

PALLADIUM CATALYSIS IN ORGANOTIN CHEMISTRY: ADDITION OF HEXAALKYLDITINS TO ALKYNES

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(Received October 21st, 1985)

Summary

Tetrakis(triphenylphosphine)palladium(0) catalyses the *cis*-addition of hexaalkylditins R_6Sn_2 ($R = Me, Et, Bu$) to acetylene and 1-alkynes; for $R = Bu$, however, the reaction is not quantitative. With very few exceptions there is no reaction in the case of non-terminal alkynes. The (*Z*)-distannylalkenes can be partially or completely isomerised to the (*E*)-alkenes by UV irradiation. The 1,2-distannyl alkenes were characterised by NMR spectroscopy. Experimental details are given for the preparation of ditins R_6Sn_2 from tin hydrides R_3SnH in the presence of tetrakis(triphenylphosphine)palladium(0).

Introduction

The use of vinyl derivatives of organometallic compounds in organic synthesis is common; of particular interest are vinylsilanes [1], vinylboranes [2] and vinylalanes [3], all of which can be readily prepared, either from alkynes or by other routes.

Vinyltin compounds are also of considerable interest, as sources of vinyl groups in substitution reactions [4] and of vinyl anions (or anionoids) [5]. They can be readily prepared by hydrostannation of alkynes [6], and other methods for their preparation are available [7]. The addition of metal(loid)–metal(loid) bonds to alkynes has also been studied in recent years; if carried out regio- and stereospecifically it can yield further useful building blocks for organic synthesis. Among the systems involving elements of Group IV described in the literature are silicon–silicon [8], silicon–copper [9], silicon–magnesium, –zinc and –aluminium [10] and tin–copper [11]: such reactions usually proceed with the help of transition metal catalysts. Some examples of tin–tin additions are also known; thus Cullen et al. [12] added Me_6Sn_2 photochemically to a fluorinated alkyne, while Bulten and Budding [13] carried out the additions under polar conditions. Sherping [14] and Vorspohl [15] showed that ditins $Me_4Sn_2X_2$ ($X = \text{halogen}$) add photochemically [14] and thermally [15], but not in the presence of palladium catalysts [15]. Depending on the

reactants and conditions, *Z*- or *E*-products are formed. We now report in detail on the palladium-catalysed addition of ditins to alkynes; our initial work has been outlined in a preliminary communication [16].

Results and discussion

(a) Addition of hexamethylditin

The reaction proceeds according to eq. 1.



Details of the reactions carried out and of the products obtained are given in Table 1.

The catalyst used was in each case tetrakis(triphenylphosphine)palladium(0) (referred to below as PdL_4); for $\text{R} = \text{H}$ and *c*-Hex we have observed that palladium bis(acetylacetonate) can also function as a catalyst, but we made no systematic studies of the suitability of other catalysts. Except with acetylene itself under certain conditions (see below) only the *Z*-isomer is formed. We were not successful in extending the reaction to non-terminal alkynes except for $\text{MeOCH}_2\text{C}\equiv\text{CCH}_2\text{OMe}$

TABLE 1

FORMATION OF $(\text{Z})\text{-Me}_3\text{SnC(R)}=\text{CHSnMe}_3$: REACTION CONDITIONS, YIELDS OF ISOLATED PRODUCTS, AND BOILING POINTS (*c*-Hex = cyclohexyl; *i*-Hex = $\text{Me}_2\text{CHCH}_2\text{CH}_2\text{CH}_2$; *i*-Hex-3-en = $\text{Me}_2\text{C}=\text{CHCH}_2\text{CH}_2$)

