



Hydrostannation of norbornadiene by ${}^n\text{Bu}_3\text{SnH}$ and Ph_3SnH with molybdenum catalysts

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ABSTRACT

Photochemically activated $[\text{Mo}(\text{CO})_6]$ and $[\text{Mo}(\text{CO})_4(\eta^4\text{-nbd})]$ are demonstrated to be good catalysts for the hydrostannation of norbornadiene (nbd) by stannyl hydrides (${}^n\text{Bu}_3\text{SnH}$ and Ph_3SnH) to give stannylnorbornene in ca. 100% yield. The reaction was carried out at room temperature in *n*-heptane (20 cm³) with continued photolysis (2 h) at $\text{Mo}(\text{CO})_6$: R_3SnH :nbd = 1:50:50.

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1. Introduction

Hydrostannation of alkynes and alkenes is widely used in organic synthesis due to various applications of organostannane compounds as starting materials for further transformations [1–11]. Palladium complexes are applied the most frequently as catalysts for that reaction. However, as was recently discovered molybdenum carbonyl complexes can also be used as catalysts for the hydrostannation reaction [6–10]. In the presence of the molybdenum complex $[\text{MoBr}(\eta^3\text{-C}_3\text{H}_5)(\text{CO})_2(\text{NCMe})_2]$, tributyltin hydride instantaneously adds to various alkynes to give the corresponding vinylstannanes in good yields under mild conditions. The molybdenum complex showed comparable or even better selectivity than the palladium complex $[\text{PdCl}_2(\text{PPh}_3)_2]$ in the case of certain sterically hindered alkynes [6]. Another molybdenum complex, $[\text{Mo}(\text{CO})_3(\text{CN-}t\text{-Bu})_3]$, was found to be a catalyst for the hydrostannation reaction by Kazmaier *et al.* [7–10]. In the presence of this catalyst, the hydrostannation of propargylic alcohols, ethers, amines, and amides gave rise to functionalized vinylstannanes in good yield. However, up to now, there have been few examples of the hydrostannation of alkenes or dienes and none with a molybdenum catalyst [1,11]. As was described by Neumann and Sommer, the hydrostannation of conjugated dienes, such as butadiene, cyclopentadiene and cyclohexadiene by organotin hydrides has been realized with high yield under free-radical conditions [11].

Although the mechanism of the transition-metal-catalyzed hydrostannation reaction is not very well investigated, it has been postulated that it involves such steps as oxidative addition of the Sn–H bond of R_3SnH to the transition metal, hydrometallation of an unsaturated bond of the coordinated organic substrate, and reductive elimination of the product. The very important first step in this reaction can be realized only with an electron-rich transition metal, i.e. one in a low oxidation state. The activation of the Si–H bond of silanes and the Ge–H bond of germanes by photochemically activated molybdenum carbonyl complexes has recently been observed by spectroscopic methods (IR, NMR) in solution at low temperatures [12–15] and successfully applied in molybdenum-catalyzed hydrosilylation and hydrogermylation of norbornadiene (nbd). This inspired us to test the catalytic activity of $[\text{Mo}(\text{CO})_6]$ (1) and $[\text{Mo}(\text{CO})_4(\eta^4\text{-nbd})]$ (2) in the hydrostannation of nbd, which can give stannylnorbornene ($\text{R}_3\text{Sn-nbe}$).

In this paper, we present the hydrostannation of nbd by ${}^n\text{Bu}_3\text{SnH}$ and Ph_3SnH , giving respectively tributylstannylnorbornene (${}^n\text{Bu}_3\text{Sn-nbe}$) or triphenylstannylnorbornene ($\text{Ph}_3\text{Sn-nbe}$) (Scheme 1).

