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Synthesis and Spectral Characterization of Unsymmetric Organotin(IV) Derivatives

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Unsymmetric Organotin(IV) compounds of general formula $R_{4-n}SnR'_n$ have been synthesized by the reaction of corresponding organotin(IV) chlorides with *n*-hexyl magnesium bromide prepared in dry ether in situ, where $R = CH_3, n-C_4H_9, C_6H_5, C_6H_5CH_2$ and $R' = n-C_6H_{13}$. The reactions were carried out in dry toluene, refluxed for 6 h and the final products were purified through vacuum distillation. These compounds were characterized by multinuclear NMR ($^1H, ^{13}C, ^{119}Sn$), IR spectroscopy and mass spectrometry to predict their structures. All spectroscopic results support the four-coordination and tetrahedral geometry of the compounds.

Key Words: Unsymmetric organotin(IV) compounds, multinuclear NMR, tetrahedral organotin(IV) compounds.

Introduction

The organometallic compounds in general and organotin compounds in particular are of great importance due to their industrial and biological uses¹⁻⁵. This is partly due to their unique physical, chemical and biocidal properties favourable to the environment⁶. Organotin reagents and intermediates are also used in organotin/organic synthesis⁷. In continuation of our previous interest in the synthesis and characterization of organotin compounds⁸, in the present work we report on the synthesis of unsymmetric tetraorganotin(IV) compounds with the general formula $R_{4-n}SnR'_n$ where $R = CH_3, n-C_4H_9, C_6H_5, C_6H_5CH_2, R' = n-C_6H_{13}$ and $n = 1$ or 2 , by the reaction of *n*-hexylmagnesium bromide (Grignard reagent) with corresponding organotin(IV) chlorides. These unsymmetric organotin(IV) compounds can be used for the synthesis of various unsymmetric organotin(IV) chlorides of industrial and medicinal importance⁹. All the synthesized compounds were characterized by IR, multinuclear NMR ($^1H, ^{13}C, ^{119}Sn$) spectroscopy, and mass spectrometry to elucidate their structures.

Experimental

Chemicals

Organotin chlorides were purchased from Aldrich Chemicals (USA), toluene and ether from E. Merck Chemicals (Germany) and 1-Bromohexane was used from Alfa Aesar Chemicals (Germany). Solvents were dried in situ, using standard methods¹⁰. All the chemicals were of analytical grade and used without further purification. Dibenzyltin dichloride and tribenzyltin chloride were prepared by reported methods¹¹.

Instrumentation

Infrared spectra were recorded as neat liquids on a Perkin Elmer FT-IR Model (spectrum 1000) spectrometer. The multinuclear NMR (¹H, ¹³C, ¹¹⁹Sn) spectra were recorded on a Bruker ARX 250 NMR, using CDCl₃ and C₆D₆ as internal references. The mass spectrometric analyses were carried out on a MAT-8500 Finnigan (Germany) mass spectrometer. The m/z values were evaluated assuming that H = 1, C = 12 and Sn = 120.

General Procedure for Synthesis

To a solution of corresponding organotin(IV) chloride in toluene was added an appropriate amount of n-hexylmagnesium bromide, prepared in situ, dropwise at 0°C over a period of 30 min under inert atmosphere (details are given in Table 1). The reaction mixture was refluxed for 6 h and then cooled to room temperature. It was hydrolysed with 7% aqueous hydrochloric acid solution while placed in an ice bath. The organic layer was separated and dried overnight on anhydrous sodium sulphate. After filtration, the solvent was evaporated on a rotary evaporator. The product so obtained was purified via vacuum distillation.

