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A novel mode of access to polyfunctional organotin compounds and their reactivity in Stille cross-coupling reaction

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ABSTRACT

Mono-, di-, tri- and tetra-functional organotin compounds were easily prepared in a sonicated Barbier reaction using ultrasound technology via coupling reaction of organo halides with tin halides (Bu_3SnCl, Bu_2SnCl_2, $BuSnCl_3$, $SnCl_4$) mediated by magnesium metal. The di- and tri-functional organotin compounds were tested in a Stille cross-coupling reaction in order to ascertain how many groups were transferred.

1. Introduction

The considerable advances in the use of organotin compounds as reagents or intermediates in organic synthesis in recent years have prompted the preparation of many new organotin compounds and the development of new rapid and convenient synthesis procedures [1]. The formation of a carbon–carbon bond by organotin compounds (especially tributylstannyl compounds) and electrophiles is of considerable interest. Palladium-catalysed cross-coupling of organotin compounds with organic electrophiles (Stille reaction) has emerged as one of the main methods for the creation of new carbon–carbon bonds [2]. Organotin compounds have been extensively accessed in organic synthesis and are typically prepared by the reaction of organometallic compound (RMgX, Rli, RZnX, etc.) which will react with organotin halides (R_nSnCl_{4-n}) or by reactions of stannylanions with organic halides [3]. Alkynyltin compounds are typically prepared by condensation of 1-alkynes with $R_3SnNR'_2$ or R_3SnOR' and also by trapping of an alkynyl metal with R_3SnX [4]. These methods allow synthesis of mono-, di-, tri- and tetra-functional organotin compounds, but in particular mono-functional tributylorganotin compounds have been synthesised with yields varying according to the nature of the substituent. We were therefore interested to synthesise various mono-, di-, tri- and tetra- functional organotin compounds by sonication in order to improve yields and the methodology and to create new organotin compounds according to our laboratory experience [5]. The increased focus on the use of sonochemical methods in organic synthesis has been demonstrated by the many reports in the literature on sonochemical reactions [6]. However few publications have reported the synthesis of mono-functional tributyltin compounds [7] and none have reported the synthesis of di-, tri- and tetra-functional organotin compounds with this technology.

2. Results and discussion

2.1. Preparation of mono-, di-, tri- and tetra-functional organotin compounds

We first tested a simplified and improved one-step synthesis of organotin compounds by Barbier reaction between stannyl chlorides, magnesium turnings and organic halides using ultrasound, without the presence of dibromoethane or iodine, as previously recommended for the synthesis of only mono-functional organotin compounds [7] (Scheme 1).

A wide range of organotin compounds were subjected to this procedure to produce quite high yields of the corresponding products. A clean multi-substitution (double, triple or quadruple) reaction was also possible with di-, tri- and tetrachlorotin derivatives and yielded polyfunctional organotin products. The results are presented in Table 1.

As can be seen, this methodology was able to accommodate a variety of organic functional halides and the yield was generally nearly quantitative, except in the case of tetra-functional tin compounds (entries 22–23) where experimental conditions needed to be optimized. In the case of aryl or benzyl halides a small amount of the duplicated product was obtained, except for entries 11 and

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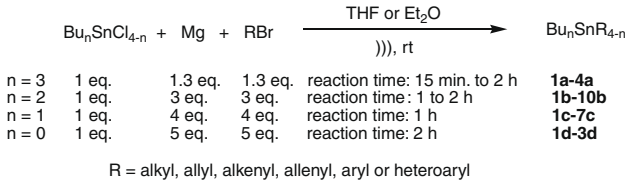


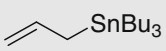
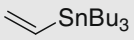
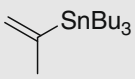
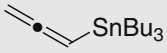
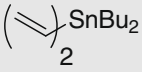
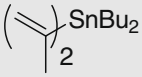
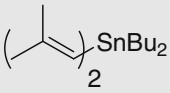
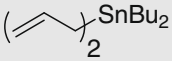
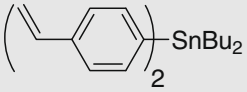
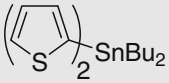
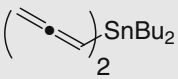
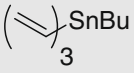
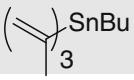
Scheme 1.

13. It should be noted that the quality of the reactants was not critical (solvent or halides), and they did not need careful purification before use, and dibromoethane or iodine were not necessary to start the reaction.

2.2. Specific preparation of 1-alkynyltin compounds

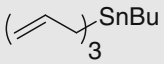
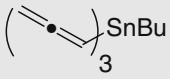
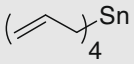
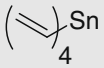
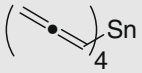
The preparation of 1-alkynyltin compounds with this methodology was not conclusive, and we therefore proceeded first with

Table 1
Preparation of mono-, di-, tri- and tetra-functional tin compounds.