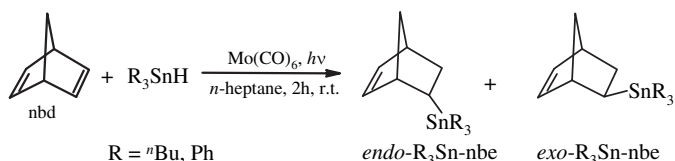
2. Experimental

2.1. General remarks

All reactions were performed using standard Schlenk techniques under an atmosphere of nitrogen and with freshly distilled substrates and solvents. $\text{Mo}(\text{CO})_6$ (1), ${}^n\text{Bu}_3\text{SnH}$, and Ph_3SnH

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Scheme 1. Transformation of nbd in the presence of photochemically activated Mo(CO)₆ and tin hydrides R₃SnH (R = ⁿBu, Ph).

(Aldrich) were used as received. The norbornadiene compound [Mo(CO)₄(η⁴-nbd)] (**2**) was obtained from a photochemical reaction of Mo(CO)₆ and nbd in *n*-heptane solution and purified by sublimation [16–18]. The photolysis source was an HBO 200 W high-pressure Hg lamp.

¹H, ¹³C NMR, and two-dimensional ¹H–¹H COSY and ¹H–¹³C HMQC NMR spectra were recorded with a Bruker AMX 300 or 500 MHz instrument. All proton and carbon chemical shifts were referenced to the residual proton signal for ¹H NMR (δ 7.24 CDCl₃ and 1.40 C₆D₁₂) or the natural abundant carbon signal of the solvent for ¹³C NMR (δ 77.0 CDCl₃ and 26.4 C₆D₁₂). ¹¹⁹Sn NMR chemical shifts were referenced relative to Ph₃SnCl (δ –44.7) used as external standard.

2.2. General procedure for hydrostannation of nbd by ⁿBu₃SnH or Ph₃SnH in the presence of Mo(CO)₆

A solution of Mo(CO)₆ (0.005 g, 0.019 mmol), nbd (0.087 g, 0.1 cm³, 0.95 mmol), and ⁿBu₃SnH (0.25 cm³, 0.95 mmol) or Ph₃SnH (0.28 g, 0.95 mmol) in *n*-heptane solution (20 cm³) was stirred and irradiated at room temperature for 2 h. All volatile materials were then evaporated under reduced pressure at room temperature, and the residue was analyzed by NMR spectroscopy, which revealed the formation of ⁿBu₃Sn-nbe or Ph₃Sn-nbe as a mixture of *endo* and *exo* isomers at an almost 50/50 ratio.

2.3. NMR characteristic of hydrostannation products

endo-ⁿBu₃Sn-nbe: ¹H NMR (δ, CDCl₃, 300 MHz): 5.96 (dd, ³J_{H–H} = 5.5 Hz, ⁴J_{H–H} = 2.9 Hz, 1H, HC²), 5.91 (dd, ³J_{H–H} = 5.5 Hz, ⁴J_{H–H} = 3.0 Hz, 1H, HC³), 3.03 (s, 1H, HC⁴), 2.89 (s, 1H, HC¹), 1.95 (ddd, ²J_{H–H} = 11.4 Hz, ³J_{H–H} = 9.5 Hz, ⁴J_{H–H} = 3.6 Hz, 1H, H₂C⁶), other signals: 1.5 (1H, H₂C⁶), 1.4 (1H, H₂C⁷), 1.2 (1H, HC⁵) and 1.1 (1H, H₂C⁷), were obscured by multiplet signals due to the ⁿBu groups centered at 1.50, 1.32, and 0.90 ppm and were identified in ¹H–¹H COSY and ¹H–¹³C HMQC spectra. ¹³C{¹H} NMR (δ, CDCl₃, 75 MHz): 135.3 (1C, HC²), 135.0 (1C, ³J_{C–Sn} = 22 Hz, HC³), 50.4 (1C, ³J_{C–Sn} = 46 Hz, H₂C⁷), 46.1 (1C, HC⁴), 41.8 (1C, ³J_{C–Sn} = 18 Hz, HC¹), 29.4 (3C, (1C, ²J_{C–Sn} = 19 Hz, CH₂-ⁿBu), 28.1 (1C, H₂C⁶), 27.6 (3C, ³J_{C–Sn} = 47 Hz, CH₂-ⁿBu), 21.1 (1C, ¹J_{C–Sn} = 362/378 Hz, ¹¹⁷Sn/¹¹⁹Sn, HSnC⁵), 13.7 (3C, CH₃-ⁿBu), 9.2 (3C, ¹J_{C–Sn} = 295/308 Hz, ¹¹⁷Sn/¹¹⁹Sn, SnCH₂-ⁿBu).