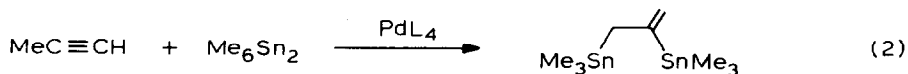
Compound	R	Reaction conditions (°C/h)	Yield (% (g))	B.p. (°C/mmHg)
1	H	60/3.5	87 (17.7)	90/14
2	n-Bu	85/100	29 (1.1)	100/0.001
3	<i>c</i> -Hex	80/20	20 ^c	^d
4	Ph	80/72	67 (28.9)	93/0.001
5	PhCH_2	85/63	43 (2.1)	100/0.001
6	HOCH_2 ^a	80/24	59 (4.2)	92/0.03
7	MeOCH_2	75/42	87 (36.4)	71/0.03
8	PhOCH_2	65/6	90 ^c	^d
9	Me_2NCH_2	70/65	43 (4.5)	68/0.001
10	HOCH_2CH_2 ^b	80/20	10 ^c	^d
11	$\text{HOCH}(\text{n-Pr})$ ^a	80/20	70 (14.9)	120/0.01
12	HOCMe_2	80/65	77 (15.8)	70/0.01
13	$\text{HOC}(\text{Me})\text{Et}$	80/20	75 (6.2)	95/0.2
14	$\text{HOC}(\text{cyclo-C}_6\text{H}_{10})$	85/16	81 (11.2)	135/0.1
15	$\text{HOC}(\text{Me})\text{i-Hex}$	80/72	83 (20.0)	85/0.01
16	$\text{HOC}(\text{Me})\text{i-Hex-3-en}$	80/20	78 (10.9)	120/0.02
17	CO_2Me	50/2	73 (4.3)	80/0.05
18	CO_2Et	25/24	82 (8.7)	90/0.06
19	MeOCH_2 ^e	75/44	48 (4.6)	99/0.01

^a For by-products see text. ^b After addition of toluene to give a homogeneous mixture. ^c From proton NMR spectrum. ^d Not distilled. ^e Product from $\text{MeOCH}_2\text{C}\equiv\text{CCH}_2\text{OMe}$.

and 1,4-diphenylbutadiyne; no reaction was observed with $t\text{-BuC}\equiv\text{CH}$, $\text{Me}_3\text{SiC}\equiv\text{CH}$, or $\text{Me}_3\text{SnC}\equiv\text{CH}$. No solvent is necessary when liquid alkynes are used, but in the case of acetylene itself the reaction is not reproducible when no solvent is used, large quantities of the *E*-isomer often being obtained. We have found use of THF (or dioxane) and a reaction temperature of 65°C to be suitable in this case; less than 5% of the *E*-isomer is then present (for details see Experimental).

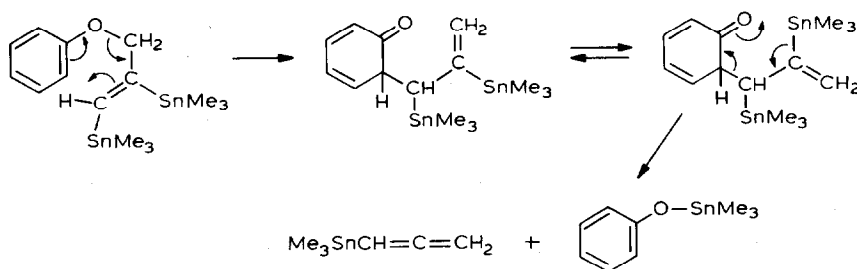
Propiolic acid only undergoes polymerisation, while methyl propiolate reacts normally. Propargyl bromide and propargyl amine do not undergo addition, while *N,N*-dimethylpropargylamine again reacts normally.

Two further acetylenes also showed abnormal behaviour. Propyne yielded an unexpected product, 2,3-bis(trimethylstannyl)-1-propene (eq. 2).

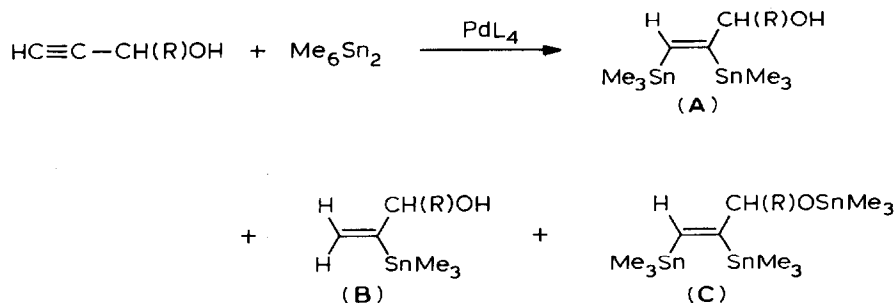


As later work has shown [16], this is the product of addition of the ditin to allene; the addition to allenes is a general reaction, and we must assume that under the reaction conditions used propyne is isomerised to allene.