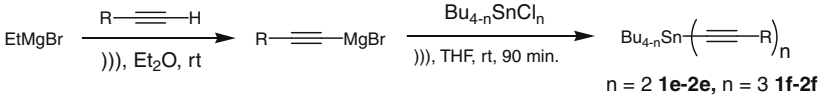
Entry	*n*	R	Product	Yield[a] (%)	Literature yield (%)/(ref)	No.
1	3	Allyl		91	Commercial 95/[29]	**1a**
2	3	Vinyl		89	Commercial 85/[3b]	**2a**
3	3	Isopropenyl		90	78/[11]	**3a**
4	3	Allenyl		78	Commercial 99/[12]	**4a**
5	2	Methyl	Me_2SnBu_2	68[b]	64/[3p]	**1b**
6	2	Vinyl		85	Commercial 91/[14]	**2b**
7	2	Isopropenyl		89	–	**3b**
8	2	Isobutenyl		45	–	**4b**
9	2	Allyl		79	Commercial 78/[3p]	**5b**
10	2	Phenyl	Ph_2SnBu_2	83	Commercial 78/[13]	**6b**[c]
11	2	*p*-Vinylphenyl		94	60/[15]	**7b**
12	2	Thienyl		58	[16]	**8b**
13	2	Benzyl	Bn_2SnBu_2	79[b]	81/[3o]	**9b**
14	2	Propargyl		77	[18]	**10b**
15	1	Ethyl	Et_3SnBu_2	88[b]	Commercial 85/[3p]	**1c**
16	1	Vinyl		73	[3b]	**2c**
17	1	Isopropenyl		77	–	**3c**

(*continued on next page*)

Table 1 (*continued*)

Entry	*n*	R	Product	Yield[a] (%)	Literature yield (%)/(ref)	No.
18	1	Allyl		82	90/[20a]	**4c**
19	1	Phenyl	Ph_3SnBu	63	Commercial [21]	**5c**[d]
20	1	Benzyl	Bn_3SnBu	87[b]	[22]	**6c**[e]
21	1	Propargyl		62	–	**7c**
22	0	Allyl		57	Commercial 66/[28]	**1d**
23	0	Vinyl		59	Commercial [3b]	**2d**
24	0	Allenyl		90	61/[23]	**3d**

[a] Estimated by ^{1}H NMR.
[b] Isolated yield.
[c] 10% of Ph_2 was observed.
[d] 5% of Ph_2 was observed.
[e] 6% of Bn_2 was observed.

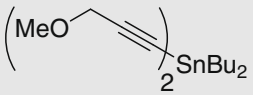
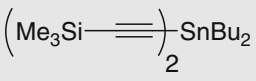
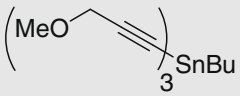
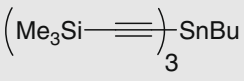


Scheme 2.

the sonicated preparation of EtMgBr, then synthesis of the magnesium acetylide by hydrogen–metal exchange and finally condensation of the latter with halogenotins using ultrasound (Scheme 2).

A clean double or triple-substitution reaction was possible and yielded di- or tri-functional tin products, which had never previously been described (entries 1–4). The results are presented in Table 2.

Table 2
Synthesis of 1-alkynyltin compounds.

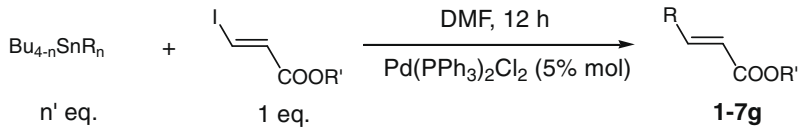
Entry	*n*	R	Product	Yield (%)	No.
1	2	Methoxymethyl		86[a] 45[b]	**1e**
2	2	Trimethylsilyl		92[a] 52[b]	**2e**
3	3	Methoxymethyl		82[a] 47[b]	**1f**
4	3	Trimethylsilyl		87[a] 55[b]	**2f**

[a] Estimated by ^{1}H NMR.
[b] Isolated yield.

2.3. Stille cross-coupling reaction with functional organotin compounds

In view of the surprising lack of literature reports on the reactivity of multi-functional organotin compounds, and as we obtained various di-, tri- and tetra-functional organotin compounds [8], we decided to use them in a Stille cross-coupling reaction in the presence of palladium complex catalysis [9], while monitoring the stoichiometry of the reaction in order to ascertain how many groups were transferred. This type of reaction has never previously been carried out to our knowledge. Just one example of allylation of carbonyl compounds demonstrated that a tetraallyltin can transfer several groups [10]. We therefore carried out the Stille cross-coupling reaction with different iodovinylic acids (or esters), first introducing a half or a third equivalent of di- or tri-functional organotin compounds and then adding the necessary quantity so that the reaction was quantitative (followed by ^{1}H NMR spectroscopy) (Scheme 3). The results are presented in Table 3.

