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1,4-Distanna-[4]ferrocenophanes by Pt(0)-catalysed distannation of alkynes

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Abstract

1,1,2,2-Tetramethyl-1,2-distanna-[2]ferrocenophane **1** reacts with alkynes R–C≡CH [R = H (**a**), Me (**b**), ^{n}Pr (**c**), ^{n}Bu (**d**), ^{n}Pen (**e**), Ph (**f**), Me_3SiOCH_2 (**g**), Et(HO)CH (**h**), EtO(O)C (**i**)], in the presence of a catalytic amount of bis(triphenylphosphane)etheneplatinum **3**, to give the 1,1,4,4-tetramethyl-1,4-distanna-[4]ferrocenophanes **4a**–**i** by 1,2-distannation of the C≡C bond. The same products are obtained by the stoichiometric reaction of the alkynes with 1,1,3,3-tetramethyl-2,2-bis(triphenylphosphane)-1,2-distanna-3-platina-[3]ferrocenophane **2**. This platinum(II) complex also reacts with cyclooctyne and dimethyl acetylene dicarboxylate to give the [4]ferrocenophanes **5** and **6**, respectively. Thus, complex **2** must be considered as the active species in the Pt(0)-catalysed distannation of alkynes. The products were characterised by ^{1}H-, ^{13}C- and ^{119}Sn-NMR, using one- and two-dimensional techniques. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Ferrocene; Platinum; Tin; Distannation; Alkynes; NMR spectroscopy

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1. Introduction

Metal-catalysed addition of main group metal fragments to C≡C bonds [1,2] provides an elegant route to novel reactive olefins which are difficult to prepare by other methods. Distannation of many alkynes has been shown to proceed under mild reaction conditions by Pd(0) catalysis [3]. Recently we have obtained the 1,3-distanna-2-platina-[3]ferrocenophane **2** [4] by oxidative addition of the 1,2-distanna-[2]ferrocenophane **1** [5] to the bis(triphenylphosphane)platinum(0) fragment (generated in situ from the ethene complex **3**), as shown in Scheme 1. The compound **2** can be readily converted into other bis(phosphane) complexes [6], all of which must be considered as the relevant intermediates in the Pt(0)-catalysed distannation of alkynes [6,7], as reported for **2** in a preliminary note [4].

In the present work we report on the reactions of **1** with alkynes in the presence of catalytic amounts of the ethene complex **3** and of alkynes with a stoichiometric amount of the 1,3-distanna-2-platina-[3]ferrocenophane **2**. ^{1}H-, ^{13}C- and ^{119}Sn-NMR spectroscopy serve for the structural characterisation of the products in solution.

2. Results and discussion

2.1. Distannation of terminal alkynes

It is shown in Scheme 1 that both the catalytic (starting from **1**) and the stoichiometric reaction (starting from **2**) afford the 1,4-distanna-[4]ferrocenophanes **4a**–**i**. In the case of **4b**, an isomer **4b′** is also formed. In the Pd(0)-catalysed reaction of hexamethyldistannane with propyne, the isomer analogous to **4b′** is the only product [3b]. The attempt to catalyse the addition of **1** to both C≡C bonds in 1,5-hexadiyne gave only a small amount of the mono-addition product **4j** which could

