

# Synthesis and characterization of new disilane derivatives of tungsten

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## Abstract

By use of salt elimination,  $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{R}$  ( $\text{R} = \text{Me}, \text{Cl}, \text{Br}$ ),  $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3$ ,  $(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Cl}$  ( $\text{C}_9\text{H}_7 = \text{indenyl}$ ) and  $(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3\text{-SiMe}_2\text{SiMe}_2(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3$  were prepared and characterized. A hydridotris(3,5-di-methylpyrazolyl)borate ( $\text{Tp}^*$ ) stabilized tungsten disilane complex,  $\text{Tp}^*\text{W}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$  was prepared by novel one pot synthesis, isolated and characterized. © 1998 Elsevier Science S.A.

**Keywords:** Tungsten; Disilanes; Transition metal silicon compounds

## 1. Introduction

Although numerous complexes with bonds between silicon and transition metals are known [1–4], compounds including one transition metal center bond to a chain with at least two silicon atoms are poorly explored. In early publications, only insufficient spectroscopic data ( $^{29}\text{Si}$  and  $^{13}\text{C}$  NMR) of transition metal silicon compounds are available only [5–7]. Only a few series of transition metal silyl compounds have been studied well spectroscopically like the  $(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2\text{-silyl}$  systems [8] and  $(\eta^5\text{-C}_9\text{H}_7)\text{Fe}(\text{CO})_2\text{-silyl}$  systems [9]. But the potential of silyl complexes for applications in synthesis is still far from being exploited. Recently we have reported on syntheses and properties of oligosilane derivatives of

iron and molybdenum [10]. We have referred synthesis level, analytical data and the possibility to create new materials and catalysts from transition metal silicon compounds. Our interest to prepare such compounds systematically derives from two sources. Ligand systems, like hydridotris(3,5dimethylpyrazolyl)-borate, indene and the steric pretentious ethyltetramethylcyclopentadiene should enable synthesis of transition metal substituted disilanes with different properties regarding stability and thermal decomposition pathways. Our efforts succeed new disilane derivatives of tungsten as representative transition metal.

In interrelation of alternative synthetic pathways we found a new way to build a transition metal silicon bond. We report the one pot synthesis of a tungsten disilane complex starting from  $\text{W}(\text{CO})_3(\text{CH}_3\text{CN})_3$  and  $\text{KTp}^*$ .

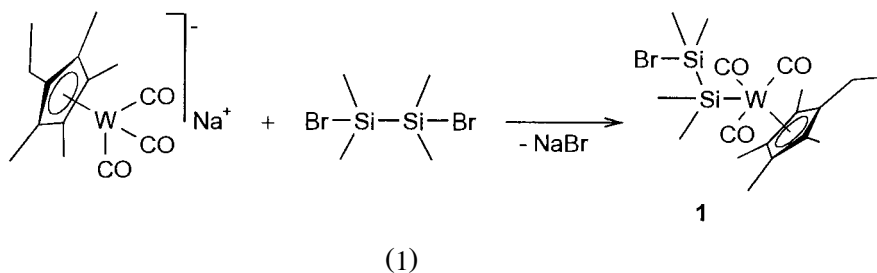
## 2. Results and discussion

A solution of  $\text{Na}[\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3]$  in THF was added dropwise to a solution of  $\text{BrMe}_2\text{SiSiMe}_2\text{Br}$  in

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pentane at ambient temperature. The solvent was removed, the residue was extracted with pentane, and the

mixture was filtered. Recrystallization from pentane gave  $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Br}$  **1** (Eq. (1)).



Structure of **1** was clearly established by spectroscopic data (see Table 1, Fig. 1). The substituted cyclopentadienyl ring is indicated by the  $^1\text{H}$  NMR spectrum which exhibits a set of two singlets at 2.04 and 2.05 ppm (methyl groups), one triplet at 1.08 ppm and one quartet at 2.32 ppm for the ethyl group. The resonances of the silicon bonded methyl groups are found at 0.14 and 0.03 ppm. The assigned structure was proofed by the  $^{13}\text{C}$  NMR spectrum (Table 1). The  $^{29}\text{Si}$  NMR spectrum of compound **1** exhibits two resonances for the disilyl ligand bonded to the metal atom (see Table 1). The IR spectrum of **1** shows three carbonyl stretching modes at 1934, 1970 and 2038  $\text{cm}^{-1}$  as

expected for compounds of the type  $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{M}(\text{CO})_3\text{L}$ .

