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Convenient preparation of Li[(η^5-C_5Me_5)M(CO)$_2$] (M = Ru, Fe) by the reaction of (η^5-C_5Me_5)M(CO)$_2$H with *n*-BuLi

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Abstract

The anionic ruthenium and iron complexes Li[(η^5-C_5Me_5)M(CO)$_2$] (M = Ru (**1-Ru**), Fe (**1-Fe**)) were generated by deprotonation of the transition metal hydride (η^5-C_5Me_5)M(CO)$_2$H with *n*-BuLi in tetrahydrofuran (THF). The reaction proceeded at −45 °C immediately. Reactions of **1** with various electrophiles (Me_3SiCl, $^{p}Tol_2HSiCl$, Me_2SiCl_2, $MeSiCl_3$, $SiCl_4$, $Me_3SiSiMe_2Cl$, and Ph_2GeCl_2) afforded the corresponding nucleophilic substitution products (η^5-C_5Me_5)M(CO)$_2$ER$_3$ (E = Si, Ge) via salt-elimination.

Keywords: Anionic complex; Silyl complex; Germyl complex; Deprotonation; Salt-elimination

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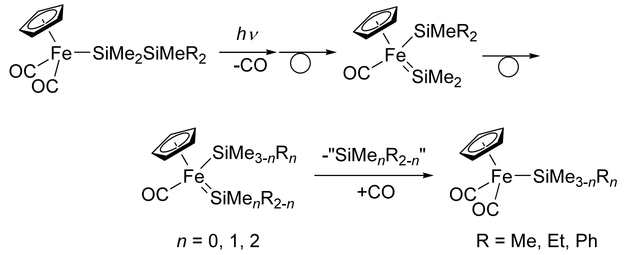
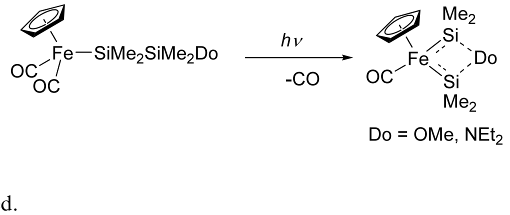
1. Introduction

The anionic transition-metal complexes [(η^5-C_5R_5)M(CO)$_2$]$^-$ (R = H, Me, M = Ru, Fe) have widely been recognized as versatile synthons for synthesis of Group 8 transition-metal complexes [1,2]. Reduction of [(η^5-C_5H_5)M(CO)$_2$]$_2$ (M = Ru [4], Fe [1a,3]) by using Na–K alloy or sodium amalgam is a conventional route to the anions [(η^5-C_5H_5)M(CO)$_2$]$^-$. Treatment of the anions with electrophiles affords complexes of the type (η^5-C_5H_5)M(CO)$_2$ER$_n$ (M = Fe, Ru, ER$_n$ = H, CR$_3$, SiR$_3$, GeR$_3$) [1,2]. Upon photolysis, these complexes can easily be converted into the coordinatively unsaturated complexes (η^5-C_5H_5)M(CO)ER$_n$ via dissociation of a carbonyl ligand. This simple reaction opens up rich chemistry. For example, irradiation of (η^5-C_5H_5)Fe(CO)$_2$SiMe$_2$SiMeR$_2$ (R = Me, Et, Ph) gives a mixture of monosilyliron complexes (η^5-C_5H_5)Fe(CO)$_2$-SiMe$_{3-n}$R$_n$ (n = 0–2), with scrambling of alkyl and aryl groups on the silicon atoms (Scheme 1) [5,6]. The reaction is assumed to proceed via the formation of a silyl(silylene)iron intermediate and the subsequent rapid intramolecular 1,3-migration from the silyl group to the silylene ligand. Actually, when the photoreaction is performed in the presence of the intramolecular donor such as alkoxy or amino groups, the donor-bridged bis(silylene) complexes are formed almost quantitatively Eq. (1) [7]. The methoxy-bridged bis(silylene) ruthenium complex can also be isolated in the same manner Eq. (2) [2c,2d].



Scheme 1.

