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Palladium-catalysed addition of tin–tin and tin–silicon bonds to allenes

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Abstract

Hexamethyl-, hexaethyl- and hexabutyl-ditin add to a variety of allenes in the presence of $Pd(PPh_3)_4$; the reaction is reversible. Trimethylsilyltrimethyl- and -tributyl-stannane also add under comparable conditions, the silyl moiety becoming attached to the central carbon atom of the allene skeleton; this reaction is, however, apparently not reversible.

Introduction

Palladium-catalysed reactions in which Si–Si [1], Si–Sn [2], Ge–Sn [3] and Sn–Sn [4] bonds undergo addition to alkynes have been described in the last few years. Other multiply-bonded systems have also been used: thus the α-addition of Si–Sn [5] bonds to isonitriles is known, and the Si–Si bond has also been shown to undergo addition to allenes [6]. We have published preliminary results on the addition of Me_6Sn_2 [7] and $Me_3GeSnBu_3$ [8] to allenes and also initial observations on the addition of $Me_3SiSnMe_3$ to 1,1-dimethylallene [9].

The present paper deals with extension of the ditin addition reactions to include hexaethyl- and hexabutyl-ditin and presents a detailed study of the reactions of Me_3SiSnR_3 ($R = Me$, Bu) with allenes. In view of the ready availability of allenes [10] and of the synthetic importance of organotin intermediates [11] such addition reactions, leading as they do to molecules containing two easily replaceable labile functional groups, should offer considerable scope for synthetic elaboration.

Results and discussion

1. Addition of R_6Sn_2 ($R = Me, Et, Bu$)

We have previously shown [7] that Me_6Sn_2 adds readily to allenes in the presence of $Pd(PPh_3)_4$, and that both kinetic and thermodynamic control of the reaction can be achieved.

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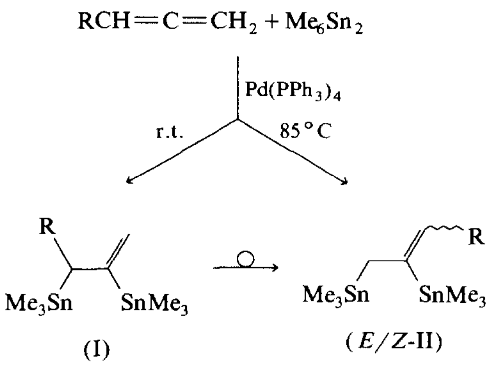
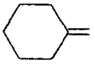
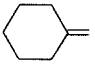
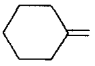


We have now extended the range of allenes used in our previous study [7] and in addition allowed several allenes to react with Et_6Sn_2 and Bu_6Sn_2. Table 1 lists

Table 1

Reaction conditions, yields and isomer distributions in additions to allenes $RCH{=}C{=}CH_2$ of ditins R'_6Sn_2

R	R′	Reaction conditions (°C/h)	Yield (%)	B.p. (°C/mmHg)	Isomer ratio I	E-II	Z-II
Me	Me	85/48	39	50–51/0.005	12	59	29
Bu	Me	75/24	92	88/0.06	3	82	15
Bu	Et	85/17	92	132–136/0.01		76	24
Bu	Bu	85/66	90			66	34
^{c}Hex	Me	90/24	75	dec.	5	55	40
^{c}Hex	Et	90/40	74		6	75	19
^{c}Hex	Bu	85/144	51		4	79	17
^{t}Bu	Me	85/48	51	73–74/0.001	93	6	1
^{t}Bu	Et	95/192	14		9	85	6
^{t}Bu	Bu	85/144	5			100	
	Me	80/8	77	80–90/0.005	3	97	
	Et	80/20	77	110–113/0.001		100	
	Bu	80/94	60			100	
Ph	Et	85/19	51	120–123/0.005		90	10
Ph	Bu	80/17	53			96	4
MeO	Et	85/20	80	107–110/0.005		48	52
MeO	Bu	75/96	89	172–176/0.001	3	65	32
EtO_2CCH_2	Me	80/20	72	102–110/0.01	7	69	24
EtO_2CCH_2	Et	80/20	80	107–110/0.005	8	78	14
EtO_2CCH_2	Bu	80/20	85		15	74	11
Me_3Sn	Me	85/48	80		75	25	
Me_3Sn	Et [a]	90/40	71				
Me_3Sn	Bu [a]	85/72	70				

[a] Because of the large number of products formed (see text) yields could not be determined.