Phenyl propargyl ether reacted normally at temperatures below 80°C , but at higher temperatures there was decomposition of the product to phenoxytrimethyltin and allenyltrimethyltin, possibly via a double Claisen-type rearrangement:



An additional complication observed in the reactions of two acetylenic alcohols was the formation of product mixtures containing mono-, bis- and tris-stannylated products:



The product ratios were as follows: $\text{R} = \text{H}$, $\text{A/B/C} = 70/6/24$; $\text{R} = n\text{-Pr}$, $\text{A/B/C} = 72/6/20$.

We have not attempted to clarify the reaction mechanism in detail, but we

suggest that the formation of the *E*-isomer in the acetylene reaction under certain conditions gives an important clue. Oxidative addition of the ditin to PdL_4 is likely to lead to the formation of $\text{cis-PdL}_2(\text{SnMe}_3)_2$, which in the absence of a further reactant can rearrange to the more stable *trans*-isomer; our reasoning here is based on the behaviour observed for platinum [15]. If the acetylene concentration is too low, the *cis-trans* isomerisation can compete with the reaction of the intermediate with acetylene; $\text{cis-PdL}_2(\text{SnMe}_3)_2$ is the precursor for the (*Z*)-distannylalkene, $\text{trans-PdL}_2(\text{SnMe}_3)_2$ that for the *E*-product. Polar solvents such as THF or dioxane dissolve sufficient acetylene to permit the formation of only the required *Z*-product.

(b) *Addition of other hexaalkylditins*

We have observed that hexaethyl- and hexa-*n*-butyl-ditin also add to 1-alkynes: for example, hexaethylditin adds readily ($85^\circ\text{C}/16\text{ h}$) to 2-methyl-3-butyn-1-ol to give the required product in 71% isolated yield. However, in the case of hexa-*n*-butylditin the reactions are normally incomplete, and cannot be shifted to the product side by the use of an excess of (10–50%) 1-alkyne, as shown by thin layer chromatography and ^{119}Sn NMR; the excess 1-alkyne appears to react with the catalyst. Separation of the product from hexabutylditin presents a problem; distillation is not satisfactory, leading to partial decomposition, and column chromatography (SiO_2) leads to replacement of one of the tin moieties by a proton, so that the product isolated is of the form $\text{Bu}_3\text{Sn}(\text{R})=\text{CH}_2$.

(c) *Preparation of hexaalkylditins*

Symmetrical hexaorganoditins can be prepared by treating triorganotin halides with sodium in ether [18] or liquid ammonia [19]; both methods are inconvenient when large amounts of ditins are required. We found that tetrakis(triphenylphosphine)palladium(0) catalyses the conversion of triorganotin hydrides to hexaorganoditins. This method has been independently developed by Beletskaya et al. [20], but because Russian literature is often not readily available, we have included preparative details in the experimental section.

In our hands, catalyst concentrations of less than 0.05 mol% suffice, and we have converted up to 100 g of $n\text{-Bu}_3\text{SnH}$ or over 200 g of Me_3SnH to the ditins in essentially quantitative yields. The method is unfortunately not applicable to the preparation of unsymmetrical ditins; reaction of Me_3SnH and Bu_3SnH or Ph_3SnH (1/1) with the palladium catalyst gave 1/2/1 mixtures of Me_6Sn_2 , $\text{Me}_3\text{SnSnR}_3$, and R_6Sn_2 ($\text{R} = n\text{-Bu, Ph}$).

