

THE PREPARATION OF A NEW SERIES OF BRIDGED TRINUCLEAR COMPLEXES BY REACTION OF η^5 -(C₅H₄Li) COMPOUNDS WITH ALKYLTRICHLOROSILANES

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Summary

Several new bridged trinuclear complexes of the general formula $\text{RSi}[\eta^5\text{-C}_5\text{H}_4\text{M}(\text{CO})_n\text{R}^1]_3$, have been prepared in good yield from the reaction of the lithiated compounds $(\eta^5\text{-C}_5\text{H}_4\text{Li})\text{M}(\text{CO})_n\text{R}^1$ ($\text{M} = \text{Mn, W, Fe}$; $\text{R}^1 = \text{Me, Et, and benzyl}$) with various alkyl trichlorosilanes. The crystal and molecular structure of $\text{MeSi}[\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{C}_2\text{H}_5]_3$ is presented. In addition, some limitations of the direct cyclopentadienyl ring metalation are discussed.

Introduction

In most cases bridged-cyclopentadienylmetal complexes are prepared by synthesizing the bridging ligand, followed by attachment of the appropriate number of metals [1]. In cases where the bridging ligand is unsymmetrical, selective complexation of one site has been achieved. This latter approach has been used quite successfully for the following ligands, $\text{Li}[(\text{C}_5\text{H}_4)\text{CH}_2\text{CH}_2\text{PPh}_2]$ [2], $\text{Li}[(\text{C}_5\text{H}_4)\text{CH}_2\text{CH}_2\text{AsPh}_2]$ [2], $\text{Li}[(\text{C}_5\text{H}_4)\text{CH}_2\text{CH}_2\text{CH}_2(\text{C}_5\text{H}_5\text{N})]$ [2], $\text{Li}[(\text{C}_5\text{H}_4)\text{PPh}_2]$ [3], $\text{Li}[(\text{C}_5\text{H}_4)\text{SiMe}_2\text{CH}_2\text{PPh}_2]$ [4], and $\text{Tl}[(\text{C}_5\text{H}_4)\text{PPh}_2]$ [5].

Ullmann coupling reactions of $\eta^5\text{-(C}_5\text{H}_4\text{I)M}$, where $\text{M} = \text{Mn}(\text{CO})_3$ [6], $\text{Co}(\eta^4\text{-C}_4\text{Ph}_4)$ [7], $\text{Fe}(\text{C}_5\text{H}_5)$ [8], and $\text{Ru}(\text{C}_5\text{H}_5)$ [9], have yielded bimetallic $\eta^5\text{:}\eta^5$ -fulvalene complexes. In a related reaction, treatment of $(\eta^5\text{-C}_5\text{H}_4\text{Br})\text{Co}(\text{CO})_2$ first with $n\text{-BuLi}$, then CoCl_2 , and finally H_2O , gave the coupled product, $(\eta^5\text{:}\eta^5\text{-fulvalene})\text{Co}_2(\text{CO})_4$, in 28% yield [10]. The X-ray structure was reported for the dicobalt complex.

Bis(cyclopentadienyl) bridging ligands are useful because of their robust nature, producing bimetallic complexes which are stable under a variety of reaction condi-

tions [11]. As a consequence, dinuclear complexes containing a bridged-bis(cyclopentadienyl) ligand are attractive systems for studying mechanisms involving two metal reaction sites [1e,1f].

Recently, direct metalation of the η^5 -cyclopentadienyl ring in several transition-metal (TM) complexes containing carbonyls has appeared in the literature [12]. By utilizing metalated η^5 -cyclopentadienyl-TM complexes we have prepared a new series of trinuclear compounds in which all three metals are linked by an alkylsilane. The crystal and molecular structure of $\text{CH}_3\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{CH}_2\text{CH}_3]_3$ (**1c**) has been completed and is also reported.

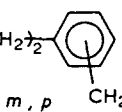
Results and discussion

Synthesis and characterization

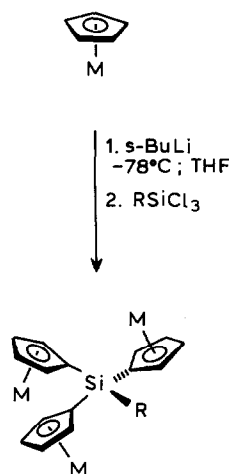
Reaction of η^5 -cyclopentadienyl complexes with *s*-BuLi for 20 min, at -78°C in THF, produces the corresponding η^5 -cyclopentadienyl-lithiated compounds, $\eta^5\text{-(C}_5\text{H}_4\text{Li)MCCO)}_n\text{R}^1$, which can then be treated with a variety of alkyltrichlorosilanes to afford a new series of bridged trinuclear complexes (Scheme 1). The trinuclear compounds are obtained in good to excellent yield after purification by column chromatography.