The reaction of one equivalent of  $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3]$  with  $\text{ClSiMe}_2\text{SiSiMe}_2\text{Cl}$  in THF/pentane at ambient temperature results  $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Cl}$  (**2**, Scheme 1). The  $^1\text{H}$  NMR signals of the ethyltetramethylcyclopentadienyl ligand are observed at  $\delta = 2.01$  ppm (d, methyl) and at  $\delta = 0.97$  ppm (t) and 2.34 ppm (q) with coupling of the signals in the ethyl group. The  $^1\text{H}$  NMR resonances for the  $\text{SiMe}_2$  groups appear in a typical range at  $\delta$  values nearly zero ( $\delta = 0.13$  and 0.02 ppm). The  $^{29}\text{Si}$  NMR spectrum exhibits two resonances for the disilyl

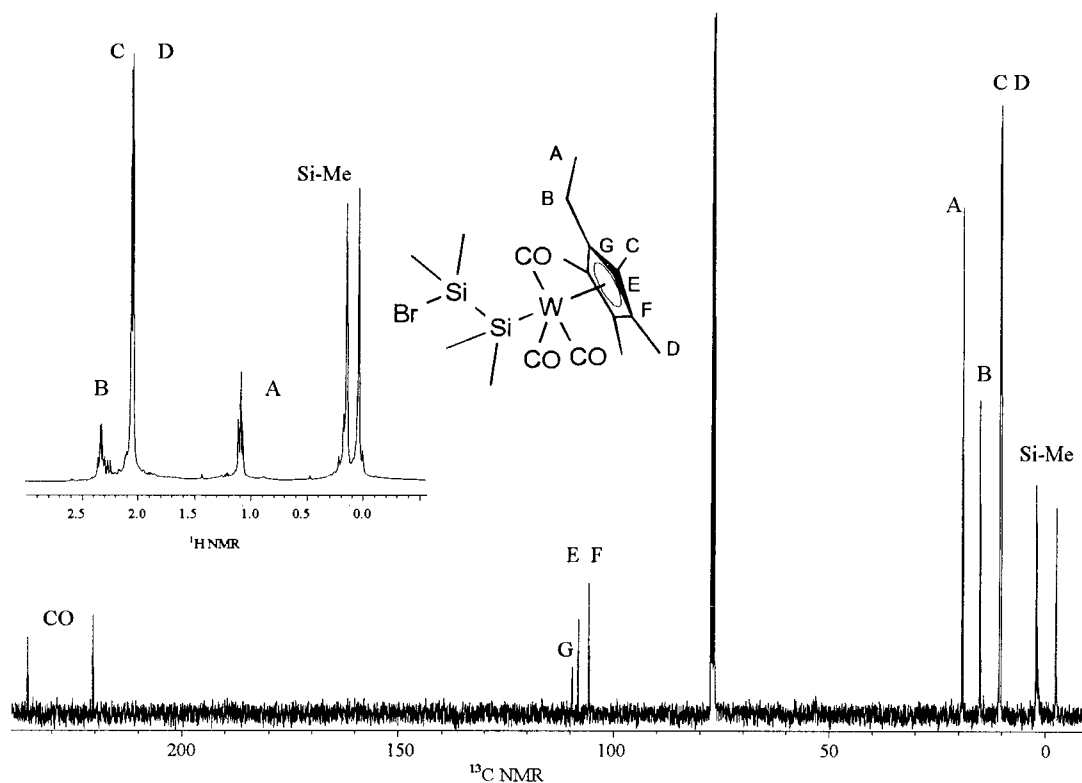


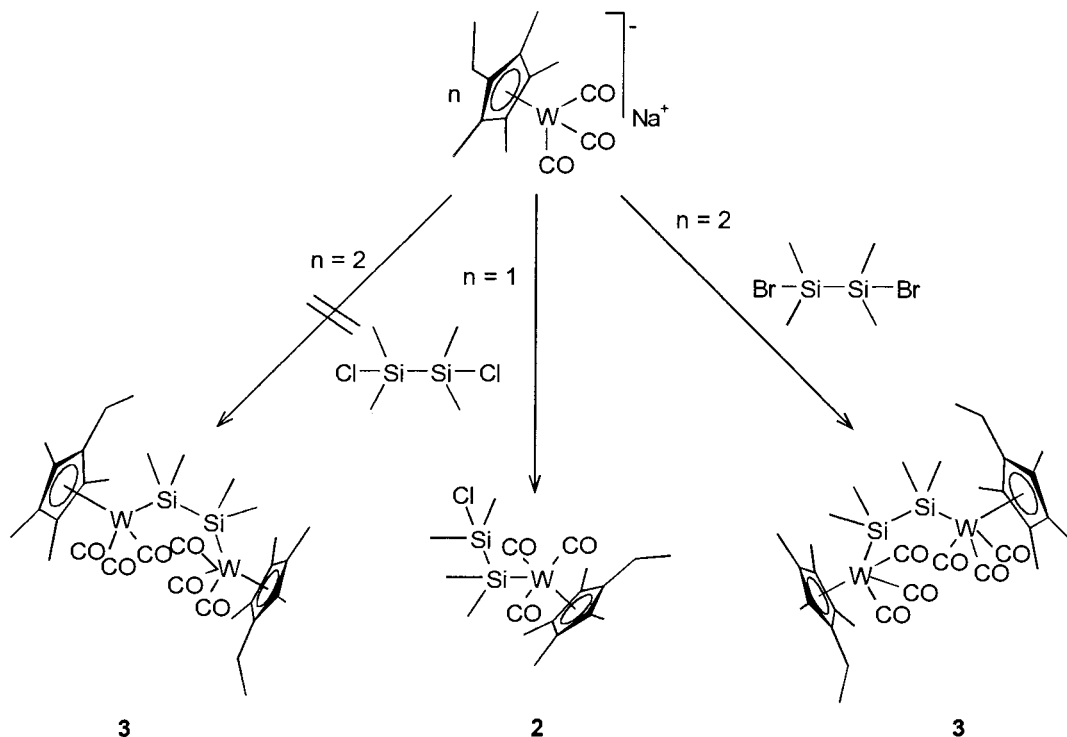
Fig. 1.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra of **1**;  $\text{CDCl}_3$  (25°C).

Table 1  
 $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{29}\text{Si}$  NMR data of the new tungsten-disilane derivatives, data in ppm are relative to  $\text{Me}_4\text{Si}$  at 0.0 ppm (25°C)

Compound	$\delta^1\text{H}$		$\delta^{13}\text{C}$			$\delta^{29}\text{Si}$
	L	Si–Me	C = O	Si–Me	L	
