparation of dibutylmethylpentyltin from dibutyltin dichloride

The compound, Bu_2Ph_2Sn, obtained in 78% yield from Bu_2SnCl_2 (0.04 mol) and PhMgI in Et_2O (from PhI, 0.09 mol), was treated with iodine in hexane until almost all the Bu_2Ph_2Sn had reacted, as shown by GC-FID. Fractional distillation of the mixture gave pure Bu_2PhSnI (b.p. 150–155 °C/12 torr; D_4^{20} 1.77); treatment of Bu_2PhSnI (0.025 mol) with the pentyl-Grignard reagent from PeBr (0.03 mol) gave $Bu_2PePhSn$ which was purified by column chromatography and then by distillation.

Table 1

Synthesised compounds: purity and analysis

Compound	Purity (%) by GLC-FID (GLC-FPD)	Sn analysis (Found (calc.) (%))
Me_3BuSn	> 99.5 (98.2)	53.2 (53.7)
Me_2Bu_2Sn	97.4 (98.1)	45.6 (45.2)
$MeBu_3Sn$	96.5 (96.4)	38.7 (38.9)
Bu_3PeSn	96.7 (94.5)	32.9 (32.9)
Bu_2Pe_2Sn	95.7 (> 99.5)	31.3 (31.7)
$BuPe_3Sn$	95.6 (97.9)	30.6 (30.5)
Pe_4Sn	97.0 (99.1)	29.5 (29.5)
Me_2Pe_2Sn	> 99.5 (> 99.5)	40.8 (40.8)
$Me_2BuPeSn$	96.1 (96.5)	42.6 (42.9)
$MeBu_2PeSn$	97.6 (> 99.5)	37.2 (37.3)
$MeBuPe_2Sn$	94.0 (93.8)	35.6 (35.2)
$Me_2BuPhSn$	96.2 (99.1)	41.6 (42.0)
$Bu_2PePhSn$	93.4 (97.5)	31.7 (31.2)
$BuPe_2PhSn$	93.4 (92.8)	29.6 (30.0
Pe_2Ph_2Sn	99.2 (97.7)	28.5 (28.6)

Reaction of $Bu_2PePhSn$ (0.010 mol) in CCl_4 with iodine (1 equiv.) gave Bu_2PeSnI (b.p. 172–174 °C/12 torr; D_4^{20} 1.52; n_D^{25} 1.5345; Anal. Found: Sn, 27.9; calc: 27.6%). Redistilled Bu_2PeSnI (0.075 mol) was treated with the Grignard reagent, made from MeI (0.01 mol) in Et_2O, to give $MeBu_2PeSn$, which was purified by fractional distillation under reduced pressure.

Preparation of butylmethyldipentyltin from diphenyltin dichloride

The compound Pe_2Ph_2Sn was obtained from Ph_2SnCl_2 (0.03 mol) in Et_2O and the reagent made from PeBr (0.07 mol); purification was achieved by column chromatography on Fluorisil. Treatment of Pe_2Ph_2Sn (0.025 mol) in hexane with iodine (1 equiv.), initially at room temperature and then under reflux, gave Pe_2PhSnI (b.p. 190–200 °C/12 torr). Reaction of Pe_2PhSnI (0.020 mol) in Et_2O with BuMgBr made from BuBr (0.025 mol) provided $Pe_2SnPhBu$, which was purified by column chromatography on Fluorisil, and then by fractional distillation under reduced pressure. Iodine and $BuPe_2PhSn$ (both 0.021 mol) in hexane at room temperature gave $BuPe_2SnI$ (b.p. 177–180 °C/12 torr); $BuPe_2SnI$ (0.0075 mol) was treated with MeMgI made from MeI (0.01 mol) in Et_2O to give $MeBuPe_2Sn$, which was column chromatographed and then fractionally distilled under reduced pressure.

The purity of all compounds were checked by both GLC-FID and GLC-FPD. Analytical and purity data of the prepared tetraorganotin compounds are listed in Table 1. Mass spectral data are given in Table 2. NMR spectral and other physical data are given in Tables 3 and 4.