In the synthesis of **1a–1c** we find that use of *n*-BuLi results in the recovery of the starting cyclopentadienyl compound and low conversion to the trinuclear product. In contrast, both the tungsten and manganese systems are observed to lithiate cleanly with *n*-BuLi at -78°C in 30 min. This difference in reactivity most likely arises from kinetic factors as there is little doubt *n*-BuLi is basic enough, in a thermodynamic sense [12g,12h], to deprotonate the iron systems.

The new trinuclear complexes have spectroscopic data consistent with the proposed structures. The infrared spectra in the carbonyl region are very similar to their

Product	R	M	Yield (%)
1a	$-\text{CH}_3$	$\text{Fe}(\text{CO})_2\text{Me}$	87
1b		$\text{Fe}(\text{CO})_2\text{Bn}^a$	83
1c		$\text{Fe}(\text{CO})_2\text{Et}$	78
2		$\text{Mn}(\text{CO})_3$	93
3a		$\text{W}(\text{CO})_3\text{Me}$	90
3b		$\text{W}(\text{CO})_3\text{Bn}$	91
4	$-(\text{CH}_2)_3\text{Cl}$	$\text{Fe}(\text{CO})_2\text{Me}$	85
5	$-(\text{CH}_2)_2$  CH_2Cl <i>m, p</i>	$\text{Fe}(\text{CO})_2\text{Me}$	70

^a Bn = CH_2Ph



SCHEME 1

parent analogues and their ^1H and ^{13}C NMR spectra are in agreement with previously reported silyl-bridged bis(cyclopentadienyl) compounds [11a].

In the ^1H NMR spectra, all of the trinuclear complexes exhibit an AA'BB' pattern for the cyclopentadienyl ring resonances. These data indicate that rotation about the cyclopentadienyl-silicon bond is rapid on the NMR time scale at 25°C ; otherwise, all four cyclopentadienyl ring protons would be visible as distinct signals. Even at -80°C , complex **2** showed no signs of line broadening and retained the AA'BB' pattern.

Compounds **1**–**3** are crystalline solids except for **3b** which has only been obtainable as a yellow oil. This is quite surprising because of the high crystallinity associated with the $\eta^5\text{-C}_5\text{H}_5\text{W(CO)}_3\text{CH}_2\text{Ph}$ starting material. Attempts to crystallize **3b** by slow diffusion at -25°C have been unsuccessful. On the other hand, **1b**, is a microcrystalline solid that is virtually insoluble in chloroform, methylene chloride, tetrahydrofuran, acetonitrile, and benzene! The reasons for the dramatic change in the physical properties of **1b** and **3b** from their respective starting mononuclear complexes is not clear at this time.

The preparation of complexes **4** and **5** demonstrates reaction site selectivity as an important aspect. In both cases, the chloride on silicon is displaced by the lithiated complex with high selectivity over the alkyl or benzylic chloride. Hence, we have the ability to prepare trinuclear complexes with a useful functional group tethered to the complex.