<b>1<sup>a</sup></b>	(L = $\eta^5\text{-C}_5\text{Me}_4\text{Et}$ ) 1.08 (t), 2.04 (s), 2.05 (s), 2.32 (q)	0.14, 0.03	225.37, 223.75	–2.52, 2.07	109.62, 108.2, 105.77, 19.16, 15.26, 10.65, 10.52	12.05, 9.96
<b>2<sup>a</sup></b>	(L = $\eta^5\text{-C}_5\text{Me}_4\text{Et}$ ) 0.97 (t), 2.01 (s), 2.34 (q)	0.13, 0.02	221.04, 229.02	–2.93, 1.99	109.24, 103.65, 102.75, 19.75, 16.38, 11.46, 11.2	14.15, 3.06
<b>4<sup>b</sup></b>	(L = $\eta^5\text{-C}_5\text{Me}_4\text{Et}$ ) 1.03 (t), 2.1 (s), 2.11 (s), 2.44 (q)	0.55, 0.62	220.44, 235.75	–0.14, 0.04	108.36, 103.46, 102.36, 19.49, 15.62, 11.09, 10.85	–10.75, –12.28
<b>3<sup>a</sup></b>	(L = $\eta^5\text{-C}_5\text{Me}_4\text{Et}$ ) 1.05 (t), 2.19 (s), 2.5 (q)	0.21	220.35, 236.11	2.4	105.73, 103.64, 102.71, 19.73, 16.37, 11.20, 10.51	3.4
<b>5<sup>a</sup></b>	(L = $\eta^5\text{-C}_9\text{H}_7$ ) 5.55 (t), 6.01 (d), 7.01 (dd), 7.50 (dd)	0.58, 0.66	214.66, 217.11	1.64, 4.20	79.45, 92.47, 109.16, 124.22, 126.70	31.1, –9.2
<b>6<sup>b</sup></b>	(L = $\eta^5\text{-C}_9\text{H}_7$ ) 5.63 (t), 5.93 (d), 7.06 (dd), 7.44 (dd)	0.21	214.82, 217.90	2.26	77.19, 93.23, 110.04, 124.75, 126.10	3.9
<b>7<sup>a</sup></b>	(L = Tp <sup>*</sup> ) 2.01, 2.46, 5.84, 2.58, 2.46, 2.38	0.14, 0.20	199.73	2.44, 2.00	153.45, 144.57, 107.38, 16.41, 12.72	0.5, 3.6