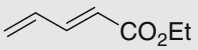
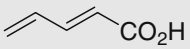
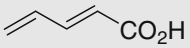
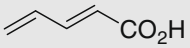
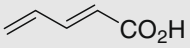
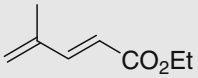
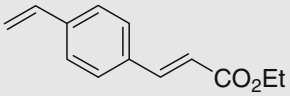
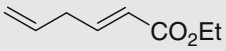
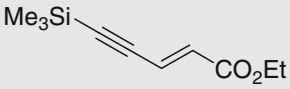
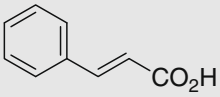
As can been seen, we transferred more than one group, but we could not prove that we had transferred two or three groups because we introduced a slight excess of organotin compounds so that the reaction was complete. As it is known that it is always necessary to use an excess of organotin compounds in a Stille reaction, in the case of the allylstannanes (entry 8) we must introduced a



R = alkenyl, aryl, alkynyl and allyl

Scheme 3.

Table 3
Reactivity of functional organotin compounds in a Stille cross-coupling reaction.

Entry	*n*	R	R′	*n*′, *T* °C	Product	Rdt (%)[a]	No.
1	2	Vinyl	Et	0.65, 25		81	**1g**
2	2	Vinyl	H	0.75, 25		86	**2g**
3	2	Vinyl	$SnBu_3$	0.6, 25		73[b]	**2g**
4	3	Vinyl	$SnBu_3$	0.5, 25		59[b]	**2g**
5	4	Vinyl	H	0.55, 30		40	**2g**
6	2	Isopropenyl	H	0.65, 70		74	**3g**
7	2	*p*-Vinylphenyl	Et	0.65, 70		89	**4g**
8[*]	2	Allyl	Et	1.2, 60		77	**5g**
9	2	Trimethylsilyl ethynyl	Et	0.65, 25		89	**6g**
10	3	Phenyl	H	0.55, 60		51	**7g**

[a] Isolated yield.
[b] Deprotected with HCl 1 M.
[*] $Pd(PPh_3)_4$ is used.

considerable excess of organotin compounds as already reported [10].

In conclusion, we have described here a new procedure allowing effective synthesis of various organotin compounds using ultrasound. The main advantages of this procedure are: (a) the quality of the reactants is not critical (solvents and tin halides) and they do not need careful purification before use (see Section 3), (b) the use of an additive is not necessary and (c) fair to good yields are obtained. The catalytic couplings utilizing part of these organotin compounds enabled us to transfer more than one or two groups, but using these di- and tri-functional organotin compounds made it possible to decrease the quantity of tin and thus the reactions were cleaner.

3. Experimental

^1H NMR spectra were recorded on a Bruker AC 200 (200 MHz) using $CDCl_3$ as solvent. The results, reported using the residual proton resonance of $CDCl_3$ (δ_H = 7.25 ppm) as the internal reference, were as follows (in order): chemical shift (d in ppm relative to Me_4Si), multiplicity (s, d, t, m, b for singlet, doublet, triplet, multiplet, broad), coupling constants (*J* in Hz). ^{13}C NMR spectra were recorded at 50.3 MHz on the same instrument using the $CDCl_3$ solvent peak at δ_C = 77.0 ppm as reference. Mass spectra were obtained on a Hewlett Packard (engine 5989A) in GC–MS (70 eV) mode. The isotopic patterns are given for ^{120}Sn (isotopic values 33%) in organotin fragments; this means that the reported values (values in brackets) for organotin fragments were only roughly a third of the correct value, taking into account the 10 isotopes of tin compared to those of the organic fragment. IR spectra were recorded on a Perkin–Elmer 781 Infrared Spectrophotometer or on a Perkin–Elmer Spectrum-One. Standard column chromatography was performed on Merk silica gel (60 Å, 230–400 mesh silica gel) or on neutral alumina. DMF was dried by distillation over calcium hydride prior to use. Tin halides (Bu_3SnCl, Bu_2SnCl_2, $BuSnCl_3$, $SnCl_4$) were commercially available and were used

without any purification. The compounds are commercially available as **1a**, **2a**, **4a**, **2b**, **5b**, **6b**, **1c**, **5c**, **1d**, **2d**, **1g**, **7g** or fully described as **3a** [11], **4a** [12], **1b** [13], **2b** [14], **4c** [20], **1d** [28], **3d** [23], **2g** [24], **3g** [25].