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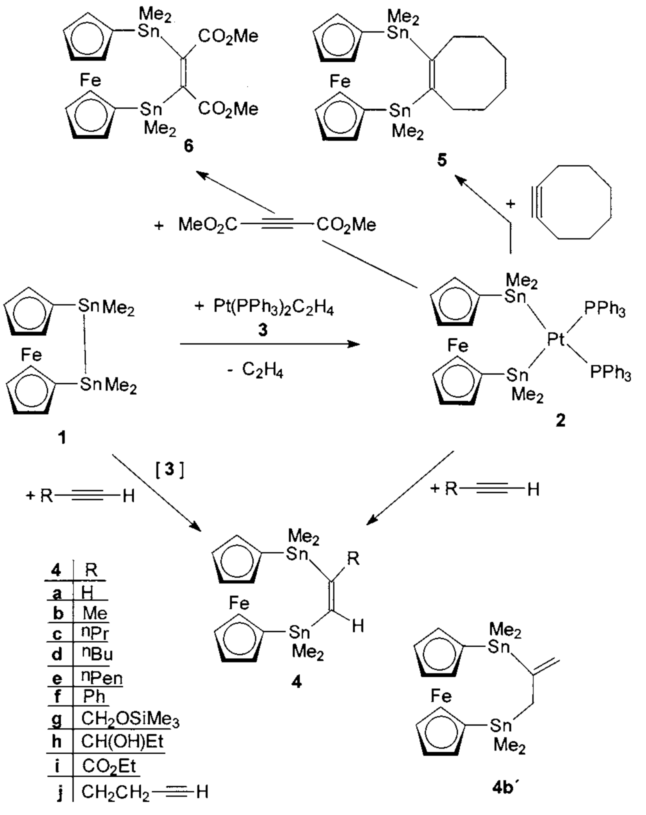

Scheme 1.

not be separated from **1**. There was no appreciable reaction of **1** (in the presence of **3**) with 1-alkynes HC≡CR bearing a more bulky substituent R (R = cyclopentyl, ^{t}Bu, $SiMe_3$, $SnMe_3$). Although ^{1}H-NMR spectra prove the high purity of **4a**–**i**, **5** and **6** (vide infra), all products could be isolated only as yellow oils, in spite of numerous attempts to obtain crystalline material.

The use of other potential catalysts such as $Pt(PPh_3)_4$, $Pd(PPh_3)_4$, $Pd(dba)_2$ (dba = *trans,trans*-dibenzylideneacetone) was not successful. If the catalytic activity of **3** would depend not only on dissociation of ethene from the Pt(0) centre but also on the cleavage of one of the Pt–P bonds, the presence of an excess of PPh_3 should have an effect. However, the yield of **4** did not change under such conditions (PPh_3 and **3** in a 1:1 ratio). It was found that the complex **2** and also its dppe congener [6] are active catalysts in the reactions leading to **4**. Therefore, it appears to be a safe assumption that **2** is indeed the important intermediate in the described Pt(0)-catalysed distannation of alkynes.

2.2. *Distannation of non-terminal alkynes and attempts at the distannation of alkenes and dienes*

There was no reaction of **1** (in the presence of **3**) with unstrained or non-activated non-terminal alkynes (3-hexyne and diphenylethyne), or with terminal and non-terminal alkenes (1-nonene, cyclooctene, norbornene), nor with dienes (norbornadiene). However, **2** reacts smoothly with cyclooctyne to give **5**. There was also a reaction in the case of dimethyl acetylene dicarboxylate and **2**: the desired compound **6** is formed only as a side product; the main reaction is trimerisation to give hexamethyl benzene hexacarboxylate, $C_6(CO_2Me)_6$.