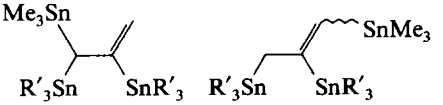
details of our present results in a comparative form: this is of interest both from a mechanistic point of view and because tributylstannyl groups are preferred for purely synthetic applications because of their lower toxicity.

It can be seen that in general the yields do not decrease on going from Me_6Sn_2 to Bu_6Sn_2, though the latter may require the use of longer reaction times. However, t-butylallene is an exception, the yield falling very significantly from 51% for Me_6Sn_2 to only 5% for Bu_6Sn_2: steric effects in the catalytic cycle are apparently important here.

The isomer ratios given in Table 1 reflect a situation at or near thermodynamic equilibrium: for lower reaction temperatures and/or shorter reaction times isomer I predominates, as we showed previously [7]. Since reactions of Et_6Sn_2 and Bu_6Sn_2 require more forcing conditions, kinetic control is in practice unattainable for their reactions when good yields are achieved. Distillation is not normally feasible for hexabutylditin adducts because of their decomposition at the high temperatures required.

The reaction between ditins and allenes is apparently reversible. As reported previously [12,13], 2,3-bis(trimethylstannyl)-1-propene decomposes on heating in the presence of palladium catalysts (e.g. with $Pd(PPh_3)_4$, 20% Me_6Sn_2 formed after 17 h at 80 °C; with $PhCH_2PdCl(PPh_3)_2$, 35% Me_6Sn_2 formed after 2.5 d at 70 °C); allene could not be detected.

The reactions of Et_6Sn_2 or Bu_6Sn_2 with $Me_3SnCH{=}C{=}CH_2$ provided further evidence for reversibility. Tin-119 NMR showed the formation of a very large number of products in each case, all of structures I or II. The initial adducts undergo cleavage to give either the starting materials or $Me_3SnSnR'_3$ and R'_3Sn-$CH{=}C{=}CH_2$. These then react in a nonregiospecific manner, forming the final mixture which contains all theoretically possible isomers.

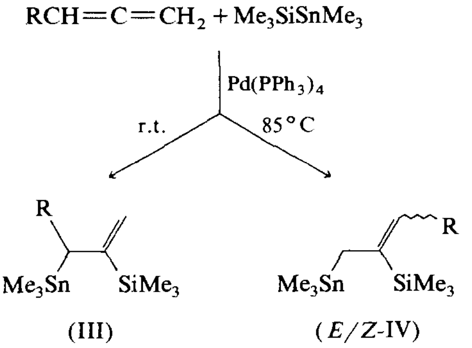


It was however somewhat surprising to find that heating of the cyclohexylallene/Me_6Sn_2 adduct with Et_6Sn_2 in the presence of $Pd(PPh_3)_4$ for several days at 80 °C did not lead to a rapid scrambling of the stannyl groups; nor did such a scrambling occur when the cyclohexylallene/Et_6Sn_2 adduct was heated with Me_6Sn_2. In both cases a very slow reaction was observed in which only a product of type I was formed in which both R_3Sn residues had been exchanged.

The reversibility does, unfortunately, have a consequence in the chemistry of ditin/allene adducts; attempts to use these in palladium-catalysed cross-coupling reactions (e.g. with allyl bromide) fail because the ditin reacts faster than I or II, causing the equilibrium to shift back towards the allene, which can in these cases be identified by IR and NMR spectroscopy. The sole organotin products are the corresponding allyltrialkyltin and trialkyltin bromide [13]. Ditin/allene adducts do not react with allyl bromide in the absence of a palladium catalyst [14].

2. *Addition of Me_3SiSnR_3 ($R = Me, Bu$)*

Our preliminary investigations [9] on addition of $Me_3SiSnMe_3$ to allenes indicated that the reaction occurs in a regiospecific manner:



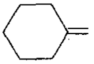
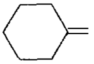
Such adducts again contain two labile functional groups with very different reactivities; because of their synthetic potential we decided to include the less toxic $Me_3SiSnBu_3$ in our investigations, and the results (again in a comparative form) are summarised in Table 2.