Table 1. Experimental details^{1,2}

Compd. No.	General formula	R	*Molar ratio	Mg (g)	1-Bromohexane (ml)
I	R ₂ SnR' ₂	C ₄ H ₉	1:2	0.770	4.62
II	R ₃ SnR'	C ₄ H ₉	1:1	0.374	2.30
III	R ₂ SnR' ₂	CH ₃	1:2	1.104	6.60
IV	R ₃ SnR'	CH ₃	1:1	0.610	3.60
V	R ₂ SnR' ₂	C ₆ H ₅	1:2	0.730	4.30
VI	R ₃ SnR'	C ₆ H ₅	1:1	0.194	1.45
VII	R ₂ SnR' ₂	C ₆ H ₅ .CH ₂	1:2	0.528	3.20
VIII	R ₃ SnR'	C ₆ H ₅ .CH ₂	1:1	0.300	1.72

¹Molar ratio order, organotin(IV) chloride: 1-bromohexane

²R' = n-C₆H₁₃

Results and Discussion

Unsymmetric organotin(IV) compounds of the general formula R_{4-n}SnR'_n were prepared in dry toluene by reaction of di- and triorganotin chlorides Grignard reagent (R'MgX where R' = n-C₆H₁₃) in 1:2 and 1:1 molar ratio, respectively. Most of the compounds are liquids with only a few exceptions. They are air stable and miscible with the common organic solvents.

IR Spectroscopy

The IR spectra of all compounds were scanned in the range 4000-250 cm^{-1} . The characteristic absorption bands are listed in Table 2. The important absorption frequencies are $\nu(\text{Sn-C})$ stretching, $\nu(\text{C-H})$ stretching in CH_2 , CH_3 , $\nu(\text{Sn-Ph})$, $\nu(\text{C=C})$ bending in ring (in aryl derivatives), and $\nu(\text{C-H})$ bending in CH_2 , CH_3 which provide conclusive evidence for the formation of an Sn-C bond.

The assignment of different vibration bands were made by the comparison of spectra with precursors. The vibration band due to $\nu(\text{Sn-C})$ stretching usually lies in the range 625-475 cm^{-1} for tetrahedral tetraorganotin(IV) compounds¹². The absorption frequencies in the range 608-595 cm^{-1} and 534-500 cm^{-1} are assigned as $\nu_{\text{asym}}(\text{Sn-C})$ and $\nu_{\text{sym}}(\text{Sn-C})$ stretching, respectively. The absence of a $\nu(\text{Sn-Cl})$ band at ~ 333 cm^{-1} in the synthesized compounds indicates the formation of an Sn-C bond. Furthermore, the presence of $\nu_{\text{asym}}(\text{C-H})$ and $\nu_{\text{sym}}(\text{C-H})$ in CH_2 , $\nu(\text{C-H})$ bending in CH_2 , CH_3 also support the existence of an $n\text{-C}_6\text{H}_{13}$ group in these compounds.

NMR Spectroscopy

Multinuclear NMR spectra were carried out in CDCl_3 or C_6D_6 . The expected resonances were assigned on the basis of their intensity and multiplicity pattern as well as their coupling constants. The peak integrations of the spectra were in accordance with the number of protons proposed for each fragment of the molecule. The aromatic carbon resonances were assigned by comparing experimental chemical shifts with those calculated by the incremental method and literature values¹³.

^1H NMR

For compounds I and II, methylene (CH_2) protons directly attached to tin (Sn-CH_2) and signals of CH_3 protons appeared as complexed multiplets in the range 0.66-0.91 ppm, all of which makes it difficult to resolve the individual protons. The long chain alkyl group ($n\text{-C}_6\text{H}_{13}$), due to their electronic and steric interaction, may initiate this complexity¹⁴ while multiplets at 1.20-1.52 ppm are assigned to other CH_2 protons (Table 3).

In compounds III and IV the analogous methylene protons (Sn-CH_2) exhibit almost the same behaviour and appear in the range 0.76-0.88 ppm. However, it is more pronounced in compound III than in IV because the former has one more highly electron releasing long chain $n\text{-C}_6\text{H}_{13}$ group. In compound III the protons of CH_3 attached to tin appeared as a sharp singlet at a high resonance frequency (-0.02 ppm) with nicely resolved 2J [$^{117/119}\text{Sn-}^1\text{H}$] couplings, which show the electropositive nature of tin and the effect of two $n\text{-C}_6\text{H}_{13}$ groups. While in compound IV the same protons are shifted slightly downfield (0.10 ppm), this may be again due to the occurrence of only one $n\text{-C}_6\text{H}_{13}$ group. The coupling constants 2J [$^{117/119}\text{Sn-}^1\text{H}$] observed for compound III and IV are given in Table 3 and fall in the range of the tetrahedral environment around the tin atom while coupling constants for Sn-CH_2 protons were not observed¹⁵.