3.1. Preparation of mono-, di-, tri- and tetra-functional organotin compounds (entries 1–24)

A mixture of 0.49 g (0.02 at-gr) of magnesium turnings and 0.005 mol Bu_nSnCl_{4-n} was placed in a Schlenk tube, then covered with 5 mL of solvent (THF or Et_2O) to which a few drops of pure RX were added. The Schlenk tube was plunged into a commercial ultrasound cleaning bath (Branson B1200 E1, working frequency: 47 kHz, 300 W). When the reaction had started, the rest of the RX diluted in 15 mL of the solvent (C = 1.2 mol/L) was added. When the reaction was complete, the mixture was washed with a saturated solution of sodium chloride and extracted with diethyl ether. The organic layers were dried over magnesium sulfate, the solvents are removed under reduced pressure and the compound obtained was purified by column chromatography *on neutral alumina* (100% petroleum ether) or on silica gel (100% petroleum ether previously neutralised with triethylamine).

3.1.1. Di-n-butyldi-isopropenyltin (**3b**)

IR: 3040, 1634; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.94 (t, J = 7.2 Hz, 6H), 1.00–1.62 (m, 12H), 2.02 (dd, J = 1.4 Hz, J = 1.5 Hz, $^3J_{Sn-H}$ = 45 Hz, 6H), 5.17 (dq, J = 2.8 Hz, J = 1.4 Hz, 2H), 5.78 (dq, J = 2.8 Hz, J = 1.5 Hz, 2H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 9.9 ($^1J_{Sn-C}$ = 334–351 Hz, 2C), 14.1 (2C), 27.6 ($^2J_{Sn-C}$ = 47 Hz, 2C), 27.8 ($^3J_{Sn-C}$ = 57 Hz, 2C), 29.4 ($^2J_{Sn-C}$ = 21 Hz, 2C), 126.8 ($^2J_{Sn-C}$ = 30 Hz, 2C), 149.5 (2C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −74.3; MS (70 eV) *m/z*: organotin fragments 259 ($M^+-C_4H_9$, 56), 219 (11), 203 (100), 163 (41); 161 (66), 135 (15), 121 (30); organic fragments 41 (81), 39 (58).

3.1.2. Di-n-butyldi-isobutenyltin (**4b**)

IR: 3063, 1610; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.92 (t, J = 7.2 Hz, 6H), 1.29–1.58 (m, 12H), 1.82 (s, 6H), 1.93 (s, 6H), 5.51 (s, 2H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 11.7 ($^1J_{Sn-C}$ = 348–365 Hz, 2C), 14.1 (2C), 26.3 (2C), 27.7 ($^3J_{Sn-C}$ = 54 Hz, 2C), 29.1 (2C), 29.6 ($^2J_{Sn-C}$ = 20 Hz, 2C), 123.2 ($^1J_{Sn-C}$ = 430–450 Hz, 2C), 152 (2C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −109.4; MS (70 eV) *m/z*: organotin fragments 287 ($M^+-C_4H_9$, 54), 231 (100), 177 (20), 175 (36), 135 (40), 121 (13); organic fragments 67 (16), 55 (11), 53 (10), 41 (68), 39 (25).

3.1.3. Di-n-butyldi-p-vinylphenyltin (**7b**) [15]

IR: 3080, 3050, 1625, 1590, 1490; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.92 (t, J = 7.2 Hz, 6H), 1.28–1.69 (m, 12H), 5.29 (dd, J = 0.9 Hz, J = 10.8 Hz, 2H), 5.81 (dd, J = 0.9 Hz, J = 17.6 Hz, 2H), 6.75 (dd, J = 10.8 Hz, J = 17.6 Hz, 2H), 7.42 (d, J = 8.2 Hz, 2H), 7.50 (d, J = 8.2 Hz, 2H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 10.8 ($^1J_{Sn-C}$ = 353–369 Hz, 2C), 14.1 (2C), 27.8 ($^3J_{Sn-C}$ = 60 Hz, 2C), 29.4 ($^2J_{Sn-C}$ = 21 Hz, 2C), 114.4 (2C), 126.4 ($^3J_{Sn-C}$ = 45 Hz, 4C), 137.4 ($^2J_{Sn-C}$ = 33 Hz, 4C), 138.0 (2C), 140.7 (2C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −64.6; MS (70 eV) *m/z*: organotin fragments 383 ($M^+-C_4H_9$, 23), 327 (32), 223 (44), 197 (15), 121 (15); organic fragments 77 (16), 57 (14), 51 (14), 41 (100), 39 (27).