Results and discussion

Series of $Me_{4-n}Sn$ and $Bu_nPe_{4-n}Sn$ were prepared by appropriate alkylations of Bu_nSnCl_{4-n} [3] with Grignard reagents. For other tetra-organotin compounds, use was made of sequences involving both Grignard alkylations of organotin halides

(*Continued on p. 73*)

Table 2

Physical data [a]

Compound (M)	B.p. (°C/torr) (lit. value)	n_D^{25} (lit. value)	D_4^{20} (lit. value)	MS (20 eV) m/z (%)
Me_4Sn (180)	74–75/760 (76/760)	1.4390 (1.4386)	1.29 (1.2995)	165(100, Me_3Sn); 150(25, Me_2Sn); 135(28, MeSn); 120(14, Sn)
Me_3BuSn (222)	44–46/12 (42–43/12)	1.4535 (1.4553)	1.18 (1.1830)	207(58, Me_2BuSn); 192(4, MeBuSn); 177(2, Busn); 121(13, SnH); 165(100, Me_3Sn); 151(96, Me_2SnH); 135(32, MeSn)
Me_2Bu_2Sn (264)	50–52/12 (70/4.4)	1.4630 (1.4640)	1.12 (1.124) (D_4^{25})	249(7, $MeBu_2Sn$); 207(63, Me_2BuSn); 193(18, MeBuSnH); 177 (3, BuSn); 151(100, Me_2SnH); 135(33, MeSn); 121(13, SnH)
$MeBu_3Sn$ (306)	124–126/12 (122–124/12)	1.4700 (1.4680)	1.09 (1.0898)	291(2, Bu_3Sn); 249(65, $MeBu_2Sn$); 234(2, Bu_2Sn); 207(30), 193(100, MeBuSnH); 177(15, BuSn); 151(40); 135(55, MeSn); 121(23, SnH)
Bu_4Sn (348)	147–149/12 (147–152/12)	1.4735 (1.4730)	1.05 (1.0572)	291(66, Bu_3Sn); 235(62, Bu_2SnH); 179(100, $BuSnH_2$); 121(37, SnH)
Bu_3PeSn (362)	157–159/12	1.4745	1.04	305(62, Bu_2PeSn); 291(21, Bu_3Sn); 249 (42, BuPeSnH); 235(54, Bu_2SnH); 193(32, $PeSnH_2$); 179(100, $BuSnH_2$); 121(40, SnH)
Bu_2Pe_2Sn (376)	169–172/12	1.4750	1.06	319(62, $BuPe_2Sn$); 305(52, Bu_2PeSn); 264(10); 249(55, BuPeSnH); 235(17, Bu_2SnH); 179(85, $BuSnH_2$); 121(90, SnH)
$BuPe_3Sn$ (390)	173–175/12	1.4770	1.06	333(23, Pe_3Sn); 319(62, $BuPe_2Sn$); 263(32, Pe_2SnH); 249(37, BuPeSnH); 193(64, $PeSnH_2$); 179(30, $BuSnH_2$); 121(50, SnH)

Pe_4Sn (404)	192–194/12 (191/13)	1.4765 (1.4720) (n_D^{20})	1.01 (1.0159)	333(74, Pe_3Sn); 263(88, Pe_2SnH); 193(100, $PeSnH_2$); 121(24, SnH)
Me_2Pe_2Sn (292)	119–121/12 (68–70/1)	1.4665 (1.4676) (n_D^{20})	1.09 (1.098)	277(8, $MePe_2Sn$); 221(64, Me_2PeSn); 207(23, MePeSnH); 191(4, PeSn); 165(5); 151(100, Me_2SnH); 135(33, MeSn); 121(12, SnH)
$Me_2BuPeSn$ (278)	105–107/12	1.4715	1.20	263(12, MeBuPeSn); 221(36, Me_2PeSn); 207(37, Me_2BuSn); 193(10, MeBuSnH); 151(100, Me_2SnH); 135(35, MeSn); 121(10, SnH)
$MeBu_2PeSn$ (320)	136–138/12	1.4760	1.15	305(4, Bu_2PeSn); 263(62, MeBuPeSn); 249(32, $MeBu_2Sn$); 207(52, MePeSnH); 193(100, MeBuSnH); 177(141, BuSn); 135(34, MeSn); 121(10, SnH)
$MeBuPe_2Sn$ (334)	154–156/12	1.4740	1.13	319(6, $BuPe_2Sn$); 277(42, $MePe_2Sn$); 263(76, MeBuPeSn); 207(100, MePeSnH); 193(74, MeBuSnH); 177(15, BuSn); 135(24, MeSn); 121(37, SnH)
$Bu_2PePhSn$ (382)	184–186/12	1.5075	1.23	325(59, BuPePhSn); 311(28, Bu_2PhSn); 305(12, Bu_2PeSn); 269(15, PePhSnH); 255(32, BuPhSnH); 235(6, Bu_2SnH); 197(100, PhSn); 179(16, $BuSnH_2$); 177(16, BuSn); 121(24, SnH)
$BuPe_2PhSn$ (396)	190–192/12	1.5105	1.30	339(30, Pe_2PhSn); 325(57, BuPePhSn); 319 (5, Bu_2PeSn); 269(24, PePhSnH); 255(16, BuPhSnH); 197(100, PhSn); 179(6, $BuSnH_2$); 177(6, BuSn); 121(10, SnH)
Pe_2Ph_2Sn (416)	235–240/12	1.5520	1.35	345(94, $PePh_2Sn$); 339(4, Pe_2PhSn); 275(100, Ph_2SnH); 197(100, PhSn); 121(30, SnH)