Table 2. Infrared Spectral Data (cm^{-1}) for $\text{R}_2\text{SnR}'_2$ and $\text{R}_3\text{SnR}'$

Comp. No.	IR $\nu(\text{cm}^{-1})$										
	$\nu(\text{Sn-C})$ (sym)	$\nu(\text{Sn-C})$ (asym)	$\nu(\text{C-H})$ bending in C_6H_5	$\nu(\text{C-H})$ bending in C_6H_5	$\nu(\text{C-H})$ bending in CH_3	$\nu(\text{C-H})$ bending in CH_2	$\nu(\text{C=C})$ bending in ring (C_6H_5)	$\nu(\text{C-H})$ stretching in CH_2 (sym)	$\nu(\text{C-H})$ stretching in CH_2 (asym)	$\nu(\text{C-H})$ stretching in CH_3	$\nu(\text{C-H})$ stretching in C_6H_5
I	506(w)	595(w)	–	–	1377(w)	1461(m)	–	2853(sh)	2923(s)	2957(sh)	–
II	508(w)	595(w)	–	–	1376(m)	1460(s)	–	2856(sh)	2923(s)	2959(sh)	–
III	520(m)	597(w)	–	–	1377(w)	1460(m)	–	2855(sh)	2922(s)	2957(sh)	–
IV	523(m)	597(w)	–	–	1378(w)	1461(w)	–	2855(sh)	2924(s)	2958(sh)	–
V	500(w)	600(w)	698(s)	726(m)	1376(w)	1428(sh)	1460(m)	2854(sh)	2924(s)	2950(sh)	3062(sh)
VI	516(w)	602(w)	698(s)	729(s)	1377(w)	1429(s)	1460(sh)	2855(sh)	2925(s)	2951(sh)	3062(sh)
VII	531(w)	598(w)	687(sh)	717(m)	1407(s)	1455(w)	1546(m)	2856(sh)	2926(s)	3030(w)	3065(w)
VIII	534(w)	608(w)	699(m)	744(m)	1406(w)	1459(s)	1555(w)	2859(sh)	2926(s)	3027(sh)	3064(sh)

Note: m = medium, s = strong, sh = shoulder, w = weak

Table 3. ^1H NMR Data¹ for the Investigated Compounds (in CDCl_3)