3.1.4. Di-n-butyldithien-2-yltin (**8b**) [16]

IR: 3075, 1640; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.99 (t, J = 7.2 Hz, 6H), 1.39–1.82 (m, 12H), 7.25–7.45 (m, 4H), 7.73–7.80 (m, 2H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 13.2 ($^1J_{Sn-C}$ = 394–412 Hz, 2C), 14.1 (2C), 27.6 ($^3J_{Sn-C}$ = 65 Hz, 2C), 29.1 ($^2J_{Sn-C}$ = 23 Hz, 2C), 128.5 ($^3J_{Sn-C}$ = 46 ppm, 2C), 131.8 ($^2J_{Sn-C}$ = 16 Hz, 2C), 134.7 (2C), 136.6 ($^3J_{Sn-C}$ = 31 Hz, 2C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −67.6; MS (70 eV) *m/z*: organotin fragments 343 ($M^+-C_4H_9$, 26), 287 (23), 203 (35), 121 (10); organic fragments 57 (20), 41 (100), 39 (35).

3.1.5. Dibenzyldi-n-butyltin (**9b**) [17]

IR: 3020, 1600, 1490; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.90 (t, J = 7 Hz, 6H), 1.22–1.47 (m, 12H), 2.36 (s, $^2J_{Sn-H}$ = 57 Hz, 4H), 6.97–7.08 (m, 6H), 7.20–7.27 (m, 4H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 10.3 ($^1J_{Sn-C}$ = 312–325 Hz, 2C), 14.2 (2C), 18.9 ($^1J_{Sn-C}$ = 236–246 Hz, 2C), 27.8 ($^3J_{Sn-C}$ = 55 Hz, 2C), 29.3 ($^2J_{Sn-C}$ = 20 Hz, 2C), 123.6 ($^5J_{Sn-C}$ = 13 Hz, 2C), 127.6 ($^3J_{Sn-C}$ = 22 Hz, 4C), 128.9 ($^4J_{Sn-C}$ = 11 Hz, 4C), 143.4 ($^2J_{Sn-C}$ = 35 Hz, 2C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −10.6; MS (70 eV) *m/z*: organotin fragments 416 (M^+, 2), 325 ($M^+-C_7H_7$, 87), 269 (48), 213 (58), 211 (100), 177 (20), 121 (14); organic fragments 91 (89), 65 (25), 39 (11).

3.1.6. Diallenyldi-n-butyltin (**10b**) [18]

IR: 3100, 1940; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.94 (t, J = 7 Hz, 6H), 1.17 (t, J = 8.1 Hz, 3H), 1.32–1.66 (m, 8H), 4.27 (d, J = 7.1 Hz, $^4J_{Sn-H}$ = 33–47 Hz, 4H), 5.07 (t, J = 7.1 Hz, $^2J_{Sn-H}$ = 29 Hz, 2H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 11.9 ($^1J_{Sn-C}$ = 370–387 Hz, 2C), 14.1 (2C), 27.3 ($^3J_{Sn-C}$ = 58 Hz, 2C), 28.9 ($^2J_{Sn-C}$ = 24 Hz, 2C), 64.7 ($^3J_{Sn-C}$ = 47 Hz, 2C), 74.6 ($^1J_{Sn-C}$ = 344–362 Hz, 2C), 210.9 (2C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −61.8; MS (70 eV) *m/z*: organotin fragments 273 ($M^+-C_3H_3$, 70), 255 ($M^+-C_4H_9$, 22), 234 (14), 217 (17), 177 (100), 161 (23), 159 (81), 121 (38); organic fragments 41 (37), 39 (30).

3.1.7. n-Butyltriethyltin (**1c**) [19]

IR: 2955, 2920, 2900, 2862; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.66-1.05 (m, 12H), 1.17–1.61 (m, 12H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 0.4 ($^1J_{Sn-C}$ = 305–319 Hz, 3C), 8.1 ($^1J_{Sn-C}$ = 300–314 Hz), 11.4 (3C), 14.5, 27.8 ($^3J_{Sn-C}$ = 50 Hz), 29.7 ($^2J_{Sn-C}$ = 20 Hz); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): 2.1; MS (70 eV) *m/z*: organotin fragments 235 ($M^+-C_4H_9$, 49), 207 (14), 183 (17), 179 (100), 151 (54), 149 (66), 121 (36); organic fragments 41 (24), 39 (12).

3.1.8. n-Butyltrivinyltin (**2c**)

IR: 3042, 1630; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.91 (t, J = 7.1 Hz, 3H), 1.05–1.63 (m, 6H), 5.76 (dd, J = 20 Hz, J = 3.8 Hz, $^3J_{Sn-H}$ = 81–85 Hz, 1H), 6.24 (dd, J = 13.8 Hz, J = 3.8 Hz, $^3J_{Sn-H}$ = 17.4–21.5 Hz, 1H), 6.46 (dd, J = 20 Hz, J = 13.8 Hz, $^2J_{Sn-H}$ = 65–70 Hz, 1H); $^{13C\ NMR\ \delta}$ ppm ($CDCl_3$, 50 MHz): 10.7 ($^1J_{Sn-C}$ = 391–409 Hz), 14.1, 27.4 ($^3J_{Sn-C}$ = 58 Hz), 29.1 ($^2J_{Sn-C}$ = 22 Hz), 135.7 (3C), 137.2 ($^1J_{Sn-C}$ = 445–466 Hz, 3C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −123.9; MS (70 eV) *m/z*: organotin fragments 201 ($M^+-C_4H_9$, 100), 175 (44), 149 (18), 147 (55), 121 (24); organic fragments 41 (65), 39 (32).