(d) *Photochemical isomerisation to the E-isomers*

UV irradiation of the *Z*-isomers using a mercury high pressure lamp led to partial isomerisation to the *E*-isomers, as shown by NMR spectroscopy. The extent of isomerisation is shown in Table 2.

Compound **6** underwent polymerisation on prolonged irradiation. Addition of catalytic amounts of Me_3SnH did not affect the degree of isomerisation.

(e) *NMR spectroscopic characterisation of Z- and E-isomers*

The relevant data are collected in Tables 3–5. For routine use proton NMR is extremely convenient: the signal for the vinyl proton shows sets of satellites corresponding to 2J and $^3J(\text{SnH})$. The 2J values are generally between 70 and 90 Hz

TABLE 2

Z/E ISOMERISATION OF $\text{Me}_3\text{SnC(R)=CHSnMe}_3$: % CONVERSION OF THE Z- INTO THE E-ISOMER ON IRRADIATION

Compound	Max. conversion (% E) ^a	Time (h)
1	95	72
2	38	15
3	93	100
4	100	500
6	44	12 ^b
7	100	22
12	93	100
13	74	100
14	85	100
15	60	100
16	60	100
18	100	72
19 ^c	95	72

^a As determined by proton NMR spectroscopy. ^b Further irradiation leads to polymerisation. ^c Product from $\text{MeOCH}_2\text{C}\equiv\text{CCH}_2\text{OMe}$.

in the Z-isomer and are increased slightly in the E-isomer to around 100 Hz. $^3J_{\text{trans}}$ in the Z-isomer is of the order of 190–215 Hz, $^3J_{\text{cis}}$ in the E-isomer 100–115 Hz. The trimethyltin protons absorb at ca. 0.15 to 0.2 ppm, the two sets often giving only one resonance.

^{119}Sn NMR shows that the value of $^3J_{\text{cis}}$ (Sn–Sn) is ca. 400 Hz while $^3J_{\text{trans}}$ is as expected larger at ca. 900–1000 Hz. However, in two cases $^3J_{\text{trans}}$ is below 800 Hz,

TABLE 3

SELECTED PROTON NMR DATA FOR COMPOUNDS OF THE TYPE $\text{Me}_3\text{SnC(R)=CHSnMe}_3$ (δ in ppm. vs. TMS, J in Hz; values in square-brackets are for the E-isomer)

Compound	$\delta(\text{=CH})$	$^2J(\text{SnH})$	$^3J(\text{SnH})$
1	7.26 [6.94]	103 [121]	214 [121] ^a
2	6.60	90	204
3	6.65	87	214
4	6.97 [6.24]	87 [104]	192 [104]
5	6.70 [6.30]	88	196
6	6.81 [6.20]	86 [106]	196 [106]
7	6.80 [6.30]	88 [104]	196 [104]
8	6.80	86	188
9	6.73	90	201
10	6.72	88	199
11	6.71	82	202
12	6.67 [6.11]	73 [99]	210 [110]
13	6.61 [6.18]	73 [102]	213 [113]
14	6.65 [6.16]	72 [107]	210 [111]
15	6.51 [6.18]	69 [96]	205 [108]
16	6.60 [6.20]	72 [101]	212 [110]
17	8.15	77	176

^a $^3J(\text{HH})$ 17 (*cis*), 24 (*trans*) Hz.

TABLE 4

SELECTED ^{13}C NMR DATA FOR COMPOUNDS OF THE TYPE $\text{Me}_3\text{Sn}^a\text{C(R)}=\text{CHSn}^b\text{Me}_3$ (δ in ppm vs. TMS, J in Hz) ^a