<sup>a</sup>Recorded in  $\text{CDCl}_3$ .

<sup>b</sup>Recorded in  $\text{C}_6\text{D}_6$ .



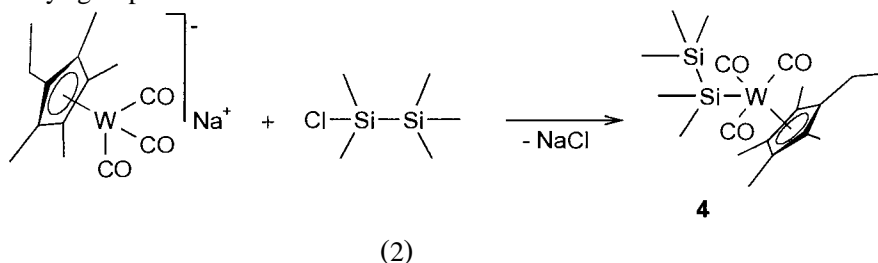
Scheme 1.

group with the metal bonded silicon atom resonance at 3.06 ppm due to the influence of the transition metal while  $\text{SiMe}_2\text{Cl}$  shows a signal at 14.15 ppm.  $^{13}\text{C}$  NMR data of **2** are in agreement with the proposed structure (Table 1). The IR spectrum shows three absorptions of  $\nu(\text{CO})$  for typical  $\text{cis-LL}'\text{M}(\text{CO})_3$  metal carbonylates.

Using an excess of sodium–tungstate–complex in comparison to  $\text{BrMe}_2\text{SiSiMe}_2\text{Br}$  we were able to synthesize the bimetallic–silyl–derivative **3** only (Scheme 1). The recrystallization from pentane gave **3** as an orange brown solid. Structure of **3** is clearly established by spectroscopic data (see Table 1). The substituted cyclopentadienyl ring is indicated by the  $^1\text{H}$  NMR spectrum which exhibits a set of two singlets at 2.19 ppm (methyl groups), one triplet at 1.05 ppm and one quartet at 2.50 ppm for the ethyl group. The resonances of the