exo-ⁿBu₃Sn-nbe: ¹H NMR (δ, CDCl₃, 300 MHz) 6.05 (dd, ³J_{H–H} = 5.5, ⁴J_{H–H} = 2.7 Hz, 1H, HC³), 5.83 (dd, ³J_{H–H} = 5.5, ⁴J_{H–H} = 3.1 Hz, 1H, HC²), 2.95 (s, 1H, HC¹), 2.85, (s, 1H, HC⁴), 1.75 (ddd, ²J_{H–H} = 11.4 Hz, ³J_{H–H} = 5.1 Hz, ⁴J_{H–H} = 3.6 Hz, 1H, H₂C⁶), other signals: 1.5 (1H, H₂C⁶), 1.2 (1H, HC⁷), 1.0 (1H, H₂C⁷), 0.7 (1H, H₂C⁵) were obscured by multiplet signals due to the ⁿBu groups centered at 1.50, 1.32, and 0.90 ppm and were identified in ¹H–¹H COSY and ¹H–¹³C HMQC spectra. ¹³C{¹H} NMR (δ, CDCl₃, 75 MHz): 136.7 (1C, ³J_{C–Sn} = 50 Hz, HC³), 132.1 (1C, HC²), 48.1 (1C, H₂C⁷), 45.0 (1C, HC⁴), 43.0 (1C, HC¹), 29.3 (3C, CH₂-ⁿBu), 28.6 (1C, H₂C⁶), 27.5 (3C, ³J_{C–Sn} = 50 Hz, CH₂-ⁿBu), 20.2 (1C, ¹J_{C–Sn} = 313/327 Hz,

¹¹⁷Sn/¹¹⁹Sn, HSnC⁵), 13.7 (3C, CH₃-ⁿBu), 8.8 (3C, ¹J_{C–Sn} = 291/304 Hz, ¹¹⁷Sn/¹¹⁹Sn, SnCH₂-ⁿBu).

endo-Ph₃Sn-nbe: ¹H NMR (δ, CDCl₃, 300 MHz): 7.8–7.4 m, 15H, Ph), 6.09 (dd, ³J_{H–H} = 5.6 Hz, ⁴J_{H–H} = 2.8 Hz, 1H, HC²), 6.03 (dd, ³J_{H–H} = 5.6 Hz, ⁴J_{H–H} = 2.8 Hz, 1H, HC³), 3.44 (s, 1H, HC⁴), 3.08 (s, 1H, HC¹), 2.30 (m, 2H, HC⁵, H₂C⁶), 1.61 (m, 2H, H₂C⁶, H₂C⁷), 1.29 (d, 1H, ²J_{H–H} = 8.4 Hz, H₂C⁷). ¹³C{¹H} NMR (δ, CDCl₃, 125 MHz): 139–128 (18C, Ph), 136.0 (1C, HC²), 135.5 (1C, ³J_{C–Sn} = 28 Hz, HC³), 50.0 (1C, ³J_{C–Sn} = 58 Hz, H₂C⁷), 45.8 (1C, ²J_{C–Sn} = 11 Hz, HC⁴), 41.9 (1C, ³J_{C–Sn} = 23 Hz, HC¹), 28.3 (1C, H₂C⁶), 24.8 (1C, ¹J_{C–Sn} = 458/478 Hz, ¹¹⁷Sn/¹¹⁹Sn, HSnC⁵).