2.3. *NMR-spectroscopic results*

The proposed structures of the new 1,4-distanna-[4]ferrocenophanes are supported by the NMR spectroscopic data (Table 1). In most cases, the ^{1}H- and/or ^{13}C-NMR spectra are well resolved and show the relevant signals accompanied by ${}^{117/119}$Sn satellite signals according to coupling constants ${}^{n}J(\mathrm{Sn}, {}^{1}\mathrm{H})$ ($n = 2, 3$) and ${}^{n}J(\mathrm{Sn}, {}^{13}\mathrm{C})$ ($n = 1$–4). The magnitude of these coupling constants is typical of the *cis*-stannyl groups at the C=C bond [3b, g, 8]. Similarly, the coupling constants ${}^{n}J({}^{119}\mathrm{Sn}, {}^{13}\mathrm{C}_{\mathrm{ferrocene}})$ ($n = 1, 2, 3$) are in the expected range [4–6,9,10]. The ^{119}Sn-NMR signals are accompanied by ^{117}Sn (AX spin system) and ^{119}Sn satellites (AB spin system) as the result of ${}^{3}J(\mathrm{Sn}, \mathrm{Sn})$. In the case of the symmetric compounds **4a**, **5** and **6**, only the ^{117}Sn satellites are observed. The magnitude of these coupling constants is in the same range as for comparable bis(trimethylstannyl) derivatives [3b, g, 8], and the changes induced by the various substituents are also similar in trend and in magnitude [8]. A positive sign of this coupling constant has been determined previously [11] by 2D heteronuclear shift correlations of the type ${}^{119}\mathrm{Sn}/{}^{1}\mathrm{H}$ in which the positive tilt of the relevant cross peaks [12] indicates alike signs of ${}^{3}K({}^{117}\mathrm{Sn}, {}^{1}\mathrm{H})$ and ${}^{3}K({}^{119}\mathrm{Sn}, {}^{117}\mathrm{Sn})$ (the reduced coupling constant K is used because of $\gamma({}^{119}\mathrm{Sn})$ and $\gamma({}^{117}\mathrm{Sn}) < 0$). Since the former is known to be positive [13], it follows that ${}^{3}K({}^{119}\mathrm{Sn}, {}^{117}\mathrm{Sn}) > 0$ [${}^{3}J({}^{119}\mathrm{Sn}, {}^{117}\mathrm{Sn}) > 0$]. A similar experiment is shown in Fig. 1, where the ${}^{13}\mathrm{C}/{}^{1}\mathrm{H}$ HETCOR for the olefinic =C–H unit reveals the relative and absolute signs of ${}^{1}J({}^{119}\mathrm{Sn}, {}^{13}\mathrm{C}{=}) < 0$ and ${}^{2}J({}^{119}\mathrm{Sn}, {=}{}^{13}\mathrm{C}) < 0$.

3. Conclusions

The new class of [4]ferrocenophanes described here is conveniently accessible from the 1,2-distanna-[2]ferrocenophane **1** by Pt(0)-catalysed distannation of terminal alkynes bearing non-bulky substituents. Since the same compounds could be prepared from the platinum complex **2** in stoichiometric reactions, **2** must be regarded as the active species in the Pt(0)-catalysed reactions. The reactivity of the Sn–C bonds [14] in the new [4]ferrocenophanes is of interest for further transformations, in particular with respect to the influence of the ferrocenediyl unit.

Table 1
^{1}H-, ^{13}C- and ^{119}Sn-NMR data[a] of the 1,4-distanna-[4]ferrocenophanes **4a**–**j**, **5** and **6**