The formation of the kinetic product III and its isomerisation to *E/Z*-IV is documented by the different isomer ratios before and after distillation observed in several cases for reactions of both $Me_3SiSnMe_3$ and $Me_3SiSnBu_3$.

Reactions of both these silylstannanes with $Me_3SnCH{=}C{=}CH_2$ do not, however, lead to complex product mixtures; the main product is III, accompanied by only small amounts of by-products. Thus here the conversion of III to IV involves only a 1,3-allyl shift of a stannyl moiety. This points to a likely initial and irreversible

Table 2

Reaction conditions, yields and isomer distributions in additions to allenes $RCH{=}C{=}CH_2$ of silylstannanes $Me_3SiSnR'_3$ (R′ = Me, Bu)

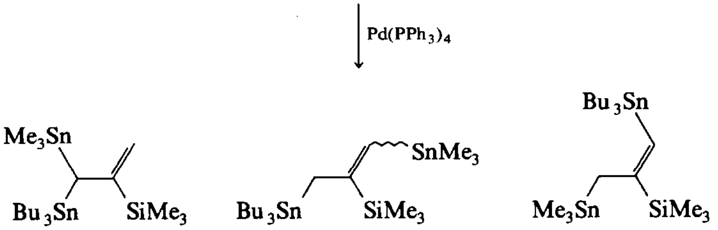
R	R′	Reaction conditions (°C/h)	Yield (%)	B.p. (°C/mmHg)	Isomer ratio [a]			
					III	*E*-IV		*Z*-IV
Bu	Me	85/1.5	70	68–72/1	41(85)	54(15)		5(0)
Bu	Bu	85/20	67	120–125/0.02		73(75)		27(23)
^{c}Hex	Me	85/1.5	70	84–90/0.9	90(90)	10(10)		
^{c}Hex	Bu	85/1.5	50	112–130/0.02	35(85)			65(15)
^{t}Bu	Me	85/36	38	61–64/0.9	95(95)	5(5)		
^{t}Bu	Bu	85/20	62	117–125/0.01	95(95)	5(5)		
Ph	Me	85/18	61	95–102/0.8	9(14)	53(54)		38(32)
Ph	Bu	85/1.25	63	154–162/0.01		90(95)		10(5)
MeO	Me	85/20	54	45/1 (dec.)		72(69)		28(31)
MeO	Bu	85/3.5	62	90–100/0.01	5(72)	20(12)		70(11)
EtO_2CCH_2	Me	85/1.5	71	85/0.8	31(62)	69(38)		
EtO_2CCH_2	Bu	85/4	62	145–155/0.01	93(90)	7(10)		
	Me	85/24	74	88–92/1	53(60)		47(40)	
	Bu	85/24	65	134–138/0.01			100(100)	
Me_3Sn	Me	85/20	64	75–83/0.01	89(95)	11(15)		
Me_3Sn	Bu [b]	85/20	38		76(76)			

[a] Isomer ratio yields in brackets determined after distillation. [b] 3 by-products observed (see text).

formation of the Si–C bond (the Me_3Si group, having a larger "effective size" than the R'_3Sn group because of the much shorter Si–C bond length, attacking the sterically most readily available carbon atom to give a σ- or π-allylpalladium intermediate) followed by a reversible formation of the Sn–C bond at the secondary carbon atom of the allene skeleton.

This picture is confirmed by the nature of the 3 by-products formed when $Me_3SnCH{=}C{=}CH_2$ is allowed to react with $Bu_3SnSiMe_3$:

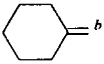
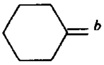
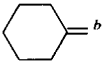
$$Me_3SiSnBu_3 + Me_3SnCH{=}C{=}CH_2$$



(III, 76%) (IV, minor) ("iso-IV", minor)

Table 3

Tin-119 NMR data for compounds of types I and II derived from allenes $RCH{=}C{=}CH_2$ and ditins R'_6Sn_2 [a]