3.1.9. n-Butyltri-isopropenyltin (**3c**)

IR: 3038, 1633; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.94 (t, J = 7.1 Hz, 3H), 1.07–1.15 (m, 2H), 1.25–1.47 (m, 2H), 1.53–1.73 (m, 2H), 2.04 (t, J = 1.5 Hz, $^3J_{Sn-H}$ = 47 Hz, 9H), 5.22 (dq, J = 2.8 Hz, J = 1.5 Hz, $^3J_{Sn-H}$ = 69 Hz, 3H), 5.82 (dq, J = 2.8 Hz, J = 1.5 Hz, $^3J_{Sn-H}$ = 155 Hz, 3H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 10.3 ($^1J_{Sn-C}$ = 353–369 Hz), 14.1, 27.5 ($^2J_{Sn-C}$ = 49 Hz, 3C), 27.8, 29.3 ($^2J_{Sn-C}$ = 21 Hz), 127.7 ($^2J_{Sn-C}$ = 32 Hz, 3C), 148.6 ($^1J_{Sn-C}$ = 416–435 Hz, 3C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −110.4; MS (70 eV) *m/z*: organotin fragments 243 ($M^+-C_4H_9$, 60), 203 (48), 163 (19), 161 (50), 135 (17), 121 (27); organic fragments 41 (95), 39 (100).

3.1.10. n-Butyltriphenyltin (**5c**) [21]

IR: 3082, 1491; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.91 (t, J = 7.2 Hz, 3H), 1.30–1.79 (m, 6H), 7.37–7.44 (m, 9H), 7.55–7.60 (m, 6H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 11.3 ($^1J_{Sn-C}$ = 381–399 Hz), 14.1, 27.8 ($^3J_{Sn-C}$ = 64 Hz), 29.2 ($^2J_{Sn-C}$ =

22 Hz), 128.9 ($^3J_{Sn-C}$ = 47 Hz, 6C), 129.2 ($^4J_{Sn-C}$ = 11 Hz, 3C), 137.5 ($^2J_{Sn-C}$ = 35 Hz, 3C), 139.6 ($^1J_{Sn-C}$ = 460–482 Hz, 3C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −97.1; MS (70 eV) *m/z*: organotin fragments 351 ($M^+-C_4H_9$, 55), 197 (52), 120 (47); organic fragments 77 (38), 57 (21), 51 (72), 41 (100), 39 (36).

*3.1.11. Tribenzyl-n-butyltin (**6c**) [22]*

IR: 3078, 3020, 1600, 1495; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.84 (t, *J* = 7.1 Hz, 3H), 1.13–1.38 (m, 6H), 2.31 (s, $^2J_{Sn-H}$ = 58 Hz, 6H), 6.88–7.05 (m, 9H), 7.22 (td, *J* = 7.6 Hz, *J* = 1.5 Hz, 6H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 10.7 ($^1J_{Sn-C}$ = 329–340 Hz), 14.1, 19.2 ($^1J_{Sn-C}$ = 241–253 Hz, 3C), 27.7 ($^3J_{Sn-C}$ = 57 Hz), 29 ($^2J_{Sn-C}$ = 20 Hz), 123.9 ($^5J_{Sn-C}$ = 13 Hz, 3C), 127.8 ($^4J_{Sn-C}$ = 23 Hz, 6C), 129 ($^3J_{Sn-C}$ = 11 Hz, 6C), 142. 7 ($^2J_{Sn-C}$ = 35 Hz, 3C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −21.2; MS (70 eV) *m/z*: organotin fragments 359 (M^+−Bn, 6), 211 (54), 121 (6), 120 (13); organic fragments 91 (100), 65 (31), 39 (16).

*3.1.12. Triallenyl-n-butyltin (**7c**)*

IR: 3092; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.95 (t, *J* = 7.1 Hz, 3H), 1.30–1.71 (m, 6H), 4.36 (d, *J* = 7.1 Hz, $^4J_{Sn-H}$ = 44–46 Hz, 6H), 5.12 (t, *J* = 7.1 Hz, $^2J_{Sn-H}$ = 32 Hz, 3H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 11.9 ($^1J_{Sn-C}$ = 373–388 Hz), 14.1, 27.1 ($^3J_{Sn-C}$ = 61 Hz), 28.5 ($^2J_{Sn-C}$ = 27 Hz), 66 ($^3J_{Sn-C}$ = 54 Hz, 3C), 74.6 ($^1J_{Sn-C}$ = 413–433 Hz, 3C), 211.7 (3C); MS (70 eV) *m/z*: organotin fragments 255 ($M^+-C_3H_3$, 19), 159 (100), 121 (17); organic fragments 41 (57), 39 (100), 38 (22).