No	$\delta\,^{1}$H				$\delta\,^{13}$C					$\delta\,^{119}$Sn
	H(2,5)	H(3,4)	$SnMe_2$	other	C(1)	C(2,5)	C(3,4)	$SnMe_2$	other	
4a	4.06 [10.6]	4.25 [6.1]	0.29 [54.9]	7.43 [204.6] [131.8]	66.7 [499.7] [2.0]	74.7 [52.8]	71.5 [41.1]	-8.0 [356.2]	154.9 (=CH) [528.2] [31.7]	−71.4 [444.2]
4b	4.06	4.26	0.30 [54.8] 0.24 [53.5]	6.81 (=CH) [204.8] [102.0] 2.17 (=CMe) [49.7][10.6]	67.3 [494.6] [2.0] 67.0 [496.4] [2.4]	75.0 [51.2] 74.9 [50.1]	71.44 [40.2] 71.38 [40.2]	−7.7 [340.1] −7.9 [356.4]	144.1 (=CH) [556.9] [73.0] 163.4 (=C) [544.8] [34.8] 33.9 (Me) [113.4] [86.1]	−73.1 [349.2] −61.5 [349.2]
4b′	4.06	4.26	0.31 [53.5] 0.29 [54.2]	5.08 (=CH) [80.1] [27.3] 5.66 (=CH) [166.4] [25.9] 2.30 ($SnCH_2$) [70.9]	67.8 [471.4] 67.1 [484.3]	74.7 [53.4] 74.6 [51.2]	71.3 [40.2] 71.2 [40.2]	−9.1 [362.9] −9.2 [347.4]	120.7 ($=CH_2$) [49.0] [40.2] 152.7 (=C) [518.8] [54.5] 25.2 ($SnCH_2$) [324.6] [55.6]	−40.1 [96.9] −17.9 [96.9]
4c	4.07 [10.4]	4.25	0.34 [54.1] 0.31 [54.7]	6.80 (=CH) [205.8] [102.6] 2.42 ($=C–CH_2$) [56.6][5.6] 1.46, 0.92 (CH_2CH_3)	67.3 [484.0] 67.0 [2.0] [496.4] [2.4]	74.7 [54.0] 74.5 [52.8]	71.4 [41.0] 71.3 [38.7]	−7.4 [338.6] −7.6 [357.4]	143.5 (=CH) [547.0] [71.6] 159.7 (=C) [518.2] [36.4] 49.7 (CH_2)[b] [101.5] [75.7]	−72.7 [362.2] −62.4 [362.2]
4d	4.08	4.26	0.36 [54.1] 0.31 [54.7]	6.81 (=CH) [206.1] [101.9] 2.46 ($=C–CH_2$) [54.7] [6.2] 1.0–1.4, 0.9 [$(CH_2)_2CH_3$]	67.8 [484.3] [2.0] 67.2 [499.8] [1.8]	75.0 [53.4] 74.5 [52.4]	71.4 [40.4] 71.3 [40.4]	−7.3 [337.9] −7.6 [356.4]	143.2 (=CH) [551.5] [73.0] 169.2 (=C) [524.8] [30.5] 47.4 (CH_2)[c] [102.5] [76.3]	−72.5 [313.5] −62.4 [313.3]
4e	4.08 [10.6]	4.26	0.36 [52.8] 0.34 [54.2]	6.83 (=CH) [207.0] [102.6] 2.47 ($=C–CH_2$) [56.6] [5.6] 0.9–1.3, 0.87 [$(CH_2)_3CH_3$]	67.9 [484.6] [2.0] 67.3 [498.0] [2.0]	74.6 [54.0] 74.4 [52.8]	71.4 [39.9] 71.3 [41.0]	−7.1 [341.6] −7.3 [356.8]	143.2 (=CH) [547.0] [72.8] 169.2 (=C) [516.4] [36.8] 42.7 (CH_2)[d] [101.5] [75.7]	−72.4 [341.7] −62.5 [341.7]
4f	4.10	4.24	0.33 [54.8] 0.29 [54.8]	6.98 (=CH) [205.8] [98.4] 7.10–7.40 (Ph)	67.9 [480.0] 67.0 [498.3]	74.5 [53.7] 74.3 [52.9]	71.04 [41.6] 71.03 [41.6]	−6.4 [358.3] −7.3 [365.2]	149.0 (=CH) [123.0] [65.0] 173.5 (=C) [507.4] [37.1] 149.8 (C_{ipso})[e] [101.1] [62.4]	−66.7 [308.9] −56.6 [308.9]
4g	4.09	4.25	0.44 [54.7] 0.31 [54.4]	7.01 (=CH) [202.0] [94.7] 4.33 (CH_2O) [48.2] [9.0] 0.11 ($SiMe_3$)	66.4 [465.0] [1.8] 66.2 [456.0] [1.8]	75.2 [52.8] 74.8 [51.6]	71.7 [41.0] 71.6 [41.0]	−6.6 [359.2] −7.6 [361.5]	140.8 (=CH) [538.8] [64.6] 168.2 (=C) [510.6] [35.2] 68.4 (CH_2O)[f] [128.6] [62.0]	−66.1 [347.9] −64.4 [347.9]
4h	4.11 4.09	4.30	0.50 [56.0] 0.48 [56.0] 0.35 [54.8] 0.31 [54.8]	6.80 (=CH) [201.6] [99.8] 4.0 (CHO) [50.2] [6.0] 2.50 (OH) 1.61, 0.92 (CH_2CH_3)	68.8 [474.2] [1.8] 68.6 [472.6] [1.8]	74.7 [50.0] 74.7 [52.8] 74.65 [54.1]	71.4 [43.0] 71.26 [47.5] 71.23 [47.5]	−5.1 [359.2] −5.7 [359.2] −7.39 [359.2] −7.43 [359.2]	140.5 (=CH) [539.6] [65.2] 173.5 (=C) [545.8] [29.2] 85.2 (CHO) [106.8] [56.8] 30.7, 10.5 (CH_2CH_3)	−68.3 [380.6] −67.1 [380.6]