exo-Ph₃Sn-nbe: ¹H NMR (δ, CDCl₃, 300 MHz): 7.8–7.4 m, 15H, Ph), 6.24 (dd, ³J_{H–H} = 5.6 Hz, ⁴J_{H–H} = 2.8 Hz, 1H, HC³), 6.05 (dd, ³J_{H–H} = 5.6 Hz, ⁴J_{H–H} = 2.8 Hz, 1H, HC²), 3.30 (s, ³J_{H–Sn} = 25 Hz, 1H, HC⁴), 3.08 (s, 2H, HC¹), 2.21 (ddd, ²J_{H–H} = 10 Hz, ³J_{H–H} = 7 Hz, ⁴J_{H–H} = 3.5 Hz, 1H, H₂C⁶), 1.59 (m, 1H, H₂C⁶), 1.42 (d, 1H, ²J_{H–H} = 9.5 Hz, H₂C⁷) 1.25 (d, 1H, ²J_{H–H} = 9.5 Hz, H₂C⁷). ¹³C{¹H} NMR (δ, CDCl₃, 125 MHz): 139–128 (18C, Ph), 136.5 (1C, ³J_{C–Sn} = 61 Hz, HC³), 133.2 (1C, HC²), 48.1 (1C, H₂C⁷), 45.0 (1C, HC⁴), 43.4 (1C, ³J_{C–Sn} = 11 Hz, HC¹), 28.2 (1C, H₂C⁶), 22.8 (1C, ¹J_{C–Sn} = 393/412 Hz, ¹¹⁷Sn/¹¹⁹Sn, HSnC⁵).

¹¹⁹Sn{¹H} NMR (186.5 MHz, CDCl₃, 298 K): δ = –89.6 and –91.4 for *endo*-Ph₃Sn-nbe and *exo*-Ph₃Sn-nbe.

2.4. General procedure for reaction in NMR tube

The course of the hydrostannation reaction was also followed by ¹H NMR spectroscopy. As a general procedure for NMR experiments, complex **2** (ca. 0.01 g, 0.03 mmol) was weighed into an NMR tube. The tube was then capped with a septum. A portion of ⁿBu₃SnH (ca. 0.03 cm³) and C₆D₁₂ (0.7 cm³) was then added to the NMR tube via a syringe. The tube was periodically irradiated and monitored by ¹H NMR spectroscopy over the desired length of time at 10 °C.

3. Results and discussion

3.1. Hydrostannation of nbd initiated by photochemically activated molybdenum(0) complexes **1** or **2**

The hydrostannation of nbd by ⁿBu₃SnH in the presence of a catalytic amount (ca. 2 mol%) of the molybdenum complex **1** or **2** gave ⁿBu₃Sn-nbe, which was identified by NMR methods (¹H, ¹³C {¹H}, ¹H–¹H COSY, and ¹H–¹³C HMQC) as a ca. 50/50 mixture of *endo* and *exo* isomers. The *endo* isomer alone was observed only during the monitoring of photochemical reaction of **2** with ⁿBu₃SnH by ¹H NMR spectroscopy in C₆D₁₂ solution. The olefin proton signals of *endo*-ⁿBu₃Sn-nbe appeared at the beginning of the reaction as two equal-intensity multiplets at δ = 5.96 and 5.91. In the mixture of products isolated from prolonged (2 h) reaction catalyzed by photochemically activated **1**, ⁿBu₃Sn-nbe was detected by ¹H NMR in ca. 100% yield, as a mixture (ca. 1:1) of *endo* and *exo* stereoisomers due to four equal-intensity olefin proton signals in the region of 6.1–5.8 ppm (Fig. 1). The ¹³C{¹H} NMR spectrum of ⁿBu₃Sn-nbe shows fourteen carbon signals due to seven carbons of the norbornyl moiety. Several signals clearly show satellites due to coupling with the tin atom (¹¹⁷Sn and ¹¹⁹Sn, *I* = 1/2, natural abundance of 7.61% and 8.58%). The highest value of the ¹³C–¹¹⁷Sn/¹¹⁹Sn coupling constant was obtained for signals at δ 21.1 (¹J_{C–Sn} = 362/378 Hz) and δ 20.2 (¹J_{C–Sn} = 313/327 Hz). These resonances can thus be assigned to carbons adjacent to the tin atom (C⁵) of the *endo* and *exo* isomers of ⁿBu₃Sn-nbe. The coupling constants through three bonds: ³J_{C–Sn} = 50, 22, and 18 Hz, were obtained respectively for signals at δ 50.4 (H₂C⁷), 135.0 (HC³) and 41.8 (HC¹) of the *endo*