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[a] Ref. 2.

Table 3

^{13}C NMR (20 MHz $CDCl_3$) data (δ in ppm, J in Hz)

Compound $Me_nBu_{4-n}Sn$	$\delta(J(^{119}Sn-^1H))$				
	Me	Bu			
		C_α	C_β	C_γ	C_δ
Me_4Sn	−9.40				
	(336)				
Me_3BuSn	−10.37	10.87	29.02	27.05	13.72
	(315)	(315)	(20)	(50)	(0)
Me_2Bu_2Sn	−11.50	10.22	29.14	27.20	13.72
	(315)	(315)	(21)	(52)	(0)
$MeBu_3Sn$	−12.75	9.56	29.33	27.34	13.74
	(315)	(315)	(20)	(51)	(0)
Bu_4Sn		8.92	29.51	27.63	13.78
		(313)	(20)	(50)	(0)

Others	Me	Bu					Pe				
		Ph	C_α	C_β	C_γ	C_δ	C_α	C_β	C_γ	C_δ	C_ϵ
Me_2Pe_2Sn	−11.44	–	–	–	–	–	10.46	26.48	36.44	22.35	14.08
							(315)	(20)	(50)	(0)	(0)
$Me_2BuPeSn$	−11.44	–	10.31	13.75	27.23	29.19	10.49	26.54	36.50	22.39	14.06
$MeBu_2PeSn$	−12.72	–	9.59	13.73	27.32	29.27	9.77	26.61	36.59	22.33	14.04
			(315)	(20)	(50)	(0)	(315)	(20)	(50)	(0)	(0)

MeBuPe$_2$Sn	−12.71	–	9.60	13.75	27.27	29.21	9.76 (315)	26.53 (20)	36.55 (50)	22.28 (0)	14.02 (0)
Me$_2$BuPhSn	−10.72	147.1 136.1 128.1	10.92	13.67	27.07	28.92	–	–	–	–	–
Bu$_2$PePhSn	–	142.0 136.6 128.0	9.65 (315)	13.69 (20)	27.45 (50)	29.22 (0)	9.63 (315)	26.57 (20)	36.71 (50)	22.31 (0)	14.03 (0)
BuPe$_2$PhSn	–	142.0 136.5 128.0	9.91 (315)	13.69 (20)	27.45 (50)	29.22 (0)	9.91 (315)	26.57 (20)	36.71 (50)	22.30 (0)	14.05 (0)
Pe$_2$Ph$_2$Sn	–	140.5 136.8 128.2	–	–	–	–	10.56 (320)	26.38 (20)	36.55 (50)	22.18 (0)	13.94 (0)

Bu$_n$Pe$_{4-n}$Sn	Bu					Pe			
	C_α	C_β	C_γ	C_δ	C_α	C_β	C_γ	C_δ	C_ϵ
Bu$_3$PeSn	8.92 (310)	29.43 (20)	27.52 (50)	13.79 (0)	9.14	26.75	36.82	22.39	14.15
Bu$_2$PeSn$_2$	8.96 (310)	29.52 (20)	27.62	13.79	9.15 (314)	26.84 (20)	36.92 (48)	22.47 (0)	14.13 (0)
BuPe$_3$Sn	8.96 (310)	29.47 (20)	27.58 (50)	13.78 (0)	9.18 (310)	26.81 (20)	36.86 (48)	22.43 (00	14.11 (0)
Pe$_4$Sn					9.16 (311)	27.78 (20)	36.82 (48)	22.37 (0)	14.09 (0)