R	R′	I			*E*-II			*Z*-II		
		$\delta(Sn_{all})$	$\delta(Sn_{vi})$	^3J [c]	$\delta(Sn_{all})$	$\delta(Sn_{vi})$	^3J [c]	$\delta(Sn_{all})$	$\delta(Sn_{vi})$	^3J [c]
Me	Me	1.4	−38.1	126	−7.8	−34.5	112	−6.3	−50.2	122
Bu	Me	−4.1	−38.8	138	−9.5	−34.7	110	−7.3	−50.7	118
Bu	Et				−11.9	−36.9	102	−9.8	−44.4	138
Bu	Bu				−21.7	−46.7	102	−18.8	−55.0	149
cHex	Me	−14.5	−38.0	151	−12.3	−34.0	110	−8.3	−50.7	119
cHex	Et	−18.7	−42.2	167	−13.9	−36.4	102	−10.0	−44.4	134
cHex	Bu				−23.7	−46.0	102	−19.4	−54.6	142
tBu	Me	−15.1	−32.8	332	−15.3	−28.6	98	−7.8	−49.0	114
tBu	Et				−19.7	−31.9	85			
tBu	Bu				−28.9	−41.1	85			
 [b]	Me	0.4	−39.1	97	−8.6	−47.9	117			
 [b]	Et				−11.2	−40.7	106			
 [b]	Bu				−21.1	−52.6	110			
Ph	Et				−9.2	−37.9	110	−5.9	−39.7	114
Ph	Bu	−13.7	−39.5	216	−19.3	−38.3	110	−15.1	−50.0	124
MeO	Et				−9.8	−24.5	157	−10.1	−34.7	147
MeO	Bu	−21.8	−34.6	179	−21.1	−35.3	157	−19.9	−46.1	155
EtO_2CCH_2	Me	2.6	−35.9	114	−7.9	−31.4	102	−3.8	−47.8	110
EtO_2CCH_2	Et	−7.6	−37.9	122	−10.9	−34.5	90	−6.9	−42.8	118
EtO_2CCH_2	Bu	−16.0	−48.0	122	−20.9	−44.6	94	−16.4	−54.2	126

[a] Chemical shifts in ppm w.r.t. Me_4Sn. [b] Product derived from cyclohexylidene allene. [c] $^3J(^{119}Sn, ^{119}Sn)$.

The fourth possible by-product is not observed.

In addition, heating of the methoxyallene/$Me_3SnSiMe_3$ adduct (as a mixture of III and IV in the presence of $Pd(PPh_3)_4$ with $Bu_3SnSiMe_3$ led to a very slow replacement of Me_3Sn by Bu_3Sn; only isomers of type *E*- and *Z*-IV were observed to have undergone exchange.

3. *Isomer identification by NMR spectroscopy*

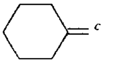
3.1. Ditin adducts. Each adduct is characterised by tin-119 NMR signals for the allylic and vinylic tins which have common coupling constants $^3J(^{119}Sn, ^{119}Sn)$ and $^3J(^{119}Sn, ^{117}Sn)$. The relevant parameter ranges are as follows:

Ditin used	$\delta(Sn_{all})$	$\delta(Sn_{vin})$	$^3J(Sn, Sn)$
Me_6Sn_2	$+5$ to -18	-31 to -51	80–330
Et_6Sn_2	-6 to -18	-25 to -44	85–160
Bu_6Sn_2	-14 to -29	-34 to -55	80–220

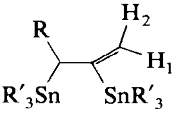
For the pair *E*/*Z*-II the vinylic tin absorbs at considerably higher field in the *Z* isomer, as expected.

Table 4

Vinyl proton chemical shifts and tin–proton coupling constants for compounds of types I and II derived from allenes $RCH{=}C{=}CH_2$ and ditins R'_6Sn_2 [a,d]