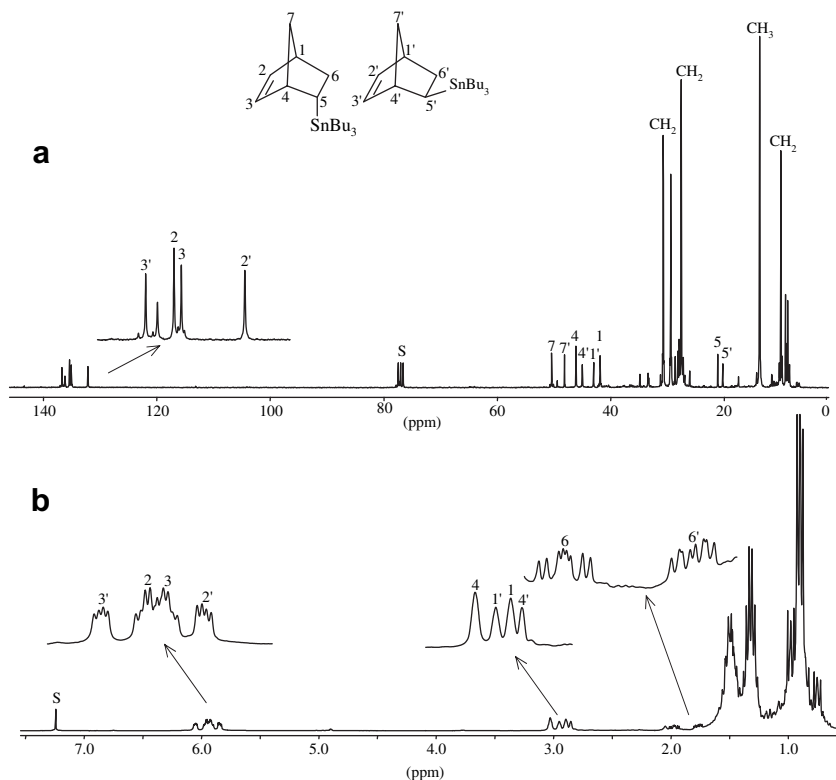


Fig. 1. (a) $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum (75 MHz, CDCl_3) and (b) ^1H NMR spectrum (300 MHz, C_6D_{12}) of $^n\text{Bu}_3\text{Sn-nbe}$. The characteristic carbon and proton signals are presented with the scale expansion. The proton and carbon signal denoted by S is due to CDCl_3 .

isomer, and $^3J_{\text{C-Sn}} = 50$ Hz for signal at δ 136.7 (HC^3) of the *exo* isomer.

A similar analysis of NMR data of products obtained in reaction of Ph_3SnH and *nbd* made it possible to detect $\text{Ph}_3\text{Sn-nbe}$ as a ca. 50:50 mixture of *endo* and *exo* isomers.