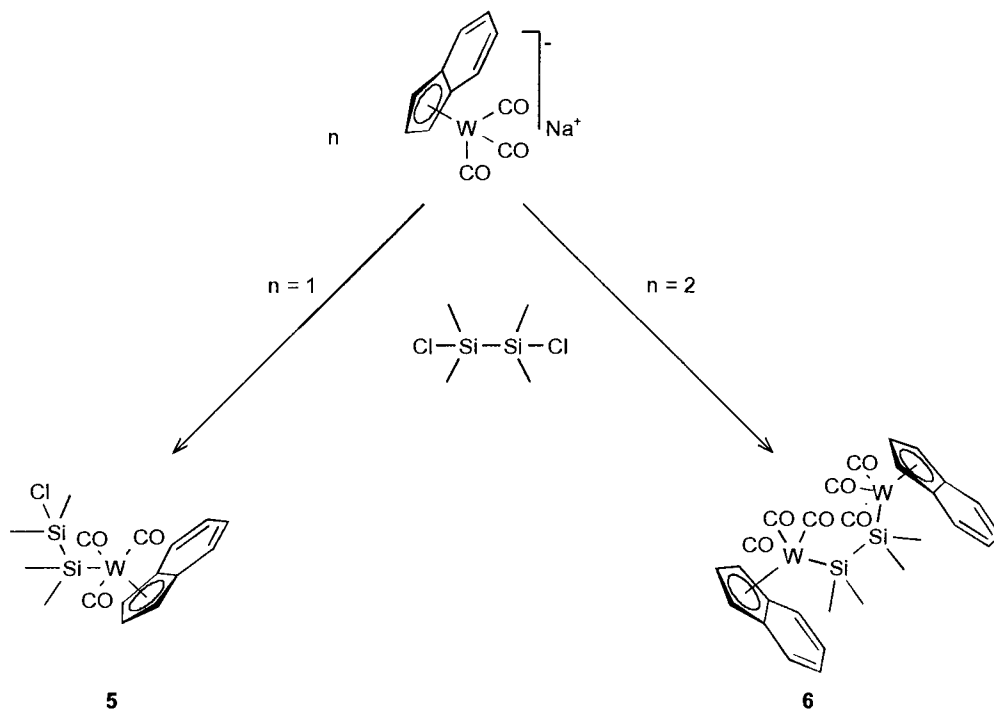
silicon bonded methyl groups are found at  $\delta = 0.21$  ppm. The complex **3** decomposes thermally at  $120^\circ\text{C}$ . We tried to prepare the bimetallic–silyl–complex **3** from 1,2-dichlorotetramethyldisilane too, but unfortunately the compound **2** and starting materials were recovered.

The compound  $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_3$  **4** was obtained in a slow reaction of chloropentamethyldisilane with  $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3]$  in a mixture of THF/pentane (Eq. (2)). After 2 days at room temperature complex **4** was isolated as pale-yellow crystalline solid. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra for this complex are quite similar to the spectra described above (see Table 1). Each one contains the typical resonances, indicating the ligands.



The  $^{29}\text{Si}$  NMR spectrum shows two signals for the disilyl group. Three typical carbonyl stretching modes in the  $2000\text{ cm}^{-1}$  region indicate  $\text{C}_s$ -symmetric- $\text{LL}'\text{M}(\text{CO})_3$  metal carbonylates.

The reaction of one equivalent of  $\text{Na}[(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3]$  with  $\text{ClMe}_2\text{SiSiMe}_2\text{Cl}$  in THF/pentane at ambient temperature results  $(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Cl}$  (**5**, Scheme 2). The com-



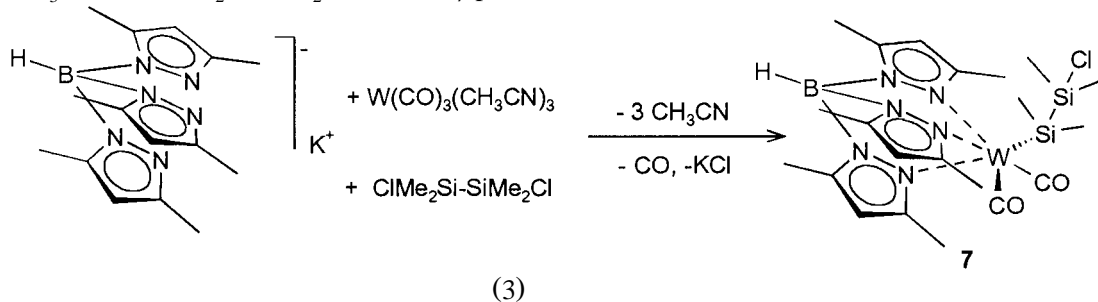
Scheme 2.

compound is obtained as yellow-brown solid from pentane. The complex **5** is prone to slow decomposition in the air. It decomposes thermally between 115 and 125°C. Due to the infrared spectra of **5** in the carbonyl stretching region, the complex should have a pseudo- $C_s$  molecular symmetry. In this case, three infrared active ( $2A' + A''$ ) carbonyl stretching modes are expected as found. The  $^1H$  NMR signals of the indenyl ligand appear at 5.55 ppm (t, 1H), 6.01 (d, 2H), 7.01 (dd, 2H) and 7.50 (dd, 2H). The  $^1H$  NMR resonances for the  $SiMe_2$  groups are observed in the typical range of  $\delta$  values (0.58 and 0.66 ppm).  $^{13}C$  NMR data of **5** are in agreement with the proposed structure (Table 1). The  $^{29}Si$  NMR spectrum exhibits two signals for the disilyl group, 31.1 ppm (the chloro bonded silicon atom resonance) and  $-9.2$  ppm (the metal bonded silicon atom) (Table 1).