Comp. No.	Chemical Shifts (ppm), Coupling Constants (Hz)
I	0.66-0.91 [m, 20H, 4SnCH ₂ , 4CH ₃]; $^2J[^{117/119}\text{Sn}-^1\text{H}] = (\text{n.o.})$; 1.20-1.34 [m, 8H, CH ₂]; 1.39-1.52 [m, 16H, CH ₂].
II	0.72-0.89 [m, 20H, Sn(CH ₂) ₄ , 4CH ₃]; $^2J[^{117/119}\text{Sn}-^1\text{H}] = (\text{n.o.})$; 1.17-1.31 [m, 8H, CH ₂]; 1.38-1.48 [m, 8H, CH ₂]; 1.52-1.65 [m, 4H, CH ₂].
III	-0.02 [s, 6H, Sn(CH ₃) ₂]; $^2J[^{117/119}\text{Sn}-^1\text{H}] = 48.5$ Hz, 50.4 Hz; 0.76-0.88 [m, 10H, 2SnCH ₂ , 2CH ₃]; 1.18-1.31 [m, 12H, CH ₂]; 1.41-1.50 [m, 4H, CH ₂].
IV	0.10 [s, 9H, Sn(CH ₃) ₂]; $^2J[^{119}\text{Sn}-^1\text{H}] = 51.1$ Hz; 0.75-0.94 [m, 5H, SnCH ₂ , CH ₃]; 1.33 [br.s, 6H, CH ₂]; 1.54-1.56 [m, 2H, CH ₂].
V	0.99-1.09 [m, 10H, 2SnCH ₂ , 2CH ₃]; $^2J[^{119}\text{Sn}-^1\text{H}] = (\text{n.o.})$; 1.39-1.69 [m, 12H, CH ₂]; 1.76-1.89 [m, 4H, CH ₂]; 7.42-7.51 [m, 6H, <i>o</i> , <i>p</i> /H]; 7.63-7.71 [m, 4H, <i>m</i> /H].
VI	1.03-1.19 [m, 5H, SnCH ₂ , CH ₃]; $^2J[^{119}\text{Sn}-^1\text{H}] = (\text{n.o.})$; 1.44-1.61 [m, 4H, CH ₂]; 1.69-1.81 [m, 2H, CH ₂]; 1.88-2.01 [m, 2H, CH ₂]; 7.54-7.63 [m, 9H, <i>o</i> , <i>p</i> /H]; 7.77-7.91 [m, 6H, <i>m</i> /H].
VII	0.97 [br, s, 6H, CH ₃]; 1.35-1.40 [m, 16H, 2Sn(CH ₂) ₄]; $^2J[^{119}\text{Sn}-^1\text{H}] = (\text{n.o.})$; 1.86 [br, s, 4H, CH ₂]; 2.37 [s, 4H, CH ₂ (Bz)]; $^2J[^{119}\text{Sn}-^1\text{H}] = (\text{n.o.})$; 7.14-7.90 [m, 10H, C ₆ H ₅].
VIII	0.94 [br, s, 3H, CH ₃]; 1.32-1.57 [m, 8H, Sn(CH ₂) ₄]; $^2J[^{119}\text{Sn}-^1\text{H}] = (\text{n.o.})$; 1.82 [br, s, 2H, CH ₂]; 2.37 [s, 6H, CH ₂ (Bz)]; $^2J[^{119}\text{Sn}-^1\text{H}] = (\text{n.o.})$; 7.21-7.86 [m, 15H, C ₆ H ₅].

¹s = singlet, m = multiplet, br = broad, (n.o) = not observed, Bz = benzyl

In compound V *ortho* and *para* protons were observed at 7.42-7.51 ppm and *meta* protons appeared at 7.63-7.71 ppm. However, in compound VI the same protons absorbed slightly downfield. In both derivatives Sn-CH₂ protons are also shifted to a lower field (1.00-1.20 ppm) compared to the other compounds. This might occur due to flow of valence electrons from tin towards the phenyl ring which deshielded the said protons. Other CH₂ protons also experience the same effect, as shown in the Table 3.

The behaviour of compounds VII and VIII to ^1H NMR was unusual. Thus, resonance peaks for protons of *n*-C₆H₁₃ group are difficult to differentiate and have been assigned ambiguously. However, methylene protons of the benzyl group in both compounds showed singlets at 2.37 ppm¹⁶. Aromatic protons in both compounds absorbed in the range 7.14-7.90 ppm while the couplings were not observed.

^{13}C NMR

The results obtained from ^{13}C NMR data coincide with that from ^1H NMR. The $\delta^{13}\text{C}$ resonances for R groups in R_{4-n}SnR'_n where R = alkyl or aryl, were observed in the regions as reported earlier⁸.

For *n*-C₆H₁₃, the individual carbons were assigned on the basis of their nJ [$^{119}\text{Sn}-^{13}\text{C}$] values and γ -effect^{17,18} (Table 4). The coupling constants, 1J [$^{117/119}\text{Sn}-^{13}\text{C}$] which are well observed in the spectra of compounds I-VI, lie in the range 300-500Hz and are consistent with the tetrahedral geometry around the tin atom¹⁹.