(1)



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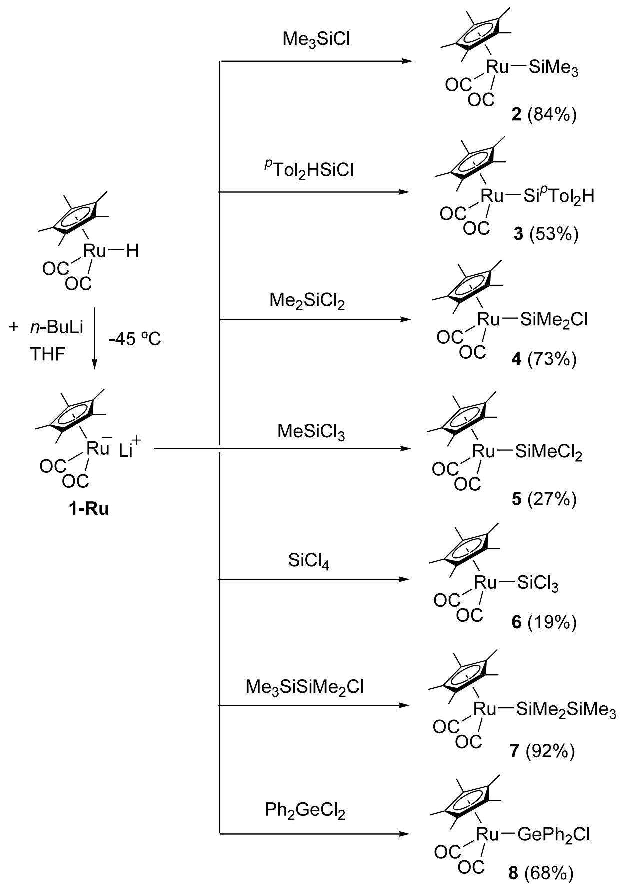
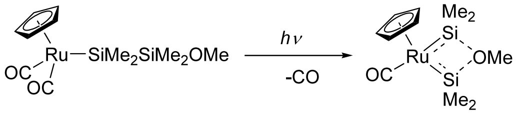


Scheme 2. Generation and reactions of Li[(η^5-C_5Me_5)Ru(CO)$_2$] (**1-Ru**).



(2)

Recently, the pentamethylcyclopentadienyl ancillary ligand (η^5-C_5Me_5) has widely been used, because it is recognized that the η^5-C_5Me_5 ligand stabilizes most of the organometallic complexes by its electron-releasing character and steric hindrance. Malisch et al. reported that the reduction of [(η^5-C_5Me_5)Ru(CO)$_2$]$_2$ with Na–K alloy proceeded almost quantitatively to generate [(η^5-C_5Me_5)Ru(CO)$_2$]$^-$ [2a]. This synthetic method of the ruthenium anion, however, has not commonly been used, because it requires very long time (3 days) for reduction and the resulting anion decomposes easily during the filtration for removal of Na–K alloy. In the previous paper, we developed a versatile method for the generation of the osmium anion [(η^5-C_5Me_5)Os(CO)$_2$]$^-$ by the reaction of (η^5-C_5Me_5)Os(CO)$_2$H with *n*-BuLi [8]. This paper describes application of the method for the generation of the ruthenium and iron anions [(η^5-C_5Me_5)M(CO)$_2$]$^-$ (M = Ru, Fe) in which the procedure such as filtration to remove excess reducing agent is not necessary.

2. Results and discussion

To a pale yellow tetrahydrofuran (THF) solution of the hydridoruthenium complex (η^5-C_5Me_5)Ru(CO)$_2$H was added 1.1 equivalents of *n*-BuLi at -45 °C. The color of the solution changed to greenish brown immediately. Subsequent addition of excess Me_3SiCl in THF gave a red solution from which trimethylsilyl complex (η^5-C_5Me_5)Ru(CO)$_2$SiMe$_3$ (**2**) was isolated in 84% yield. Similarly, the reactions of the greenish brown solution with various chlorosilanes afforded the corresponding silyl complexes (**3**–**6**) (Scheme 2). In these reactions, the first step is considered to be deprotonation of the ruthenium hydride leading to generation of the ruthenium anion [(η^5-C_5Me_5)Ru(CO)$_2$]$^-$ (**1-Ru**). The elemental analysis and spectroscopic data of the products **2**–**6** (Table 1) fully support the structures illustrated in Scheme 2. In the reactions of the ruthenium anion **1-Ru** with $MeSiCl_3$ and $SiCl_4$, the formation yields of the silylruthenium complexes **5** and **6** were quite low. In these cases, formation of a considerable amount of (η^5-C_5Me_5)Ru(CO)$_2$Cl was observed.