R	R′	I		*E*-II	*Z*-II
		$\delta(H_1)/^3J(Sn, H)$	$\delta(H_2)/^3J(Sn, H)$	$\delta(H)/^3J(Sn, H)$	$\delta(H)/^3J(Sn, H)$
Me	Me	4.93/75.0	5.43/158.1	5.40/76.8	5.90/146.3
Bu	Me	4.91/74.8	5.41/158.8	5.23/78.7	5.25/146.3
Bu	Et			5.15/67.6	5.86/141.1
Bu	Bu			5.13/71.5	5.83/141.7
^{c}Hex	Me	4.94/74.8	5.40/160.1	5.04/78.7	5.63/149.6
^{c}Hex	Et	4.95/70.2	5.47/154.2	4.99/70.2	5.68/136.4
^{c}Hex	Bu			4.98/70.9	5.65/135.1
^{t}Bu	Me	5.19/76.5	5.56/168.4		
^{t}Bu	Et	5.20/ [b]	5.56/ [b]	5.02/80.7	
^{t}Bu	Bu			5.01/82.1	
 [c]	Me	5.04/80.7	5.40/171.3		
Ph	Et			5.21/72.8	6.21/ [b]
Ph	Bu	5.31/61.7	5.71/139.1	6.21/72.8	6.20/155.5
MeO	Et			5.43/32.2	6,34/96.5
MeO	Bu	6.68/50.5	6.84/107.6	5.41/32.2	6.30/95.2
EtO_2CCH_2	Me	4.95/73.5	5.38/158.2	5.41/71.5	5.89/140.4
EtO_2CCH_2	Et			5.33/65.6	5.95/ [b]
EtO_2CCH_2	Bu	4.93/66.9	5.47/145.7	5.33/63.7	

[a] Chemical shifts in ppm w.r.t. TMS, coupling constants $^3J(^{119}SnC{=}CH)$ in Hz. [b] Not determined. [c] Product derived from cyclohexylidene allene. [d] Proton labelling in I as follows:



The low value of ^3J(Sn, Sn) is due to hyperconjugation between the π-system and the allylic Sn–C bond [13].

Isomer identification can also be readily based on the proton NMR spectrum: compounds I contain two vinylic protons as an AB system with values of ^3J(Sn–C=C–H) corresponding to the *cis* (65–78) and *trans* (140–168 Hz) geometries. Compounds II have only one vinylic proton, the coupling of which to tin establishes the isomer geometry.

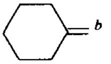
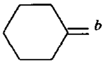
The relevant data are listed in Tables 3 (^{119}Sn) and 4 (^{1}H).

3.2. Silylstannane adducts. The regiochemistry of the addition follows from the tin-119 chemical shift typical of an allyltin moiety (Me_3Sn: +9 to −16, Bu_3Sn: 0 to −23 ppm). The distinction between III and IV follows again from the vinyl proton data: either two (AB) or one vinyl proton is observed.

A further criterion for assigning isomers III and IV is found from a more detailed study: the value of ^3J(Sn, Si) is normally considerably larger for III than for IV. When both *E* and *Z*-IV are present the assignment is simple, as the vinylic silicon nucleus absorbs to higher field in the *Z* than in the *E* isomer. When only one isomer of IV is formed, a detailed comparison of the complete data permits a decision as to which it is, the *E* isomer invariably being identified in such cases.

Table 5

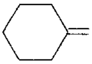
Tin-119 and silicon-29 NMR data for compounds of types III and IV derived from allenes $RCH=C=CH_2$ and silylstannanes $Me_3SiSnR'_3$ (R′ = Me, Bu) [a]

R	R′	III			*E*-IV			*Z*-IV		
		$\delta(Sn_{all})$	$\delta(Si_{vi})$	^3J [c]	$\delta(Sn_{all})$	$\delta(Si_{vi})$	^3J [c]	$\delta(Sn_{all})$	$\delta(Si_{vi})$	^3J
Bu	Me	2.9	−5.6	20	−7.6	−5.5	13	−4.0	−8.0	14
Bu	Bu				−21.2	−5.4	12	−18.5	−9.9	15
^{c}Hex	Me	−4.8	−5.7	23	−9.8	−5.3	14			
^{c}Hex	Bu	−22.5	−5.6	22	−22.2	−5.2	12			
^{t}Bu	Me	−10.7	−4.6	36	−12.0	−3.6	11			
^{t}Bu	Bu	−23.0	−4.4	36	−16.1	−3.5	10			
 [b]	Me	1.9	−7.8	12	−6.1	−5.4	10			
 [b]	Bu				−20.1	−7.7	12			
Ph	Me	6.1	−3.4	47	−4.2	−2.7	15	0.9	−6.6	14
Ph	Bu				−17.2	−2.6	13	−13.3	−6.7	15
MeO	Me	−16.0	−6.2	20	−6.8	−6.2	21	−6.1	−6.9	14
MeO	Bu	−35.6	−6.4	11	−19.3	−5.9	18	−19.1	−6.7	16
EtO_2CCH_2	Me	8.7	−5.2	16	−5.6	−4.7	12			
EtO_2CCH_2	Bu	−12.6	−5.0	16	−19.6	−4.5	11			
Me_3Sn	Me [d]	15.2	−6.0	28	−11.4	−7.3	11			
Me_3Sn	Bu [e]	−0.1	−5.8	27						