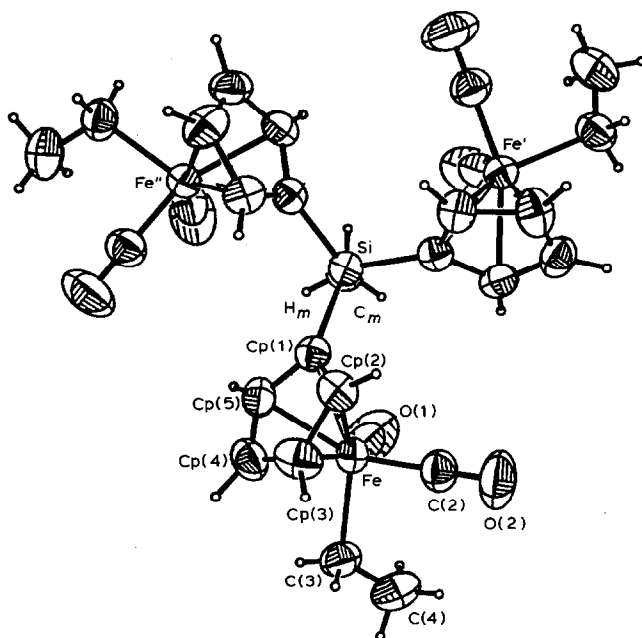


Fig. 1. Perspective ORTEP drawing of $\text{CH}_3\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{Fe(CO)}_2\text{CH}_2\text{CH}_3]_3$ (**1c**) viewed along the crystallographic C_3 axis which passes through Si and C_m . All non-hydrogen atoms are represented by thermal vibration ellipsoids drawn to encompass 50% of the electron density; hydrogen atoms are represented by arbitrarily small spheres which are in no way representative of their true thermal motion. Atoms labeled with single (') and double (") primes are related to those labeled without primes by the symmetry operations (z, x, y) and (y, z, x) , respectively. Atom C(1) and all hydrogens except H_m are not labeled.

TABLE 1

BOND LENGTHS (Å) AND BOND ANGLES (°) IN $\text{CH}_3\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{CH}_2\text{CH}_3]_3$ ^{a,b}

Fe–C(1)	1.739(3)	C(1)–O(1)	1.134(5)
Fe–C(2)	1.751(5)	C(2)–O(2)	1.141(6)
Fe–C(3)	2.087(5)	C(3)–C(4)	1.502(7)
Fe–Cp(1)	2.125(4)	Cp(1)–Cp(2)	1.406(5)
Fe–Cp(2)	2.120(4)	Cp(1)–Cp(5)	1.435(6)
Fe–Cp(3)	2.122(4)	Cp(2)–Cp(3)	1.423(6)
Fe–Cp(4)	2.122(5)	Cp(3)–Cp(4)	1.387(6)
Fe–Cp(5)	2.101(4)	Cp(4)–Cp(5)	1.402(6)
Fe–Cg ^b	1.745(–)	Si–Cp(1)	1.860(7)
Fe...Fe' ^c	6.118(1)	Si–C _m	1.868(4)
		C _m –H _m	0.96(4)
C(1)FeC(2)	96.1(2)	C _m SiCp(1)	111.7(1)
C(1)FeC(3)	85.8(2)	Cp(1)SiCp(1') ^c	107.1(1)
C(2)FeC(3)	89.2(2)		
CgFeC(1) ^b	128.8(–)	Cp(5)Cp(1)Si	128.4(3)
CgFeC(2) ^b	124.2(–)	Cp(2)Cp(1)Si	126.5(3)
CgFeC(3) ^b	120.4(–)	Cp(5)Cp(1)Cp(2)	105.2(3)
		Cp(1)Cp(2)Cp(3)	109.7(4)
		Cp(2)Cp(3)Cp(4)	107.5(4)
FeC(1)O(1)	178.0(4)	Cp(3)Cp(4)Cp(5)	108.4(4)
FeC(2)O(2)	178.8(5)	Cp(4)Cp(5)Cp(1)	109.3(3)
FeC(3)C(4)	118.0(4)	SiC _m H _m	111(2)
		H _m C _m H _m ' ^c	108(2)

^a Numbers in parentheses are the estimated standard deviations in the last significant digit. Atoms are labeled in agreement with Fig. 1. ^b Cg, refers to the center-of-gravity for the five-membered ring of the cyclopentadienyl group. ^c Atoms labeled with a prime (') are related to those without by the symmetry operation *z*, *x*, *y*.

Crystal and molecular structure of $\text{MeSi}[\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{CH}_2\text{CH}_3]_3$ (**1c**)

To more fully characterize these new bridged trinuclear complexes we have completed a single crystal X-ray diffraction study on complex **1b**. An ORTEP perspective drawing of **1c** is displayed in Fig. 1 along with the labeling scheme employed. Complex **1c** possesses a *C*₃ axis through the C_m–Si bond giving the structure a “propeller” like appearance. For each iron center, the bulky ethyl group is directed away from the center of the trinuclear complex, undoubtedly to avoid steric interactions with the silyl methyl moiety. The coordination of ligands about the iron centers is similar to mononuclear [13] and binuclear [11b] complexes previously characterized by structural studies. Table 1 gives a listing of bond angles and bond lengths for **1c**.