Compound	$\delta(\text{Sn}^a\text{Me}_3)$	$\delta(\text{Sn}^b\text{Me}_3)$	$\delta(\text{C}(1))$	$\delta(\text{C}(2))$	$\delta(\text{C}(3))$
1	-8.2 [-9.6] (340) [337]	-8.2 [-9.6] (340) [337]	154.9 [152.9] (496,1) [409]	154.9 [152.9] (496,1) [409]	- -
2	-7.4 (325)	-7.7 (341)	142.8 (515,1) (78,2)	168.9 (499,1) (33,2)	47.3 (100,3) (71,2)
4	-7.7 [-7.9] (336) [332]	-8.5 [-8.7] (350) [336]	147.4 [145.6] (490,1) [396] (66,2) [47]	167.5 [168.7] (483,1) [398] (41,2) [39]	149.3 [148.9] ^b ^b
5	-7.1 (330)	-7.4 (342)	145.3 (503,1) (76,2)	166.5 (471,1) (37,2)	53.8 (98,3) (71,2)
6	-7.7 (338)	-7.9 (345)	141.5 (499,1) (67,2)	167.5 (483,1) (37,2)	73.5 (74,3) (51,2)
7	-7.6 [-8.9] (339) [334]	-7.8 [-7.5] (334) [339]	144.1 [140.9] (497,1) [407] (64,2) [16]	165.2 [165.0] (490,1) [421] (39,2) [44]	84.3 [79.9] (110,3) [69] (60,2) [64]
8	-7.6 (338)	-7.3 (338)	145.0 (490,1) (64,2)	163.6 (485,1) (41,2)	79.3 (108,3) (60,2)
9	-7.1 (336)	-7.6 (343)	142.8 (505,1) (73,2)	169.3 (518,1) (38,2)	75.6 (106,3) (55,2)
12	-5.0 (335)	-7.0 (346)	137.2 (508,1) (69,2)	177.5 (519,1) (32,2)	77.9 (96,3) (46,2)
13	-5.1 (334)	-6.9 (346)	138.2 (509,1) (70,2)	176.7 (520,1) (32,2)	80.1 (90,3) (45,2)
14	-4.9 (334)	-7.0 (344)	137.2 (510,1) (71,2)	178.5 (524,1) (32,2)	78.5 (94,3) (42,2)
15	-5.0 (334)	-6.9 (345)	137.9 (509,1) (70,2)	177.1 (520,1) (31,2)	80.0 (94,3) (43,2)
16	-4.9 (334)	-6.9 (346)	138.2 (508,1) (69,2)	177.1 (518,1) (32,2)	80.3 (94,3) (46,2)
17	-7.4 (355)	-8.1 (355)	157.0 (444,1) (55,2)	162.6 (470,1) (48,2)	168.5 (108,3) (64,2)
19	-5.9 (341)	-5.9 (341)	157.8 (524,1) (62,2)	157.8 (524,1) (62,2)	74.8 (91,3) (36,2)

^a Numbering: C(1) is the proton-bearing vinylic carbon, C(3) the carbon of R nearest to Sn^a. Numerals following the magnitude of nJ in brackets denote the value of n . Values in square brackets are for the *E*-isomers. ^b Not determined.

though the *cis* value is of the normal magnitude, while both values are exceptionally low when $\text{R} = \text{CO}_2\text{R}'$ ($\text{R}' = \text{Me}, \text{Et}$). The product from 1,4-diphenylbutadiyne, which was only isolated as a *Z/E*-isomer mixture, also shows low values (*cis* 261,

TABLE 5

^{119}Sn NMR DATA FOR COMPOUNDS OF THE TYPE $\text{Me}_3\text{Sn}^a\text{C(R)}=\text{CHSn}^b\text{Me}_3$ (δ in ppm vs. Me_4Sn , J in Hz)

Compound	$\delta(\text{Sn}^a)$	$\delta(\text{Sn}^b)$	$^3J(^{119}\text{Sn}-^{119}\text{Sn})$
1	-60.6 [-52.3]	-60.6 [-52.3]	491 [1013]
2	-51.3 [-47.5]	-63.5 [-70.8]	386 [997]
4	-45.9 [-39.0]	-59.4 [-62.0]	397 [861]
6	-49.7	-57.8	384
7	-49.2 [-46.7]	-59.8 [-69.3]	388 [905]
9	-53.5 [-59.0]	-61.9 [-74.1]	407 [760]
11	-54.4	-58.8	401
12	-56.6 [-42.1]	-57.2 [-73.0]	446 [1004]
13	-57.0	-57.9	452
14	-57.2	-57.7	459
15	-57.7	-57.7	456
16	-57.0	-57.8	450
17	-39.0	-52.3	287
18	-39.4 [-35.9]	-52.3 [-61.0]	289 [619]
19 ^a	-48.4 [-59.3]	-48.4 [-59.3]	443 [771]