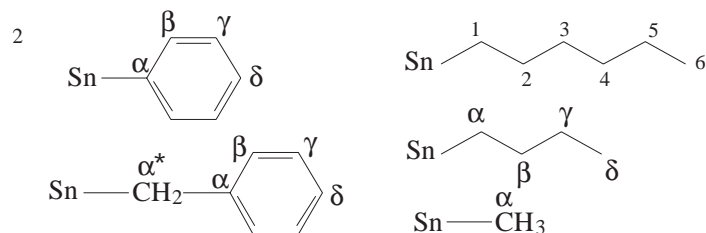
^{119}Sn NMR

The ^{119}Sn chemical shift data for investigated compounds are presented in Table 4 and are comparable with reported values. The chemical shift values of ^{119}Sn for four-coordinated tin compounds are considered to range from -44.7 to -122.0 ppm²⁰ and chemical shift data for compounds I-III and V-VIII are in agreement with these values.

Table 4. ^{13}C and ^{119}Sn NMR Data^{1,2} for the Investigated Compounds

Carbon ↓	I	II	III	IV	V	VI	VII	VIII
1	9.2, $^1J(299.7, 313.8)$	9.0, $^1J(299.9, 313.9)$	10.6, $^1J(333.5, 349.0)$	11.2, $^1J(351.8, 368.2)$	10.5, $^1J(350.1, 366.3)$	11.1, $^1J(379.5, 397.3)$	13.9, $^1J(\text{n.o.})$	10.9, $^1J(\text{n.o.})$
2	27.2, $^2J(19.6)$	27, $^2J(19.9)$	26.9, $^2J(20.6)$	26.8, $^2J(20.8)$	26.7, $^2J(20.0)$	26.5, $^2J(22.2)$	29.3	29.3
3	34.4, $^3J(49.2)$	34.2, $^3J(48.5)$	34.1, $^3J(51.7)$	33.9, $^3J(53.2, 50.9)$	34.0, $^3J(56.1)$	33.9, $^3J(58.4, 61.1)$	31.8	31.8
4	31.8	31.6	31.8	31.7	31.4	31.2	29.6	29.6
5	22.9	22.7	22.9	22.8	22.6	22.5	22.6	22.5
6	14.2	14.1	14.3	14.2	14.1	14.0	14.0	13.9
α	8.9, $^1J(299.3, 313.1)$	8.7, $^1J(299.4, 313.3)$	-11.4, $^1J(287.0, 300.1)$	-10.3, $^1J(303.5, 317.6)$	140.4, $^1J(415.9, 435.7)$	139, $^1J(458.3, 479.4)$	134.3	134.3
β	29.5, $^2J(19.5)$	29.3, $^2J(19.4)$	–	–	136.7, $^2J(32.7)$	136.9, $^2J(\text{n.o.})$	128.8	128.3
γ	27.6	27.5	–	–	128.2, $^3J(63.7)$	135.7, $^3J(46.0, 48.2)$	129.6	128.8
δ	13.8	13.7	–	–	128.3	136.0	128.3	128.2
α^*	–	–	–	–	–	–	29.5, $^1J(\text{n.o.})$	29.5, $^1J(\text{n.o.})$
$\delta^{119}\text{Sn}$	-12.0	-11.8	-1.9	0.01	-71.1	-98.9	-16.7	-23.9

¹Chemical shifts in ppm, ⁿJ[^{117/119}Sn–¹³C] in Hz; n.o. = not observed.



In non-coordinating solvent (CDCl_3) the ^{119}Sn characteristic chemical shift values, reported for four-coordinated tribenzyltin(IV) derivatives, occur in the range +55 to -25 ppm²¹. The observed ^{119}Sn chemical shift value of compound VIII covers nearly the same range. For four-coordinated tri- and dimethyltin(IV) compounds, the δ (^{119}Sn) ranges from +113 to -166 ppm²² while observed δ (^{119}Sn) for compounds III and IV are +0.01 ppm and -1.90 ppm, respectively, showing compatibility with the literature values. Thus, the four-coordinated nature of compounds (I-VIII) is reflected by ^{119}Sn chemical shift values (Table 4).

Mass Spectrometry

Mass spectral fragmentation patterns and relative abundance (%) are given in Tables 5 and 6. The molecular ion peaks, as reported earlier⁸, were not observed for some compounds whereas in some cases low intensity peaks were recorded.