Table 4

^{1}H NMR (220 MHz $CDCl_3$) spectral data (δ in ppm, J in Hz)

Compound	δ ($J(^{119}Sn-^1H)$)										
	Me	Ph	Bu				Pe				
			H_α	H_β	H_γ	H_δ	H_α	H_β	H_γ	H_δ	H_ϵ
Me_4Sn	0.07 (53)										
Me_3BuSn	0.04 (51)		0.88 (48)	1.48	1.27	0.88					
Me_2Bu_2Sn	0.00 (51)		0.80 (48)	1.47	1.28	0.87					
$MeBu_3Sn$	−0.04 (51)		0.80 (48)	1.47	1.28	0.84					
Bu_4Sn			0.75 (48)	1.44 (63)	1.26	0.84					
$Bu_nPe_{4-n}Sn$ ($n = 0-3$)			0.76 (48)	1.45	1.26	0.86	0.76 (48)	1.46	1.26	1.26	0.86
Pe_4Sn							0.75 (48)	1.46	1.26	1.26	0.85
Me_2Pe_2Sn	0.00 (51)						0.80 (48)	1.50	1.28	1.28	0.87
$Me_2BuPeSn$	0.02 (51)		0.82 (48)	1.50	1.29	0.89	0.82 (48)	1.50	1.29	1.29	0.89
$MeBu_2PeSn$	−0.02 (51)		0.82 (48)	1.49	1.31	0.90	0.82	1.49	1.31	1.31	0.90
$MeBuPe_2Sn$	+0.04 (51)		0.79	1.49	1.27	0.87	0.79	1.47	1.27	1.27	0.87
$Me_2BuPhSn$	0.28 (53)	7.50(*o*) 7.35(*m*, *p*)	1.07 (53)	1.56	1.35	0.80					
$Bu_2PePhSn$		7.43(*o*) 7.28(*m*, *p*)	1.01 (52)	1.50	1.27	0.85	1.01 (52)	1.50	1.27	1.27	0.85
$BuPe_2PhSn$		7.45(*o*) 7.29(*m*, *p*)	1.03 (50)	1.54	1.28	0.85	1.03 (50)	1.54	1.28	1.28	0.85
Pe_2Ph_2Sn		7.45(*o*) 7.30(*m*, *p*)					1.26 (50)	1.60	1.28	1.28	0.86

and iodine cleavage of phenyl–tin bonds [4] as illustrated by the syntheses of $Me_2BuPeSn$, $MeBu_2PeSn$ and $MeBuPe_2Sn$ depicted in Schemes 1–3.

$$\begin{tikzcd}
Me_2SnCl_2 \arrow[r, "(i)"] & Me_2Ph_2Sn \arrow[r, "(ii)"] & Me_2PhSnI \arrow[d, "(iii)"] \\
Me_2BuPeSn & Me_2BuSnI \arrow[l, "(iv)"'] & Me_2BuPhSn \arrow[l, "(iii)"']
\end{tikzcd}$$

Scheme 1. Reagents: (i) 2PhMgI, Et_2O: (ii) I_2, hexane; (iii) BuMgBr, Et_2O; (iv) PeMgBr, Et_2O.

$$\begin{tikzcd}
Bu_2SnCl_2 \arrow[r, "(i)"] & Bu_2Ph_2Sn \arrow[r, "(ii)"] & Bu_2PhSnI \arrow[d, "(iii)"] \\
MeBu_2PeSn & Bu_2PeSnI \arrow[l, "(v)"'] & Bu_2PePhSn \arrow[l, "(iv)"']
\end{tikzcd}$$

Scheme 2. Reagents: (i) 2PhMgI, Et_2O; (ii) I_2, hexane; (iii) PeMgBr, Et_2O; (iv) I_2, CCl_4; (v) MeMgI, Et_2O.

$$\begin{tikzcd}
Ph_2SnCl_2 \arrow[r, "(i)"] & Pe_2Ph_2Sn \arrow[r, "(ii)"] & Pe_2PhSnI \arrow[d, "(iii)"] \\
MeBuPe_2Sn & BuPe_2SnI \arrow[l, "(iv)"'] & BuPe_2PhSn \arrow[l, "(ii)"']
\end{tikzcd}$$

Scheme 3. (i) PeMgBr, Et_2O; (ii) I_2, hexane; (iii) BuMgBr, Et_2O; (iv) MeMgI, Et_2O.