However, the formation of $\text{Ph}_3\text{Sn-nbe}$ is always accompanied by the appearance of stannyl-substituted tricyclane, which was identified due to carbon signals, of very low intensity compared with signals of $\text{Ph}_3\text{Sn-nbe}$, at $\delta = 34.5, 34.4, 33.8, 33.1, 14.1, 10.7$ and 10.2 ppm,

3.2. Activation of the H–Sn bond of stannane in photochemical reaction of complex **2**

Monitoring the reaction of **2** with $^n\text{Bu}_3\text{SnH}$ in C_6D_{12} solution at 10°C by ^1H NMR spectroscopy allowed us to observe the decay of proton resonance arising from the H–Sn bond of $^n\text{Bu}_3\text{SnH}$ ($\delta = 4.82$, septet, $^3J_{\text{H-H}} = 2$ Hz, $^1J_{\text{H-Sn}} = 1541/1609$ Hz, $^{117}\text{Sn}/^{119}\text{Sn}$), and the appearance of several high-field resonances, in the region characteristic of hydride ligands, at $\delta = -4.59$ ($J_{\text{H-Sn}} = 85$ Hz), -5.46 ($J_{\text{H-Sn}} = 116$ Hz), -5.68 ($J_{\text{H-Sn}} = 97$ Hz), -5.91 ($J_{\text{H-Sn}} = 267$ Hz),

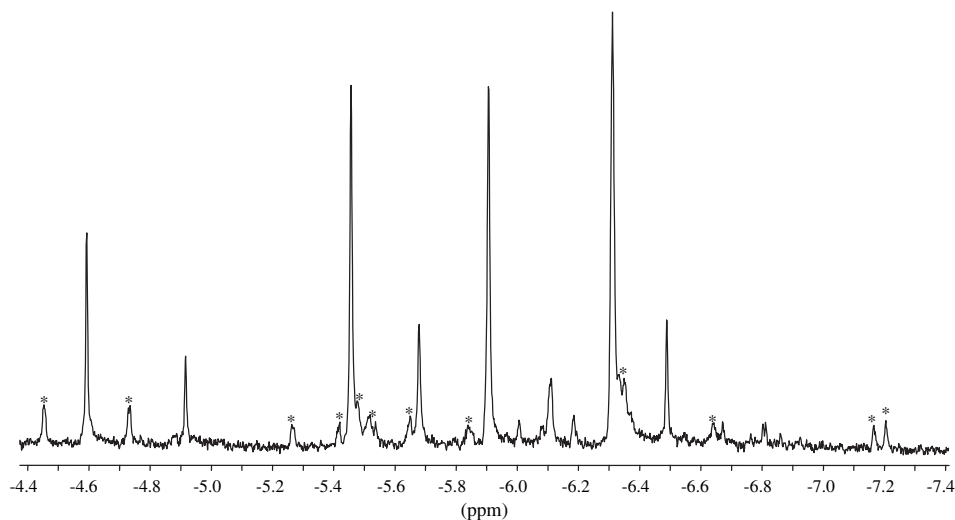
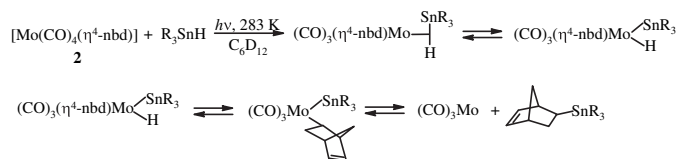


Fig. 2. Partial (hydride region) ^1H NMR spectrum (300 MHz, C_6D_{12} , 10°C) showing hydride signals of compounds formed during photochemical reaction of the norbornadiene complex **2** and $^n\text{Bu}_3\text{SnH}$. Asterisks denote $^{117}/^{119}\text{Sn}$ satellites ($I = 1/2, 7.61/8.58\%$).



Scheme 2. The formation of ${}^n\text{Bu}_3\text{Sn-nbe}$ in photochemical reaction of the norbornadiene complex **2** and ${}^n\text{Bu}_3\text{SnH}$.