The treatment of two equivalents of  $Na[(\eta^5-C_9H_7)W(CO)_3]$  with  $ClMe_2SiSiMe_2Cl$  in THF/pen-

tane at ambient temperature results **6** (Scheme 2) which is obtained as cinnamon brown solid after recrystallization from pentane. The signals of NMR measurements are listed in Table 1. The  $^1H$ ,  $^{13}C$  and  $^{29}Si$  NMR spectra of **6** are in agreement with the proposed structure and the observations are in accordance with other experimental results [9].

We were not able to synthesize compound **7** via the same route as **1–6** by salt elimination from  $K[Tp^*W(CO)_3]$  and 1,2-dichlorotetramethyldisilane. We tried to initiate the reaction in different solvents and time intervals. Unfortunately the starting materials were recovered in all cases. Starting from a mixture of  $W(CO)_3(CH_3CN)_3$  and 1,2-dichlorotetramethyldisilane in acetonitrile and dropwise addition of a solution of  $KTp^*$  in acetonitrile the preparation was more successful and yielded **7** as an ivory coloured solid after work up (Eq. (3)).



The compound **7** was identified by its  $^1H$  NMR,  $^{13}C$  NMR,  $^{29}Si$  NMR and IR spectra. The IR spectrum of

$Tp^*W(CO)_2SiMe_2SiMe_2Cl$  exhibits two bands of  $\nu(CO)$  according to  $A'$  and  $A''$  modes in point group  $C_s$ .

The B–H and C=N stretching vibrations give rise to the bands  $2506\text{ cm}^{-1}$  (B–H) and  $1493\text{ cm}^{-1}$  (C=N). Unfortunately complex **7** is very air-sensitive and subjects to hydrolysis.

All described compounds are stable against light with no evidence of decomposition in solution checked by NMR. The complexes are more stable as comparable cyclopentadienyl compounds [6].

### 3. Experimental

All preparative work and handling of the samples were carried out under Ar, using dry glassware and dry solvents. Starting materials  $\text{Si}_2\text{Me}_6$  and  $\text{W}(\text{CO})_6$  were purchased from Fluka Chemie. Syntheses of  $\text{ClMe}_2\text{SiSiMe}_3$  [11],  $\text{ClMe}_2\text{SiSiMe}_2\text{Cl}$  [12],  $\text{BrMe}_2\text{SiSiMe}_2\text{Br}$  [13],  $\text{W}(\text{CO})_3(\text{CH}_3\text{CN})_3$  [14],  $\text{KTP}^*$  [15],  $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3]$  [16] and  $\text{Na}[(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3]$  [17,18] were carried out according to known procedures. NMR spectra were recorded on Bruker MSL 200. Elemental analyses were performed on a CHN-O-RAPID (Heraeus), carbon values are lower due to formation of carbide. IR spectra were recorded from  $4000$  to  $400\text{ cm}^{-1}$  on Specord 75 IR instrument.

#### 3.1. Preparation of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Br}$ **1**

The THF solution (50 ml) containing 6 mmoles of  $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3]$  (2.64 g) was added dropwise to the solution of 6 mmoles of 1,2-dibromotetramethyldisilane (1.6 g) and 50 ml pentane. The reaction mixture was stirred at ambient temperature for 3 h. The solvents were removed under reduced pressure and the residue was extracted three times with 25 ml portions of pentane. After the solvent volume was reduced to 10 ml and the dark orange-coloured solution was cooled down to  $-76^\circ\text{C}$  **1** crystallized as a dark yellow solid. Yield: 1.2 g (51%), m.p.:  $68\text{--}70^\circ\text{C}$ , Anal. Found: C, 35.02; H, 4.55.  $\text{C}_{18}\text{H}_{29}\text{BrO}_3\text{Si}_2\text{W}$  (613) Calc.: C, 35.99, H, 4.73 IR ( $\text{cm}^{-1}$ ): 1934, 1970, 2038 (CO).