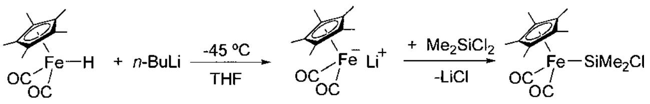
Table 1
Elemental analysis, IR, and mass spectral data for ruthenium and iron complexes

Complex	Anal. Found (Calc.) (%)		IR (cm^{-1})	Mass (EI, 70 eV), *m*/*z*
	C	H	(νCO)	
Cp*Ru(CO)$_2$SiMe$_3$ (**2**)	49.33 (49.29)	6.39 (6.62)	1992, 1923	366 (M$^+$, 59), 292 (M$^+$–SiMe$_3$, 100)
Cp*Ru(CO)$_2$Si^{p}Tol$_2$H (**3**)	61.71 (62.00)	5.82 (6.00)	1992, 1932	504 (M$^+$, 3), 447 (M$^+$–2CO–H, 100)
Cp*Ru(CO)$_2$SiMe$_2$Cl (**4**)	43.90 (43.57)	5.36 (5.49)	1996, 1944	386 (M$^+$, 24), 330 (M$^+$–2CO, 100)
Cp*Ru(CO)$_2$SiMeCl$_2$ (**5**)	38.57 (38.43)	4.44 (4.46)	2015, 1963	406 (M$^+$, 20), 236 (M$^+$–SiMeCl$_2$–2CO, 100)
Cp*Ru(CO)$_2$SiCl$_3$ (**6**)	34.64 (33.77)	3.52 (3.52)	2035, 1984	426 (M$^+$, 16), 236 (M$^+$–SiCl$_3$–2CO, 100)
Cp*Ru(CO)$_2$SiMe$_2$SiMe$_3$ (**7**)	47.88 (48.22)	7.18 (7.09)	1990, 1932	424 (M$^+$, 24), 351 (M$^+$–SiMe$_3$, 100)
Cp*Ru(CO)$_2$GePh$_2$Cl (**8**)	51.94 (51.98)	4.47 (4.54)	1996, 1942	554 (M$^+$, 12), 526 (M$^+$–CO, 100)
Cp*Fe(CO)$_2$SiMe$_2$Cl (**9**)	49.46 (49.35)	6.07 (6.21)	1978, 1928	340 (M$^+$, 26), 284 (M$^+$–2CO, 100)

Disilanyl complex (η^5-C_5Me_5)Ru$(CO)_2SiMe_2SiMe_3$ (**7**) can also be synthesized in 92% yield by the reaction of **1-Ru** with $Me_3SiSiMe_2Cl$. The ^{29}Si resonances of the disilanyl ligand appear at -1.7 and -13.1 ppm.

Treatment of the ruthenium anion with Ph_2GeCl_2 afforded germylruthenium complex **8** as shown in Scheme 2. The elemental analysis and spectroscopic data are consistent with the formation of the germyl complex **8**.

The silyliron complex (η^5-C_5Me_5)Fe$(CO)_2SiMe_2Cl$ was obtained by a procedure similar to that for the ruthenium analog. Thus, treatment of the hydridoiron complex (η^5-C_5Me_5)Fe$(CO)_2H$ with 1.1 equivalents of *n*-BuLi at -45 °C in THF gave a deep red solution of **1-Fe**. Subsequent addition of Me_2SiCl_2 and workup gave the silyliron complex (η^5-C_5Me_5)Fe$(CO)_2SiMe_2Cl$ (**9**) in 89% yield (Eq. (3)).



1-Fe   9 (89%)

(3)

We reported previously the generation and characterization of osmium anion $[(\eta^5\text{-}C_5Me_5)Os(CO)_2]^-$ by the reaction of (η^5-C_5Me_5)Os$(CO)_2H$ with *n*-BuLi, which reacted with various electrophiles to give the nucleophilic substitution products [8]. We applied this preparative method of $[(\eta^5\text{-}C_5Me_5)Os(CO)_2]^-$ for synthesis of the ruthenium and iron analogs, and succeeded in removing the following difficulties accompanying the conventional methods.