Table 1 (continued)

No	δ^1H				$\delta^{13}C$					$\delta^{119}Sn$
	H(2,5)	H(3,4)	$SnMe_2$	other	C(1)	C(2,5)	C(3,4)	$SnMe_2$	other	
4i	4.06	4.25	0.23 [52.6] 0.26 [52.8]	3.32, 1.08 (OCH_2CH_3)	68.0 [432.6] 68.4 [442.6]	74.5 [54.1] 74.3 [52.8]	71.0 [42.7] 70.9 [42.3]	−6.8 [355.8] −7.0 [346.8] [346.8]	140.5 (=CH) [498.0] [56.2] 168.2 (=C) [475.5] [44.2] 165.2 (C=O) [98.6] [45.7] 60.1, 14.5 (OCH_2CH_3)	−58.5 [294.0] −54.2 [294.0]
4j	4.08	4.27	0.28 [52.3] 0.23 [52.0]	6.80 (=CH) [200.5] [103.4] 2.6 (CH_2) [51.2] [7.4] 2.2, 2.5 (CH_2CH_2) 1.8 (≡CH)	68.0 [456.3] 68.0 [456.3]	74.3 [54.0] 74.0 [53.6]	70.9 [41.0] 70.95 [41.9]	−7.6 [358.2] −6.8 [361.5]	144.8 (=CH) [542.0] [69.8] 162.0 (=C) [508.6] [42.4] 45.4 (CH_2) [101.2] [72.8] 72.9, 66.8 (C≡CH)	−78.7 [352.0] −71.7 [352.0]
5	4.06 [10.7]	4.26	0.33 [51.6]	2.6 (CH_2) [69.8] [6.2] 1.5 [$(CH_2)_4$]	68.0 [468.9] [2.0]	74.5 [52.8]	71.3 [39.5]	−6.7 [334.3]	158.3 (=C) [572.9] [63.8] 33.7 (CH_2) [86.7] [73.8] 29.2 (CH_2) [13.6] [6.2] 26.6 (CH_2)	−63.3 [488.3]
6	4.05	4.28	0.23 [54.2]	3.33 (OCH_3)	68.2 [465.3]	74.7 [54.4]	70.8 [42.5]	−6.5 [360.2]	160.2 (=C) [475.5] [44.2] 165.2 (C=O) [102.4] [49.8] 56.8 (OMe)	−35.7 [309.9]

[a] In C_6D_6 at room temperature; coupling constants ${}^nJ({}^{119}Sn, {}^1H)$ (±0.5 Hz), ${}^nJ({}^{119}Sn, {}^{13}C)$ (±1 Hz), and ${}^nJ({}^{119}Sn, {}^{117}Sn)$ (±1.5 Hz) are given in brackets.