#### 3.2. Preparation of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Cl}$ **2**

The THF solution (50 ml) containing 6 mmoles of  $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3]$  (2.64 g) was added dropwise to the solution of 6 mmoles of 1,2-dichlorotetramethyldisilane (1.1 g) and 50 ml pentane. The reaction mixture was stirred at ambient temperature for 6 h. The solvents were removed under reduced pressure. The residue was extracted three times with 10 ml portions of pentane and the solvent volume was reduced to 10 ml. Finally the dark coloured solution was cooled down to  $-20^\circ\text{C}$  and **2** crystallized as a pale-brown solid. Yield: 2.2 g (65%), m.p.:  $85\text{--}87^\circ\text{C}$ , Anal. Found: C, 36.72; H,

5.02.  $\text{C}_{18}\text{H}_{29}\text{ClO}_3\text{Si}_2\text{W}$  (568.5) Calc.: C, 37.99; H, 5.10 IR ( $\text{cm}^{-1}$ ): 1918, 1932, 2010 (CO).

#### 3.3. Preparation of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3$ **3**

6 mmol (1.65 g) of  $\text{Si}_2\text{Me}_4\text{Br}_2$  were dissolved in 50 ml pentane. A solution of  $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3]$  prepared from 50 ml THF and 7.5 mmol (6.25 g) of bis-(1-ethyl-2,3,4,5-tetramethylcyclopentadienyl-tricarbonyl-tungsten) [16] was added dropwise. After stirring for 1 day the solvents were evaporated to dryness under vacuum. The orange residue was extracted with 50 ml pentane. The resulting orange solution was cooled to  $-20^\circ\text{C}$ . After one week the compound **3** was crystallized. Yield: 3.06 g (54%), m.p.:  $74^\circ\text{C}$ , Anal. Found: C, 39.96; H, 4.80.  $\text{C}_{32}\text{H}_{46}\text{O}_6\text{Si}_2\text{W}_2$  (950) Calc.: C, 40.42; H, 4.84 IR ( $\text{cm}^{-1}$ ): 1890, 1932, 1982 (CO).

#### 3.4. Preparation of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_3$ **4**

The mixture of 50 ml pentane and 6 mmoles of chloropentamethyldisilane (1 g) was added dropwise to a tetrahydrofuran solution containing 6 mmoles of  $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{W}(\text{CO})_3]$  (2.64 g). The solution was stirred at room temperature for 4 days. The solvents were removed under reduced pressure and the residue was extracted three times with 30 ml portions of pentane. The solvent was reduced to 10 ml and the orange-coloured solution was cooled to  $-20^\circ\text{C}$ . After one day the compound **4** crystallized. Yield: 2.7 g (82%), m.p.:  $82^\circ\text{C}$ , Anal. Found: C, 41.82; H, 5.83.  $\text{C}_{19}\text{H}_{32}\text{O}_3\text{Si}_2\text{W}$  (548) Calc.: C, 41.60; H, 5.83 IR ( $\text{cm}^{-1}$ ): 1902, 1925, 2011 (CO).

#### 3.5. Preparation of $(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Cl}$ **5**

To a stirred solution of 1,2-dichlorotetramethyldisilane (1.1 g, 6 mmoles) in 50 ml of pentane a solution of indenyltungsten-tricarbonyl-sodium (2.4 g, 6 mmoles) in 50 ml of THF was added slowly. The reaction mixture was stirred at room temperature for 2 h. The solvents were removed and the brown residue was extracted with 50 ml of pentane. The mixture was cooled and **5** crystallized as a honey yellow solid. Yield: 1.2 g (38%), m.p.:  $64^\circ\text{C}$ , Anal. Found: C, 34.75; H, 3.87.  $\text{C}_{16}\text{H}_{19}\text{ClO}_3\text{Si}_2\text{W}$  (534.5) Calc.: C, 35.94; H, 3.55 IR ( $\text{cm}^{-1}$ ): 1962, 1987, 2040 (CO).