Table 5. Fragmentation Pattern and Relative Abundance (%) of Common Ions Observed at 70 eV for $\text{R}_2\text{SnR}'_2$ Compounds

Fragment Ions	I	III	V	VII
$[\text{R}_2\text{Sn}(\text{CH}_2(\text{CH}_2)_4\text{CH}_3)_2]^+$	8	1	—	—
$[\text{R}_2\text{SnCH}_2(\text{CH}_2)_4\text{CH}_3]^+$	25	100	100	5
$[\text{RSn}(\text{CH}_2(\text{CH}_2)_4\text{CH}_3)_2]^+$	—	9	—	16
$[\text{SnCH}_2(\text{CH}_2)_4\text{CH}_3]^+$	—	3	—	10
$[\text{RSn}(\text{CH}_2(\text{CH}_2)_4\text{CH}_3)\text{H}]$	100	10	—	—
$[\text{R}_2\text{SnH}]^+$	28	70	53	—
$[\text{SnH}^+/\text{Sn}^+]$	8	3	4	4
$[\text{C}_6\text{H}_5\text{CH}_2]^+$	—	—	—	61
$[\text{CH}_3(\text{CH}_2)_4\text{CH}_2]^+$	3	1	4	100
$[\text{CH}_3(\text{CH}_2)_2\text{CH}_2]^+$	1	—	5	15
$[\text{CH}_3\text{CH}_2\text{CH}_2]^+$	3	—	16	90

Table 6. Fragmentation Pattern and Relative Abundance (%) of Common Ions Observed at 70 eV for $\text{R}_3\text{SnR}'$ Compounds

Fragment Ions	II	IV	VI	VIII
$[\text{R}_3\text{SnCH}_2(\text{CH}_2)_4\text{CH}_3]^+$	5	—	0.3	—
$[\text{R}_2\text{SnCH}_2(\text{CH}_2)_4\text{CH}_3]^+$	100	75	—	5
$[\text{R}_3\text{Sn}]^+$	31	—	100	9
$[\text{R}_2\text{Sn}]^+$	—	28	4	5
$[\text{RSn}]^+$	38	31	51	21
$[\text{R}_3\text{SnH}]^+$	—	3	33	—
$[\text{R}_2\text{SnH}]^+$	58	100	8	—
$[\text{SnH}^+/\text{Sn}^+]$	33	9	6	4
$[\text{C}_6\text{H}_5\text{CH}_2]^+$	—	—	—	100
$[\text{CH}_3(\text{CH}_2)_4\text{CH}_2]^+$	30	5	9	60
$[\text{CH}_3(\text{CH}_2)_2\text{CH}_2]^+$	91	10	10	53
$[\text{CH}_3\text{CH}_2\text{CH}_2]^+$	71	10	31	58

Compounds I and II exhibit almost the same behaviour. In both compounds primary fragmentation involves the loss of C_4H_9 or C_6H_{13} and secondary fragmentation may occur via the loss of C_6H_{12} or C_4H_8 ,

ending in the $\text{SnH}^+(121)$ fragment. Compound III adopts almost the same fragmentation pattern, but compound IV shows different fragmentation behaviour. For example, secondary and tertiary decompositions in compound III occur through the loss of C_6H_{12} and CH_4 respectively while in compound IV secondary decomposition may involve the loss of CH_3 or C_6H_{13} or CH_4 and tertiary fragmentation occurs via the loss of CH_4 or CH_3 . Finally, both end in the $\text{SnH}^+ (121)$ fragment.

Compounds V and VI show simple fragmentation patterns. In both compounds primary fragmentation occurs through the loss of C_6H_{13} and secondary fragmentation involves the loss of C_6H_{12} and C_6H_5 , respectively. In these compounds the last decomposition occurs through the elimination of the $\text{Sn}^+ (120)$ fragment. Compounds VII and VIII almost follow the same fragmentation pattern and give $\text{Sn}^+ (120)$ as a final fragment, as reported in Tables 5 and 6.

Conclusion

All spectroscopic analysis (multinuclear NMR, IR, and mass spectrometry) confirm the formation of the products. The results obtained are consistent with each other, which probably leads to the conclusion that synthesized compounds in non-coordinating solvents are monomeric with tetrahedral geometry.

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