Products were purified by column chromatography on Fluorisil and/or fractional distillation under reduced pressure. The purity of each product was checked by both GLC-FID and GLC-FPD and was usually $> 95\%$. The GLC retention time data will be reported separately.

The mass spectra at 20 eV of the products clearly showed that scrambling of organic groups had not occurred in the preparation (or during the MS) of mixed alkyl compounds, $R_nR^1_{4-n}Sn$ or $R_2R^1R^2Sn$. In no case was the molecular ion observed in the mass spectrum, the heaviest mass ions always being triorganotin ions. For mixed tetraorganotin species, $R_nR^1_{4-n}Sn$ and $R_2R^1R^2Sn$, all possible triorganotin ions were observed. It was established that the facility to lose an organic group to give a triorganotin ion was in the sequence $Pe \simeq Bu > Me \simeq Ph$; the loss of Pe and Bu groups is almost equally easy in the compounds containing both groups. From the intensities of the various trialkyltin ions, and taking into account statistical factors, the relative ease of losing the alkyl groups Me, Bu, and Pe in the mass spectra of $Me_2BuPeSn$, $MeBu_2PeSn$ and $MeBuPe_2Sn$ can be seen to be 6/36/37, 4/31/32, and 6/42/38 respectively. Kochi and co-workers quote the relative ease of loss of Me and Bu groups to be 1/8 [6], in keeping with our data. From the mass spectra of $BuPe_2PhSn$ and $Bu_2PePhSn$, similar considerations indicate the relative ease of loss of Bu, Pe, and Ph to be 30/29/5 and 29/28/12 respectively. For $Me_2BuPhSn$, the relative ease of loss of Me, Bu, and Ph is estimated to be 8/100/8.

In addition to triorganotin ions, other ions observed in the mass spectra were di- and mono-organotin ions arising from further loss of alkyl groups. Furthermore, for butyl- and pentyl-containing tetraorganotins, hydride ions, R_2SnH^+ (or RR^1SnH^+) and $RSnH_2{}^+$ (by loss of an alkene fragment) were also observed.

In the ^1H NMR spectra of tetraalkyltins in $CDCl_3$ at 220 MHz, it was possible to distinguish chemical shifts for H_α, H_β, H_γ and H_δ of the butyl-tin group (viz. δ: 0.75–0.88; 1.44–1.50; 1.26–1.31 and 0.84–0.90) respectively and for H_α, H_β, $(H_\gamma + H_\delta)$ and H_ϵ of the pentyl-tin groups (viz. δ: 0.76–0.82; 1.46–1.50; 1.26–1.31 and 0.85–0.90). For phenyltrialkyltins and diphenylalkyltins, $\delta(H_\alpha)$ values (for both butyl and pentyl groups) shift to 1.03–1.07 and 1.26 respectively. Coupling constants, $J(^{119}Sn-^1H)$ were ca. 51 Hz for Me-Sn and ca. 48 Hz for Bu-Sn and Pe-Sn in tetraalkyltins. Phenyl substitution slightly increases these J values (by 2 Hz).

In the ^{13}C NMR (20 MHz) spectra in $CDCl_3$, it can be seen that for the series $Me_nBu_{4-n}Sn$, $\delta(Me)$ and $\delta(C_\alpha)$ of the butyl group are increasingly shifted to lower field as n increases.

For the series, $Bu_nPe_{4-n}Sn$, there is a consistency in the chemical shift values for C_α, C_β, C_γ and C_δ for Bu (8.94, 29.47, 27.58 and 13.78), respectively, and for C_α, C_β, C_γ, C_δ and C_ϵ for Pe (9.16, 26.78, 38.86, 22.39 and 14.11, respectively). The coupling constants $J(^{119}Sn-^1H)$ were ca. 315, 20, 50 and 0 for ^1J, ^2J, ^3J , and ^4J respectively. Another observation was that successive phenyl substitution for alkyl groups in the tetraorganotins led to progressive shifts to higher field for both $\delta(C_\alpha)$ (of butyl and pentyl groups) and $\delta(Me)$.

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