[b] $\delta^{13}C = 23.5$ [11.7], 13.8 (CH_2CH_3).

[c] $\delta^{13}C = 32.7$ [9.8], 22.6, 14.2 ($CH_2CH_2CH_3$).

[d] $\delta^{13}C = 30.3$ [11.7], 23.0, 23.0, 14.2 ($CH_2CH_2CH_2CH_3$).

[e] $\delta^{13}C = 130.3$ (C_{ortho}), 128.0 (C_{meta}), 126.1 (C_{para}).

[f] $\delta^{13}C = -0.3$ (Me_3Si).

4. Experimental

4.1. General

All compounds were handled in an atmosphere of dry argon, and carefully dried solvents were used for syntheses and the preparation of the samples for NMR measurements. Starting materials were either used as commercial products without further purification (all alkynes, alkenes and dienes) or prepared as described (**1** [5], **2** [4], **3** [15]). Electron impact (EI) mass spectra: Finnigan MAT 8500 with direct inlet (the isotope distribution in calculated mass spectra is in agreement with the experimental spectra). NMR measurements: Bruker ARX 250 or DRX 500; ${}^1H$-, ${}^{13}C$-, ${}^{119}Sn$-NMR (measured either directly using inverse gated ${}^1H$ decoupling or by refocused INEPT [16], based on ${}^2J({}^{119}Sn, {}^1H) = 55$ Hz, with ${}^1H$ decoupling). Chemical shifts are given with respect to Me_4Si [δ^1H ($CHCl_3/CDCl_3$) = 7.24, (C_6D_5H) = 7.14, ($C_6D_5CD_2H$) = 2.03; $\delta^{13}C$ ($CDCl_3$) = 77.0, (C_6D_6) = 128.0, ($C_6D_5CD_3$) = 20.4], and Me_4Sn [$\delta^{119}Sn = 0$ for $\Xi({}^{119}Sn) = 37.290665$ MHz].

4.2. 1,1,4,4-Tetramethyl-1,4-distanna-[4]ferrocenophanes **4**, **5**, **6**: general procedures, starting from **1** with catalyst **3**, or from **2**

A THF solution (30 ml) containing **1** (0.12 g; 0.25 mmol), the alkyne (0.3 mmol) and **3** (0.0025 mmol) (30 ml) is heated at reflux for 72 h. In the cases of **4a**,**b**, ethyne and propyne, respectively, were introduced into the boiling THF solution containing **1** and **3**, and the reaction was complete after 4–5 h. The solvent is removed in vacuo, and the residue is extracted with hexane. After filtration, chromatography of the solution on silica and elution with hexane affords the products **4a**–**i** as orange oils.

The respective alkyne (0.15 mmol) is added to a solution of the complex **2** (0.12 g; 0.1 mmol) in THF (30 ml), and