Attempted metalations

Reaction of $\eta^5\text{-C}_5\text{H}_5\text{Fe}(\text{CO})(\text{PPh}_3)\text{CH}_3$ (**6**) with either one equiv of *n*-BuLi, *t*-BuLi, or *n*-BuLi/TMEDA for 1 h at –78°C, followed by a quench with excess methyl iodide, leads only to recovery of **6**. Similar results were obtained when attempts were made to deprotonate $(\eta^5\text{-C}_5\text{H}_5)_2\text{Zr}(\text{CH}_3)_2$. In a related system, $\eta^5\text{-C}_5\text{H}_5\text{Re}(\text{NO})(\text{PPh}_3)\text{CH}_3$ (**7**), Gladysz and co-workers [14] noted that for complete cyclopentadienyl metalation 2.1 equiv of *n*-BuLi/TMEDA were required. Unfortunately, in the preparation of the trinuclear complexes above, excess alkyl-

lithium reagents cannot be tolerated *. Consequently, we have not examined the possibility of metalating **6** using excess *n*-BuLi-TMEDA.

Conclusion

Reaction of lithiated cyclopentadienyl complexes with alkyl trichlorosilanes produces, in good yield, a new class of bridged trinuclear complexes. Both early and late transition metals as well as first and third row transition metals work well in the synthetic strategy. Current work is focused on preparing mixed-metal systems by exploiting the selective substitution of a silicon chloride over an alkyl halide [15].

Experimental

General

All reactions and subsequent manipulations were carried out using standard Schlenk techniques under a purified nitrogen atmosphere. Solvents were routinely dried and degassed by standard procedures and stored under nitrogen [16].

Spectroscopic measurements utilized the following instrumentation: ^1H and ^{13}C NMR, Varian XL300 (300 MHz); IR, Perkin-Elmer 599. NMR chemical shifts are reported in δ vs. Me_4Si assigning the CDCl_3 resonance in ^{13}C spectra to be 77.00 ppm.

The following compounds were prepared by literature methods: $\eta^5\text{-C}_5\text{H}_5\text{Fe}(\text{CO})_2\text{CH}_3$, $\eta^5\text{-C}_5\text{H}_5\text{Fe}(\text{CO})_2\text{CH}_2\text{CH}_3$, $\eta^5\text{-C}_5\text{H}_5\text{Fe}(\text{CO})_2\text{CH}_2\text{Ph}$ [17], $\eta^5\text{-C}_5\text{H}_5\text{W}(\text{CO})_3\text{CH}_3$, $\eta^5\text{-C}_5\text{H}_5\text{W}(\text{CO})_3\text{CH}_2\text{Ph}$ [18]. The $\text{W}(\text{CO})_6$, $[\eta^5\text{-C}_5\text{H}_5\text{Fe}(\text{CO})_2]_2$, and $\eta^5\text{-C}_5\text{H}_5\text{Mn}(\text{CO})_3$ were purchased from Strem Chemical Company and used as received. The alkyl trichlorosilanes were purchased from Petrarch Chemical Company and stored at -25°C over CaH_2 .

General method for the preparation of $\text{RSi}[\eta^5\text{-C}_5\text{H}_5\text{M}(\text{CO})_n\text{R}^1]_3$

A tetrahydrofuran (100 ml) solution containing 10 mmol of the cyclopentadienyl monomer is treated with 10 mmol of *s*-BuLi (1.3 *M* in cyclohexane) at -78°C and stirred for 20 min. The methyltrichlorosilane (3.3 mmol) is added dropwise via a syringe at -78°C . The mixture is allowed to warm to 20°C and the solvent is removed in vacuo. The crude residue is column chromatographed on alumina III using gradient elution. Initially hexanes are utilized and the solvent polarity increased with methylene chloride (1/1, v/v). Recovered monomer is eluted first, followed by a yellow band which corresponds to the trinuclear complex.

Preparation of $\text{MeSi}[\eta^5\text{-(C}_5\text{H}_4\text{)Fe}(\text{CO})_2\text{CH}_2\text{Ph}]_3$ (**1b**)

Complex **1b** is prepared in a similar manner as above except that the crude residue is washed on a glass-frit several times with water and finally with chloroform to yield pure **1b** as a yellow powder. Spectroscopic data for **1b** ^1H NMR (CDCl_3) 7.12–6.93 (m, 15H), 4.81 (dd, *J* 2 Hz, 6H), 4.70 (dd, *J* 2 Hz, 6H), 2.57 (s, 6H), 0.81 (s, 3H). Anal. Found: C, 61.43; H, 4.45. $\text{C}_{43}\text{H}_{36}\text{Fe}_3\text{O}_6\text{Si}$ calc: C, 61.16; H, 4.30%.

* As pointed out by a referee, varying the experimental procedures could possibly permit the use of excess lithiating reagent.