1. Preparation of $[(\eta^5\text{-}C_5Me_5)M(CO)_2]^-$ (M = Ru, Fe) by a conventional method, i.e. reduction of the dimer $[(\eta^5\text{-}C_5Me_5)M(CO)_2]_2$ with metal reducing agents takes long time [2–4 h (M = Fe, potassium mirror or sodium amalgam [1e,9])], 3 days (M = Ru, Na–K mirror [2a]). In contrast, the reaction of (η^5-C_5Me_5)M$(CO)_2H$ with *n*-BuLi proceeds instantaneously at -45 °C.
2. In the conventional method, the reducing agent such as Na–K alloy should be removed before the anion $[(\eta^5\text{-}C_5Me_5)M(CO)_2]^-$ is served for the subsequent reaction. A usual method is the filtration of the extremely air- and moisture-sensitive solution of the anion. But, this procedure sometimes leads to decomposition of the anion by air-oxidation to give the dimer $[(\eta^5\text{-}C_5Me_5)M(CO)_2]_2$. A special experimental skill is required for the conventional method. In contrast, the reaction of (η^5-C_5Me_5)M$(CO)_2H$ with *n*-BuLi requires no subsequent procedure such as filtration, and the reaction mixture can immediately be used for the next reaction.

In conclusion, a new convenient route to the ruthenium and iron anions $[(\eta^5\text{-}C_5Me_5)M(CO)_2]^-$ (M = Ru (**1-Ru**), Fe (**1-Fe**)) by the reaction of (η^5-C_5Me_5)M$(CO)_2H$ with *n*-BuLi have been established. Some new ruthenium(II) and iron(II) complexes with a η^5-C_5Me_5 ancillary ligand (η^5-C_5Me_5)M$(CO)_2ER_3$ were prepared by the reactions of the anions **1-Fe** and **1-Ru** with electrophiles.

3. Experimental

3.1. General comments

All manipulations were carried out under a dry nitrogen or argon atmosphere. Reagent-grade THF, toluene, hexane, and pentane were distilled from sodium-benzophenone immediately before use. Benzene-d_6 was dried over molecular sieves 4A. The compounds (η^5-C_5Me_5)Ru$(CO)_2H$ [10], (η^5-C_5Me_5)Fe$(CO)_2H$ [11], $Me_3SiSiMe_2Cl$ [12], H^pTol_2SiCl [13], and Ph_2GeCl_2 [14] were prepared according to published procedures. All other chemicals were used as received. All NMR spectra were recorded on a Bruker ARX-300 spectrometer. The IR spectra were recorded on Bruker IFS66v and Horiba FT-200 spectrophotometers. Mass spectra were obtained with a JEOL JMS-HX110 spectrometer. The elemental analysis data and IR and MS data for compounds were summarized in Table 1.

3.2. Synthesis of (η^5-C_5Me_5)Ru$(CO)_2SiMe_3$ (2)

To a THF (50 ml) solution of (η^5-C_5Me_5)Ru$(CO)_2H$ (846 mg, 2.88 mmol) was added *n*-BuLi (1.58 M, 2.7 ml, 4.3 mmol) at -45 °C (CH_3CN–liquid N_2 bath). After stirring the mixture for 30 min, a THF (20 ml) solution of Me_3SiCl (472 mg, 4.34 mmol) was added to the solution at -45 °C. The mixture was stirred for 30 min at -45 °C and then warmed up to room temperature (r.t.) and stirred for 1 h. After removal of volatiles under vacuum, the residue was extracted with hexane and the extract was filtered through the Celite pad. Removal of solvent from the filtrate under reduced pressure afforded a brown powder of (η^5-C_5Me_5)Ru$(CO)_2SiMe_3$ (**2**; 880 mg, 2.41 mmol, 84%). Analytically pure sample was obtained by recrystallization from hexane at -30 °C. ^{1}H-NMR (300 MHz, C_6D_6) δ 0.60 (s, 9H, $SiMe_3$), 1.60 (s, 15H, C_5Me_5). $^{13}C\{^1H\}$-NMR (75.5 MHz, C_6D_6) δ 6.8 ($SiMe_3$), 10.3 (C_5Me_5), 99.2 (C_5Me_5), 204.7 (2CO). $^{29}Si\{^1H\}$-NMR (59.6 MHz, C_6D_6) δ 25.7.