3.2. Preparation of 1-alkynyltin compounds (entries 1–4, Table 2)

A mixture of 2.18 g (0.02 mol) bromoethane, 0.49 g (0.02 at-gr) of magnesium turnings in 17 mL of anhydrous diethyl ether was placed in a dry round-bottomed three-necked flask (100 mL) equipped with a condenser and a dropping funnel flushed with argon. The round-bottomed flask were plunged into a commercial ultrasound cleaning bath (Branson B1200 E1, working frequency: 47 kHz, 300 W). When the magnesium had disappeared, 0.04 mol 1-alkynes diluted in anhydrous diethyl ether were added dropwise. When the reaction had finished, approximately 10 mL anhydrous THF was poured in and when the solution was quite homogeneous, 14 mmol Bu_3SnCl (or 7 mmol Bu_2SnCl_2) diluted in an equivalent volume of THF was added dropwise. When the reaction was complete, the mixture was washed with a saturated solution of sodium chloride and extracted with diethyl ether. The organic layers were dried over magnesium sulfate, solvents were removed under reduced pressure and the compound obtained was purified by column chromatography on neutral alumina (100% petroleum ether) or on silica gel (100% petroleum ether, previously neutralised with triethylamine).

*3.2.1. Di-n-butyl-bis(3-methoxypropynyl)tin (**1e**)*

IR: 2165; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.95 (t, *J* = 7.2 Hz, 6H), 1.19–1.72 (m, 12H), 3.42 (s, 6H), 4.15 (s, $^4J_{Sn-H}$ = 9.3 Hz); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 13.6 ($^1J_{Sn-C}$ = 464–485 Hz, 2C), 13.9 (2C), 26.8 ($^3J_{Sn-C}$ = 70 Hz, 2C), 28.6 ($^2J_{Sn-C}$ = 28 Hz, 2C), 58 (2C), 60.8 (2C), 87.1 ($^1J_{Sn-C}$ = 475–497 Hz, 2C), 106.5 ($^2J_{Sn-C}$ = 95 Hz, 2C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −146.2; MS (70 eV) *m/z*: organotin fragments 315 ($M^+-C_4H_9$, 18), 189 (13), 159 (22), 151 (37), 121 (8); organic fragments 69 (13), 57 (29), 45 (12), 41 (100), 39 (45).

*3.2.2. Di-n-butyl-bis(trimethylsilylethynyl)tin (**2e**)*

IR: 2152; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.21 (s, 18H), 0.95 (t, *J* = 7.2 Hz, 6H), 1.17–1.72 (m, 12H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 0.4 (6C), 13.9 ($^1J_{Sn-C}$ = 457–478 Hz, 2C), 14.0 (2C), 26.8 ($^3J_{Sn-C}$ = 67 Hz, 2C), 28.6 ($^2J_{Sn-C}$ = 27 Hz, 2C), 109.6 ($^1J_{Sn-C}$ = 442–461 Hz, 2C), 119.7 ($^2J_{Sn-C}$ = 67 Hz, 2C); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −158.9; MS (70 eV) *m/z*: organotin fragments 371 ($M^+-C_4H_9$, 44), 315 (10), 217 (39), 121 (10); organic fragments 97 (65), 83 (28), 73 (70), 57 (40), 45 (14), 43 (12), 41 (100), 39 (14).

*3.2.3. Tri-n-butyltrimethylsilylethynyltin (**1f**)*

IR: 2158; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.20 (s, 9H), 0.94 (t, *J* = 7.0 Hz, 9H), 1.31–1.65 (m, 18H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 0.6 (3C), 11.5 ($^1J_{Sn-C}$ = 363–380 Hz, 3C), 14.1 (3C), 27.3 ($^3J_{Sn-C}$ = 58 Hz, 3C), 29.2 ($^1J_{Sn-C}$ = 22 Hz, 3C), 113.4 ($^1J_{Sn-C}$ = 282–294 Hz), 119.1 ($^2J_{Sn-C}$ = 38 Hz); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −66.6; MS (70 eV) *m/z*: organotin fragments 331 ($M^+-C_4H_9$, 48), 275 (34), 219 (30), 217 (49), 203 (11), 121 (10); organic fragments 97 (58), 73 (22), 57 (35), 41 (100), 39 (19).