^a Product from $\text{MeOCH}_2\text{C}\equiv\text{CH}_2\text{OMe}$. Values for the *E*-isomer in square brackets.

trans 553 Hz), while replacement of methyl on tin by butyl generally causes $^3J_{cis}$ to fall below 300 Hz.

Experimental

All manipulations involving organotin compounds were carried out in an inert atmosphere. NMR spectra were recorded using Varian EM-360 (^1H) or Bruker AM-300 spectrometers.

PdL_4 was either a commercial product (e.g. from Aldrich) or prepared as described in Brauer's handbook [21].

Hexamethylditin was prepared as follows: Trimethyltin hydride (222 g, 1.34 mol) was stirred at room temperature in a 1 l two-necked flask under argon. A catalytic amount (up to 0.05 mol%) of PdL_4 was added in ca. 0.03 g portions. (After each addition hydrogen was evolved and the temperature of the reaction mixture rose so that the tin hydride began to reflux, and so an efficient condenser was necessary.) In order to ensure complete reaction, the clear yellow solution was stirred overnight. The hexamethylditin can be distilled off under reduced pressure if required (in this case the yield is reduced to 90–95%), but as prepared suitable for the addition reactions described below. The purity of the trimethyltin hydride is important; depending on the purity of the precursor for its preparation, it may contain small quantities of dimethyltin dihydride (NMR: hydride proton multiplet ca. 0.25 ppm to high field of that for Me_3SnH). The presence of Me_2SnH_2 increases the reaction time to several days and lowers the yield of ditin either slightly or (when more than ca. 5% is present) considerably; it can be removed by distillation using a spinning band column, but we recommend rapid addition of Me_3SnCl to diisobutylaluminium hydride in dibutyl ether, which appears to minimise the amount of dihydride present.

The reactions of other trialkyltin hydrides (and of triphenyltin hydride) can be

carried out analogously (the latter in benzene as solvent). Tributyltin hydride is best prepared using the "H-siloxane" method [22].

Reaction of hexamethylditin with acetylene

Hexamethylditin (16.4 g, 50 mmol) was diluted with dry dioxane (150 ml) PdL_4 (ca. 0.05 mol%) was added, the mixture was warmed to 60°C, and dry acetylene passed into the flask. After a time the contents of the flask turned brown. The reaction was complete after 3.5 h. The solvent was removed under reduced pressure and the product (17.7 g, 87%) distilled as a colourless oil, b.p. 37°C/0.005 torr. Proton NMR spectroscopy showed the Z-product to be contaminated with up to 4% E-isomer. Anal. calcd. (found) for $\text{C}_8\text{H}_{20}\text{Sn}_2$: C, 27.2 (27.1); H, 5.7 (5.2).

Reactions of hexamethylditin with other acetylenes were carried out by heating the reactants (molar ratio 1/1) and PdL_4 (ca. 0.05 mol%) without solvent under the conditions shown in Table 1. Products were isolated by simple distillation. Reactions of hexaethylditin and hexabutylditin were also carried out in this way: however, as described in the text, hexabutylditin does not react quantitatively (e.g. the hexabutyl analogues of **9** and **12** were formed in 30% yield (^1H NMR) after 50 and 15 h, respectively at, 85°C and were characterised by ^{119}Sn NMR).

Isomerisation of (Z)-distannylalkenes was carried out by irradiating the Z-isomer (generally without solvent) for the time given in Table 2 using a 150 W mercury high pressure lamp (Hanau TQ 150). The reactions were monitored by ^1H NMR spectroscopy.

All new compounds were characterised by elemental analysis and multinuclear NMR spectroscopy.

Acknowledgement

This work was supported by the Fonds der Chemischen Industrie.

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