#### 3.6. Preparation of $(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3$ **6**

4 mmol (0.74 g) of  $\text{Si}_2\text{Me}_4\text{Cl}_2$  were dissolved in 50 ml pentane. A solution of  $\text{Na}[(\eta^5\text{-C}_9\text{H}_7)\text{W}(\text{CO})_3]$  prepared from 50 ml THF and 2.5 mmol (1.9 g) of bis-(indenyl-tricarbonyl-tungsten) [17,18] was added dropwise. After stirring for 2 days the solvents were evaporated to dryness under vacuum. The brown residue was extracted with 30 ml pentane. The resulting dark

coloured solution was cooled to  $-20^{\circ}\text{C}$  and **6** crystallized as a cinnamon brown solid. Yield: 1.05 g (30%), decomp.:  $> 115^{\circ}\text{C}$ , Anal. Found: C, 37.68; H, 2.88.  $\text{C}_{28}\text{H}_{26}\text{O}_6\text{Si}_2\text{W}_2$  (786) Calc.: C, 38.09; H, 2.94 IR ( $\text{cm}^{-1}$ ): 1892, 1922, 1978 (CO).

### 3.7. Preparation of $\text{Tp}^* \text{W}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$ **7**

$\text{W}(\text{CO})_3(\text{CH}_3\text{CN})_3$  (0.65 g, 1.6 mmoles) and 1,2-dichlorotetramethyldisilane (0.3 g, 1.6 mmoles) were stirred in 100 ml acetonitrile at room temperature. A green solution resulted after 2 days. The solvent was reduced to 50 ml. This mixture was treated with a solution of 0.53 g (1.6 mmoles) of  $\text{KTp}^*$  in 50 ml of acetonitrile and stirred at room temperature for 1 day. The solvent was evaporated. The residue was extracted with pentane until solution did not colour yellow. After removing of the solvent the product crystallized as an ivory-coloured solid. Yield: 0.48 g (42%), Anal. Found: C, 36.29; H, 6.62; N, 12.69.  $\text{C}_{21}\text{H}_{34}\text{BClN}_6\text{O}_2\text{Si}_2\text{W}$  (687.5) Calc.: C, 36.68; H, 4.99; N, 12.25 IR ( $\text{cm}^{-1}$ ): 1840, 1893 (CO), 2506 (B–H), 1493 (C=N).

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### References

- [1] R.J.P. Corriu, B.P.S. Chauhan, G.F. Lanneau, *Organometallic* 14 (1995) 1646.
- [2] H.K. Sharma, K.H. Pannell, *Chem. Rev.* 95 (1995) 1351.
- [3] P.D. Luikis, *Chem. Soc. Rev.* (1992) 271 and references cited therein.
- [4] B.K. Nicholson, J. Simpson, W.T. Robinson, *J. Organomet. Chem.* 47 (1973) 403.
- [5] W. Malisch, *J. Organomet. Chem.* 39 (C28-C29) (1972).
- [6] W. Malisch, *J. Organomet. Chem.* 82 (1974) 185.
- [7] Z. Zhang, R. Sanchez, K.H. Pannell, *Inorg. Chem.* 34 (1995) 2605.
- [8] K.H. Pannell, S. Sharma, J. Cervantes, J.L. Mata-Mata, M.-C. Brun, F. Cervantes-Lee, *Organometallics* 14 (1995) 4269.
- [9] K.H. Pannell, S.-H. Lin, R.N. Kapoor, F. Cervantes-Lee, M. Pinon, L. Parkanyi, *Organometallics* 9 (1990) 2454.
- [10] W. Palitzsch, U. Böhme, G. Roewer, *J. Organomet. Chem.* 540 (1997) 83.
- [11] H. Sakurai, K. Tominaga, T. Watanabe, M. Kumada, *Tetrahedron Lett.* 45 (1966) 5493.
- [12] M. Kumada, M. Ishikawa, H. Sakurai, *J. Organomet. Chem.* 23 (1970) 63.
- [13] E. Hengge, S. Waldhör, *Monatshefte für Chemie* 105 (1974) 67.
- [14] R.G. Hayter, *J. Organomet. Chem.* 13 (1968) 1.
- [15] S. Trofimenko, *J. Am. Chem. Soc.* 91 (1969) 3183.
- [16] D. Feitler, G.M. Whitesides, *Inorg. Chem.* 15 (1976) 466.
- [17] R.B. King, M.B. Bisnette, *Inorg. Chem.* 4 (1965) 475.
- [18] D. Feitler, G.M. Whitesides, *Inorg. Chem.* 15 (1976) 466.