*3.2.4. Tri-n-butyl-3-methoxypropynyltin (**2f**)*

IR: 2160, 1105; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.93 (t, *J* = 7.1 Hz, 9H), 1.27–1.65 (m, 18H), 3.42 (s, 3H), 4.14 (s, $^4J_{Sn-H}$ = 8.5 Hz, 2H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 11.4 ($^1J_{Sn-C}$ = 366–383 Hz, 3C), 14.1 (3C), 27.4 ($^3J_{Sn-C}$ = 60 Hz, 3C), 29.3 ($^2J_{Sn-C}$ = 23 Hz, 3C), 57.6, 61.0, 90.3 ($^1J_{Sn-C}$ = 305–319 Hz), 106.0 ($^2J_{Sn-C}$ = 55 Hz); ^{119}Sn NMR δ ppm ($CDCl_3$, 149 MHz): −61.2; MS (70 eV) *m/z*: organotin fragments 303 ($M^+-C_4H_9$, 59), 247 (48), 191 (14), 189 (26), 159 (29), 121 (16); organic fragments 57 (20), 41 (100), 39 (35).

*3.3. Stille cross-coupling reaction with functional stannanes (entries **1**–**7g**)*

Dichlorobis(triphenylphosphine)palladium II (5 mol%) was added to an anhydrous DMF solution (2 mL) of acid or ester halide (1.5 mmol) in a Schlenk flask under argon, and *n'* mmol of di- or trifunctional organotin compounds were added after stirring for 15 min. The mixture was stirred for12 h at the recommended temperature. After cooling, the reaction mixture was filtered through a Celite path and then treated with a 1 M solution of potassium fluoride and ethylacetate to eliminate the tributyltin iodide thus formed. The aqueous layer was extracted with diethyl ether. The organic layer was washed with brine to eliminate the DMF and dried over $MgSO_4$. After evaporation of the solvents, the crude product was purified by column chromatography on silica gel (90/10: petroleum ether/diethyl ether) in the case of ethylic esters, and by an acid–base treatment for the acid and stannic esters, which were deprotected at the same time.

*3.3.1. (2E)-Ethyl-3-(p-vinylphenyl)-prop-2-enoate (**4g**) [25]*

IR: 3050, 3088, 1728, 1644, 1608, 1568, 1509; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 1.36 (t, *J* = 7.1 Hz, 3H), 4.28 (q, *J* = 7.1 Hz, 2H), 5.33 (dd, *J* = 10.9 Hz, *J* = 0.7 Hz, 1H), 5.82 (dd, *J* = 17.6 Hz, *J* = 0.7 Hz, 1H), 6.44 (d, *J* = 16 Hz, 1H), 6.73 (dd, *J* = 17.6 Hz, *J* = 10.9 Hz, 1H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.51 (d, *J* = 8.3 Hz, 2H), 7.68 (d, *J* = 16 Hz, 1H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 14.8, 60.9, 115.7, 118.4, 127.1 (2C), 128.7 (2C), 134.3, 136.5, 139.9, 144.5, 167.5; MS (70 eV) *m/z*: 202 (65), 174 (18), 173 (10), 158 (10), 157 (76), 130 (54), 129 (68), 128 (100), 127 (45), 115 (14), 103 (12), 102 (28), 77 (51), 76 (14), 75 (15), 64 (14), 63 (23), 51 (43), 50 (18), 43 (11), 39 (18).

*3.3.2. (2E)-Ethyl hexa-2,5-dienoate (**5g**) [26]*

IR: 3062, 3081, 1730, 1660, 1646; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 1.29 (t, *J* = 7.1 Hz, 3H), 2.95 (tq, *J* = 6.5 Hz, *J* = 1.5 Hz, 2H), 4.19 (q, *J* = 7.1 Hz, 2H), 5.05–5.16 (m, 2H), 5.76–5.92 (m, 2H), 6.98 (dt, *J* = 15.6 Hz, *J* = 6.5 Hz, 1H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 14.6, 36.5, 60.6, 117.6, 122.6, 134.3, 146.8, 166.9; MS (70 eV) *m/z*: 140 ($M^{\cdot+}$, 3), 97 (11), 95 (24), 68 (11), 67 (100), 66 (22), 65 (18), 41 (60), 40 (15), 39 (69).

*3.3.3. (2E)-Ethyl-5-trimethylsilyl-pent-2-en-4-ynoate (**6g**) [27]*

IR: 3320, 3071, 3082, 2125, 1729, 1621; ^{1}H NMR δ ppm ($CDCl_3$, 200 MHz): 0.23 (s, 9H), 1.31 (t, J = 7.1 Hz, 3H), 4.23 (q, J = 7.1 Hz, 2H), 6.26 (d, J = 16 Hz, 1H), 6.76 (d, J = 16 Hz, 1H); ^{13}C NMR δ ppm ($CDCl_3$, 50 MHz): 0.02 (3C), 14.6, 61.2, 101.7, 105.3, 125.3, 131.6, 166.2.

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