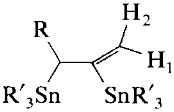
[a] Tin-119 chemical shifts w.r.t. Me_4Sn, silicon-29 shifts w.r.t. TMS. [b] Product derived from cyclohexylidene allene. [c] $^3J(^{119}Sn, ^{29}Si)$ in Hz. [d] III: $^2J(^{119}Sn, ^{119}Sn)$ 273 Hz, *E*-IV: δ(R) −69.3 ppm, $^3J(^{119}Sn, ^{29}Si)$ 100, $^4J(^{119}Sn, ^{119}Sn)$ 151 Hz. [e] δ(R) 13.1 ppm, $^3J(^{119}Sn, ^{29}Si)$ 15, $^2J(^{119}Sn, ^{119}Sn)$ 260 Hz.

Table 6

Vinyl proton chemical shifts and tin–proton coupling constants for compounds of types III and IV derived from allenes $RCH{=}C{=}CH_2$ and silylstannanes $Me_3SiSnR'_3$ [a,d]

R	R′	(III)		*E*-IV	*Z*-IV
		$\delta(H_1)/^4J(Sn, H)$	$\delta(H_2)/^4J(Sn, H)$	$\delta(H)/^4J(Sn, H)$	$\delta(H)/^4J(Sn, H)$
Bu	Me	5.27/25.0	5.36/21.7	5.44/26.3	
Bu	Bu			5.41/23.6	5.80/21.7
[c]Hex	Me	5.22/25.6	5.36/22.3	5.25/25.0	
[c]Hex	Bu	5.28/22.3	5.36/19.7		
[t]Bu	Me	5.47/22.3	5.55/17.1	5.36/27.6	
[t]Bu	Bu	5.46/17.7	5.52/15.1		
 [b]	Me	5.30/24.9	5.36/25.6		
Ph	Me	5.58/12.5	5.63/11.8	6.44/24.3	
Ph	Bu			6.41/22.3	
MeO	Me	5.22/23.6	5.55/23.0	5.75/24.3	
MeO	Bu	5.25/19.0	5.57/18.4	5.72/21.7	6.24/19.7
EtO_2CCH_2	Me	5.22/23.6	5.32/21.0	5.62/25.6	
EtO_2CCH_2	Bu	5.22/20.5	5.37/19.7	5.60/ [c]	

[a] Chemical shifts in ppm w.r.t. TMS, coupling constants $^4J(SnCC{=}CH)$ in Hz. [b] Product derived from cyclohexylidene allenes. [c] Not determined. [d] Proton labelling in III as follows:



Selected NMR data for compounds of type III and IV are given in Tables 5 (^{119}Sn, ^{29}Si) and 6 (^1H).

Experimental

All manipulations involving organotin compounds were carried out under argon. Allenes were prepared by published procedures [10]. NMR spectra were recorded on a Bruker AM-300 spectrometer (solutions in $CDCl_3$, standards TMS and Me_4Sn). GLC analyses were carried out with Carlo Erba HRGC-5300 and HRGC-4160 instruments, with either 25m CP-SIL-5 CB or 30m DB1-(5)-CB quartz columns. New compounds were also characterised by elemental analysis (C, H).

The general procedure used for addition was as follows: equimolar amounts (10 mmol) of the allene and the organotin reagent were mixed and ca. 0.1 mmol $Pd(PPh_3)_4$ added. The mixture was heated for 0.5–1 h at 40–60 ° C, after which the proton NMR spectrum was recorded in order to check which products were formed initially. The mixture was then subjected to the conditions given in Tables 1 and 2; after the stated time no further reagent consumption was noted. Water was added and the organic materials extracted with diethyl ether, the ether layer dried over $MgSO_4$, the ether removed, and the residue subjected to vacuum distillation (except when two tributyltin residues are present, in which case distillation leads to decomposition). GLC and multinuclear NMR analyses were then carried out: in some cases (see Table 2) comparative GLC analyses were carried out before and after distillation.

In cases where the product distribution varied throughout the reaction, the latter was repeated and interrupted after a short time in order to permit the unequivocal characterisation of the kinetic products.

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