3.3. Synthesis of (η^5-C_5Me_5)Ru$(CO)_2Si^pTol_2H$ (3)

Compound **3** was synthesized by a similar method to that for **2**, using (η^5-C_5Me_5)Ru$(CO)_2H$ (680 mg, 2.32 mmol) and $^pTol_2SiHCl$ (1.20 g, 4.86 mmol). The residue was extracted with hexane (60 ml) and the extract was filtered through a Celite pad. The filtrate was evaporated to dryness and the residue was recrystallized from pentane to give $C_5Me_5Ru(CO)_2Si^{p}Tol_2H$ (**3**) (613 mg, 1.22 mmol, 53%) as pale yellow crystals. ^{1}H-NMR (300 MHz, C_6D_6) δ 1.52 (s, 15H, $C_5\mathit{Me}_5$), 2.11 (s, 6H, p-$C_6H_5\mathit{CH}_3$), 5.56 (s, 1H, SiH), 7.08–7.10, 7.89–7.91 (AB q, $J = 8$ Hz, 4H × 2, Ar). ^{13}C{^{1}H}-NMR (75.5 MHz, C_6D_6) δ 9.71 ($C_5\mathit{Me}_5$), 21.4 (p-$C_6H_5\mathit{C}H_3$), 99.2 (C_5Me_5), 129.1, 135.6, 137.6, 139.0 (Ar), 204.3 (2CO). ^{29}Si{^{1}H}-NMR (59.6 MHz, C_6D_6) δ 23.9. IR (KBr pellet) 2052 cm^{-1} (νSi–H).

3.4. Synthesis of $(\eta^5\text{-}C_5Me_5)Ru(CO)_2SiMe_2Cl$ (4)

Compound **4** was synthesized by a similar method to that for **2**, using $(\eta^5\text{-}C_5Me_5)Ru(CO)_2H$ (496 mg, 1.69 mmol) and Me_2SiCl_2 (662 mg, 5.13 mmol). The reaction mixture was subjected to a silica gel flash column and elution was carried out with a toluene–hexane (1:2) mixture. Evaporation of the fraction to dryness afforded colorless crystals of **4** in 73% yield (474 mg, 1.23 mmol). ^{1}H-NMR (300 MHz, C_6D_6) δ 0.93 (s, 6H, $SiMe_2$), 1.58 (s, 15H, C_5Me_5). ^{13}C{^{1}H}-NMR (75.5 MHz, C_6D_6) δ 10.0 ($SiMe_2$), 12.1, ($C_5\mathit{Me}_5$), 100.3 (C_5Me_5), 202.9 (2CO). ^{29}Si{^{1}H}-NMR (59.6 MHz, C_6D_6) δ 69.9.

3.5. Synthesis of $(\eta^5\text{-}C_5Me_5)Ru(CO)_2SiMeCl_2$ (5)

Compound **5** was synthesized by a similar method to that for **2**, using $(\eta^5\text{-}C_5Me_5)Ru(CO)_2H$ (400 mg, 1.36 mmol) and $MeSiCl_3$ (0.32 g, 2.5 mmol). The ^{1}H-NMR spectrum revealed that the resulting mixture contains **5** and $(\eta^5\text{-}C_5Me_5)Ru(CO)_2Cl$ in the molar ratio of 1:2.7. The reaction mixture was subjected to a silica gel flash column and elution was carried out with a toluene–hexane (1:9) mixture. Removal of solvent from the fraction afforded a white powder of **5** in 27% yield (150 mg, 0.369 mmol). Recrystallization from hexane at −30 °C gave colorless needles of **5**. ^{1}H-NMR (300 MHz, C_6D_6) δ 1.26 (s, 3H, SiMe), 1.54 (s, 15H, C_5Me_5). ^{13}C{^{1}H}-NMR (75.5 MHz, C_6D_6) δ 9.7 ($C_5\mathit{Me}_5$), 17.9 (SiMe) 101.0 (C_5Me_5), 201.2 (2CO). ^{29}Si{^{1}H}-NMR (59.6 MHz, C_6D_6) δ −21.4.

3.6. Synthesis of $(\eta^5\text{-}C_5Me_5)Ru(CO)_2SiCl_3$ (6)

Compound **6** was synthesized by a similar method to that for **2**, using $(\eta^5\text{-}C_5Me_5)Ru(CO)_2H$ (336 mg, 1.15 mmol) and $SiCl_4$ (0.44 g, 2.6 mmol). The ^{1}H-NMR spectrum revealed that the resulting mixture contains **6** and $(\eta^5\text{-}C_5Me_5)Ru(CO)_2Cl$ in the molar ratio of 1:4. The reaction mixture was placed on a silica-gel flash column and elution was carried out with a toluene–hexane (1:4) mixture. Removal of solvent from the fraction afforded a white powder of **6** (95 mg, 0.22 mmol) in 19% yield. Recrystallization from hexane at −30 °C gave colorless needles of **6**. ^{1}H-NMR (300 MHz, C_6D_6) δ 1.44 (s, 15H, C_5Me_5). ^{13}C{^{1}H}-NMR (75.5 MHz, C_6D_6) δ 9.5 ($C_5\mathit{Me}_5$), 101.2 (C_5Me_5), 199.6 (2CO).