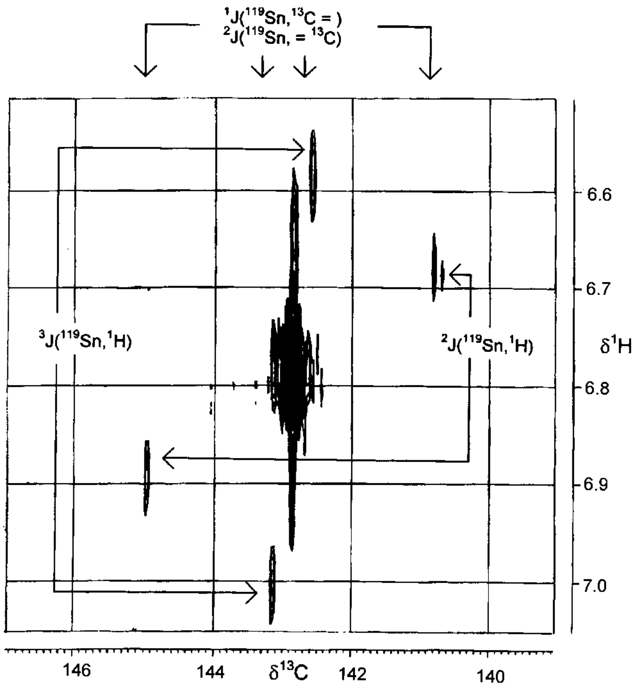

Fig. 1. Contour plot of the 2D 125.8 MHz $^{13}C/^1H$ HETCOR (based on $^1J(=^{13}C, ^1H)$) of the olefinic carbon atom of **4d**. The positive tilt of all cross peaks for the $^{117/119}Sn$ satellites shows that $^1K(Sn, ^{13}C=)$, $^2K(Sn, =^{13}C)$, $^2K(Sn, ^1H)$ and $^3K(Sn, ^1H)$ have the same sign (all positive, since $^{2/3}K(Sn, ^1H) > 0$ [13]).

the mixture is heated at reflux for 72 h (4–5 h for **4a**,**b**). The products **4a**–**i** are obtained in similar yield as described above. Furthermore, the compounds **5** and **6** are isolated by the stoichiometric reaction as orange oils. In the case of **5**, the reaction starts instantaneously.

4a	yield 87%; $C_{16}H_{22}FeSn_2$, EI-MS: *m*/*e* (%) = 507 (100) [M^+]
4b, **4b′**	yield 50%; $C_{17}H_{24}FeSn_2$, EI-MS: *m*/*e* (%) = 521 (100) [M^+]
4c	yield 45%; $C_{19}H_{28}FeSn_2$, EI-MS: *m*/*e* (%) = 549 (70) [M^+], 469 (100) [$Fe(C_5H_4)_2Sn_2Me_3^+$]
4d	yield 56%; $C_{20}H_{30}FeSn_2$, EI-MS: *m*/*e* (%) = 563 (70) [M^+], 469 (100) [$Fe(C_5H_4)_2Sn_2Me_3^+$]
4e	yield 31%; $C_{21}H_{32}FeSn_2$, EI-MS: *m*/*e* (%) = 577 (40) [M^+], 303 (100) [$Fe(C_5H_4)_2Sn^+$]
4f	yield 54%; $C_{22}H_{26}FeSn_2$, EI-MS: *m*/*e* (%) = 577 (80) [M^+], 469 (100) [$Fe(C_5H_4)_2Sn_2Me_3^+$]
4g	yield 67%; $C_{20}H_{32}FeOSiSn_2$, EI-MS: *m*/*e* (%) = 609 [M^+]
4h	yield 64%; $C_{19}H_{28}FeOSn_2$, EI-MS: *m*/*e* (%) = 565 (100) [M^+]
4l	yield 13%; $C_{19}H_{26}FeO_2Sn_2$, EI-MS: *m*/*e* (%) = 579 (20) [M^+], 469 (100) [$Fe(C_5H_4)_2Sn_2Me_3^+$]
5	yield 97%; $C_{22}H_{32}FeSn_2$, EI-MS: *m*/*e* (%) = 589 (60) [M^+], 469 (100) [$Fe(C_5H_4)_2Sn_2Me_3^+$]
6	yield 11%; $C_{22}H_{30}FeO_4Sn_2$, EI-MS: *m*/*e* (%) = 623 (100) [M^+]

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References

[1] (a) J.K. Kochi, Organometallic Mechanism and Catalysis, Academic Press, New York, 1978. (b) R.F. Heck, Palladium Reagents in Organic Synthesis, Academic Press, New York, 1985. (c) H. Watanabe, M. Kobayashi, M. Saito, Y. Nagai, J. Organomet. Chem. 215 (1991) 149. (d) N. Miyaura, A. Suzuki, Chem. Rev. 95 (1995) 2457.