–6.31 ($J_{\text{H-Sn}} = 524 \text{ Hz}$), –6.49 ($J_{\text{H-Sn}} = 92 \text{ Hz}$), at an approximate intensity ratio of 1:1.7:0.7:1.7:2:0.7 (Fig. 2 and Fig. 1S on Appendix A). The detection of distinctly different $J_{\text{H-Sn}}$ coupling constants of the hydrides indicates evidently different interactions of the H–Sn bond with the molybdenum atom in each of the new complexes formed during photolysis of **2**. In the low-field region of the ${}^1\text{H}$ NMR spectrum, two new, equal-intensity signals at $\delta = 4.61$ and 3.82 ppm, ($t, J_{\text{H-H}} = 2.5 \text{ Hz}$) can be assigned to four olefin protons in the new norbornadiene complex. The intensity of one of those signals was integrated as 2 in comparison to the intensity of the hydride ligand, at $\delta = -4.59$, integrated as 1. Other signals of norbornadiene moiety were not detected; they are obscured by signals of ${}^n\text{Bu}_3\text{Sn-nbe}$ formed selectively as the *endo* isomer in this reaction. However, within a few minutes all the hydride signals decayed, except the one at $\delta = -6.49$. It must be noted that photolysis of complex **2** in the presence of an excess of ${}^n\text{Bu}_3\text{SnH}$ and nbd leads to the formation of *endo* and *exo* isomers of ${}^n\text{Bu}_3\text{Sn-nbe}$, like in a reaction catalyzed by complex **1**.

To the best of our knowledge, these are the first results clearly showing that in the initial step of hydrostannation catalyzed by a molybdenum complex containing a diene ligand, the H–Sn bond of ${}^n\text{Bu}_3\text{SnH}$ is coordinated to the molybdenum center. The η^2 -coordination of the H–Sn bond to the molybdenum center leads to its activation (oxidative addition) followed by the addition of the $\eta^2\text{-H-Sn}{}^n\text{Bu}_3$ ligand to the olefin bond. The reaction pathway could be interpreted as depicted in Scheme 2. The reaction proceeds via σ -complex formation followed by oxidative addition of the H–Sn bond to a coordinatively unsaturated molybdenum(0) species formed during photolysis. Subsequent η^4 -nbd ligand insertion into the Mo–H bond leads to the formation of the η^1 -norbornylene ligand. Finally, reductive elimination stereoselectively gives *endo*- ${}^n\text{Bu}_3\text{Sn-nbe}$ and a coordinatively unsaturated molybdenum(0) carbonyl species, which is the true catalyst for the hydrostannation reaction.

4. Conclusions

To sum up, we have succeeded in generating stannylnorbornenes as a result of hydrostannation of nbd by tin hydrides

(${}^n\text{Bu}_3\text{SnH}$ and Ph_3SnH) catalyzed by photochemically activated $\text{Mo}(\text{CO})_6$. In reaction carried out at room temperature in *n*-heptane solution with continued photolysis (2 h) at $\text{Mo}(\text{CO})_6$: R_3SnH : nbd = 1:50:50, ca. 100% yield of hydrostannation products was achieved. Analysis of ${}^1\text{H}$ NMR spectra, revealed the formation of ${}^n\text{Bu}_3\text{Sn-nbe}$ or $\text{Ph}_3\text{Sn-nbe}$ as a mixture of *endo* and *exo* isomers at an almost 50/50 ratio for all catalytic reaction tested. The *endo* isomer alone was observed only during the monitoring of photochemical reaction of $[\text{Mo}(\text{CO})_4(\eta^4\text{-nbd})]$ with ${}^n\text{Bu}_3\text{SnH}$ by ${}^1\text{H}$ NMR spectroscopy in C_6D_{12} solution.

The hydrostannation was observed step by step during the monitoring of the photochemical reaction of the norbornadiene complex $[\text{Mo}(\text{CO})_4(\eta^4\text{-nbd})]$ with ${}^n\text{Bu}_3\text{SnH}$ by ${}^1\text{H}$ NMR spectroscopy in C_6D_{12} solution at 10°C . The initially formed intermediate complexes of molybdenum containing the $\eta^2\text{-H-Sn}{}^n\text{Bu}_3$ or hydride and stannyl ligands were detected by ${}^1\text{H}$ NMR spectroscopy due to high-field hydride resonances.

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Appendix. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2010.03.023.

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