3.7. Synthesis of $(\eta^5\text{-}C_5Me_5)Ru(CO)_2SiMe_2SiMe_3$ (7)

Compound **7** was synthesized by a similar method to that for **2**, using $(\eta^5\text{-}C_5Me_5)Ru(CO)_2H$ (658 mg, 2.25 mmol) and $Me_3SiSiMe_2Cl$ (562 mg, 3.38 mmol). Volatiles were removed under reduced pressure from the resulting mixture. The residue was extracted with hexane and the extract was filtered through the Celite pad. Removal of the solvent from the extract under reduced pressure afforded an orange oil of **7** (880 mg, 2.08 mmol, 92% yield). Further purification by medium-pressure liquid chromatography on silica-gel (hexane as eluent) afforded an analytical pure sample of **7** (colorless oil). ^{1}H-NMR (300 MHz, C_6D_6) δ 0.37 (s, 6H, $SiMe_2$), 0.63 (s, 9H, $SiMe_3$), 1.60 (s, 15H, C_5Me_5). ^{13}C{^{1}H}-NMR (75.5 MHz, C_6D_6) δ 0.5, 2.7 (SiMe), 10.4 ($C_5\mathit{Me}_5$), 99.9 (C_5Me_5), 204.7 (2CO). ^{29}Si{^{1}H}-NMR (59.6 MHz, C_6D_6) δ −13.1, −1.7.

3.8. Synthesis of $(\eta^5\text{-}C_5Me_5)Ru(CO)_2GePh_2Cl$ (8)

Compound **8** was synthesized by a similar method to that for **2**, using $(\eta^5\text{-}C_5Me_5)Ru(CO)_2H$ (497 mg, 1.69 mmol) and Ph_2GeCl_2 (0.80 g, 2.69 mmol). The reaction mixture was placed on a silica-gel flash column and elution was carried out with a toluene–hexane (1:1) mixture. Removal of solvent from the fraction afforded a light yellow powder of **8** (639 mg, 1.15 mmol) in 68% yield. ^{1}H-NMR (300 MHz, C_6D_6) δ 1.48 (s, 15H, C_5Me_5). 7.09, 7.22, 7.97 (m, Ph_2). ^{13}C{^{1}H}-NMR (75.5 MHz, C_6D_6) δ 9.8 ($C_5\mathit{Me}_5$), 100.1 (C_5Me_5), 128.2, 128.7, 133.3, 146.6 (Ph_2), 202.7 (2CO).

3.9. Synthesis of $(\eta^5\text{-}C_5Me_5)Fe(CO)_2SiMe_2Cl$ (9)

To a THF (20 ml) solution of $(\eta^5\text{-}C_5Me_5)Fe(CO)_2H$ (536 mg, 2.16 mmol) was added n-BuLi (1.49 M, 1.50 ml, 2.2 mmol) at −45 °C (CH_3CN–liquid N_2 bath). After stirring the mixture for 10 min, a THF (5 ml) solution of Me_2SiCl_2 (520 mg, 4.03 mmol) was added to the solution at −45 °C. The mixture was stirred for 30 min at −45 °C and then was warmed up to r.t. and stirred for 1 h. After removal of volatiles under vacuum, the residue was extracted with toluene and the extract was filtered through a Celite pad. The filtrate was evaporated to dryness to give an orange powder of **9** (655 mg, 1.92 mmol, 89%). ^{1}H-NMR (300 MHz, C_6D_6) δ 0.92 (s, 6H, $SiMe_2$), 1.47 (s, 15H, C_5Me_5). ^{13}C{^{1}H}-NMR (75.5 MHz, C_6D_6) δ 9.5 ($SiMe_2$), 12.0, ($C_5\mathit{Me}_5$), 95.9 (C_5Me_5), 216.1 (2CO). ^{29}Si{^{1}H}-NMR (59.6 MHz, C_6D_6) δ 87.7.

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