[2] (a) T.N. Mitchell, R. Wickenkamp, A. Amamria, R. Dicke, U. Schneider, J. Org. Chem. 52 (1987) 4868. (b) B.L. Chenard, C.M. van Zyl, J. Org. Chem. 51 (1986) 3561. (c) M. Murakami, Y. Morita, Y. Ito, J. Chem. Soc. Chem. Commun. (1990) 428. (d) W. Finckh, B.-Z. Tang, A.J. Lough, I. Manners, Organometallics, 11 (1992) 2904.

[3] (a) E. Piers, R.T. Skerjl, J. Chem. Soc. Chem. Commun. (1986). (b) T.N. Mitchell, A. Amamria, H. Killing, D. Rutschow, J. Organomet. Chem. 304 (1986) 257. (c) T.N. Mitchell, U. Schneider, J. Organomet. Chem. 407 (1991) 319. (d) H. Killing, T.N. Mitchell, Organometallics, 3 (1984) 1017. (e) T.N. Mitchell, N.M. Dornseifer, A. Rahm, J. High. Press. Res. 7 (1991) 165. (f) T.N. Mitchell, G. Kowall, J. Organomet. Chem. 437 (1992) 127. (g) M. Niestroj, W.P. Neumann, T.N. Mitchell, J. Organomet. Chem. 519 (1996) 45.

[4] M. Herberhold, U. Steffl, W. Milius, B. Wrackmeyer, Angew. Chem. 107 (1997) 1545; Angew. Chem. Int. Ed. Engl. 36 (1997) 1510.

[5] M. Herberhold, U. Steffl, W. Milius, B. Wrackmeyer, Angew. Chem. 108 (1996) 1927; Angew. Chem. Int. Ed. Engl. 35 (1996) 1803.

[6] M. Herberhold, U. Steffl, W. Milius, B. Wrackmeyer, Chem. Eur. J. 4 (1998) 1027.

[7] (a) Y. Obora, Y. Tsuji, K. Nishiyama, M. Ebihara, T. Kawamura, J. Am. Chem. Soc. 118 (1996) 10922. (b) Y. Tsuji, K. Nishiyama, S. Hori, M. Ebihara, T. Kawamura, Organometallics, 17 (1998) 507.

[8] B. Wrackmeyer, K. Wagner, Chem. Ber. 122 (1989) 857.

[9] F.H. Köhler, W.A. Geike, N. Hertkorn, J. Organomet. Chem. 334 (1987) 359.

[10] (a) M. Herberhold, U. Steffl, W. Milius, B. Wrackmeyer, J. Organomet. Chem. 533 (1997) 109. (b) M. Herberhold, U. Steffl, W. Milius, B. Wrackmeyer, Z. Anorg. Allg. Chem. 624 (1998) 386. (c) M. Herberhold, W. Milius, U. Steffl, K. Vitzithum, W. Milius, B. Wrackmeyer, R.H. Herber, M. Fontani, P. Zanello, Eur. J. Inorg. Chem., in press.

[11] B. Wrackmeyer, K. Wagner, unpublished results.

[12] (a) A. Bax, R. Freeman, J. Magn. Reson. 45 (1981) 177. (b) B. Wrackmeyer, K. Horchler, Magn. Reson. Chem. 28 (1990) 56.

[13] V.S. Petrosyan, Prog. NMR Spectrosc. 11 (1977) 115.
[14] (a) M. Pereyre, J.-P. Quintard, A. Rahm, Tin in Organic Synthesis, Butterworths, London, 1987. (b) A.G. Davies, Organotin Chemistry, VCH, Weinheim, 1997.
[15] U. Nagel, Chem. Ber. 115 (1982) 1948.
[16] (a) G.A. Morris, R. Freeman, J. Am. Chem. Soc. 101 (1979) 760. (b) G.A. Morris, J. Am. Chem. Soc. 102 (1980) 428. (c) D.P. Burum, R.R. Ernst, J. Magn. Reson. 39 (1980)163.