Spectroscopic and analytical data for complexes 1–5

$\text{CH}_3\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{CH}_3]_3$ (**1a**). ^1H NMR (CDCl_3) 4.88 (br s, 6H), 4.84 (br s, 6H), 0.80 (s, 3H, SiCH_3), 0.06 (s, 9H, FeCH_3); ^{13}C NMR (CDCl_3) 216.9 (CO), 93.4, 88.7, 83.1 (C_5H_4), -3.8 (SiCH_3), -23.0 (FeCH_3); IR (CH_2Cl_2) 2010 and 1953 cm^{-1} . Anal. Found: C, 48.54; H, 3.98. $\text{C}_{25}\text{H}_{24}\text{Fe}_3\text{O}_6\text{Si}$ calc: C, 48.74; H, 3.93%.

$\text{CH}_3\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{CH}_2\text{CH}_3]_3$ (**1c**). ^1H NMR (CDCl_3) 4.87 (dd, J 2 Hz, 6H), 4.82 (dd, J 2 Hz, 6H), 1.50 (t, J 7 Hz, 6H), 1.19 (t, J 7, 9 Hz) 0.81 (s, 3H); ^{13}C NMR (CDCl_3) 217.4 (CO), 92.8, 89.9, 83.5 (C_5H_4), 22.0 (FeCH_2CH_3), -2.3 (FeCH_2CH_3), -3.7 (SiCH_3). Anal. Found: C, 51.04; H, 4.62. $\text{C}_{28}\text{H}_{30}\text{Fe}_3\text{O}_6\text{Si}$ calc: C, 51.10; H, 4.59%.

$\text{CH}_3\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{Mn}(\text{CO})_3]_3$ (**2**). ^1H NMR (CDCl_3) 4.31 (dd, J 2 Hz, 6H), 4.11 (dd, J 2 Hz, 6H), 0.59 (s, 3H); ^{13}C NMR (CDCl_3) 224.3 (CO), 92.0, 85.0, 82.3 (C_5H_4), -4.2 (SiCH_3); IR (CH_2Cl_2) 2023 and 1943 cm^{-1} . Anal. Found: C, 45.88; H, 2.36. $\text{C}_{25}\text{H}_{15}\text{Mn}_3\text{O}_9\text{Si}$ calc: C, 46.03; H, 2.32%.

$\text{CH}_3\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{W}(\text{CO})_3\text{CH}_3]_3$ (**3a**). ^1H NMR (CDCl_3) 5.63 (dd, J 2 Hz, 6H), 5.22 (dd, J 2 Hz, 6H), 0.63 (s, 3H), 0.29 (s, 9H); ^{13}C NMR (CDCl_3) 215.1 (CO), 97.1, 96.3 (C_5H_4), 94.5 (SiC_5H_4), -4.3 (SiCH_3), -34.1 (WCH_3); IR (CH_2Cl_2) 2013 and 1923 cm^{-1} . Anal. Found: C, 30.97; H, 2.26. $\text{C}_{28}\text{H}_{24}\text{O}_9\text{SiW}_3$ calc: C, 31.02; H, 2.23%.

$\text{CH}_3\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{W}(\text{CO})_3\text{CH}_2\text{Ph}]_3$ (**3b**). ^1H NMR (CDCl_3) 7.1 (br s, 15H), 5.51 (dd, J 2 Hz, 6H), 4.91 (dd, J 2 Hz, 6H), 2.88 (s, 6H), 0.50 (s, 3H); ^{13}C NMR (CDCl_3) 217.1 (CO), 150.9, 128.0, 127.3, 124.1 (phenyl), 98.8, 97.7, 96.6 (C_5H_4), -4.2 (SiCH_3), -5.8 (WCH_2Ph). Anal. Found: C, 41.92; H, 2.90. $\text{C}_{46}\text{H}_{36}\text{O}_9\text{SiW}_3$ calc: C, 42.10; H, 2.77%.

$\text{ClCH}_2\text{CH}_2\text{CH}_2\text{Si}[\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{CH}_3]_3$ (**4**). ^1H NMR (CDCl_3) 4.89 (s, 12H), 3.64 (t, J 6 Hz, 2H), 2.34–2.01 (m, 2H), 1.49–1.26 (m, 2H), 0.14 (s, 9H); ^{13}C NMR

TABLE 2

CRYSTALLOGRAPHIC PARAMETERS FOR COMPOUND **1c**

Space group	Rhombohedral (R_{3c})
a, b, c (Å)	11.609(2)
α, β, γ (°)	101.73(2)
V (Å ³)	1450.2(5)
d_{calc} (g/cm ³)	1.51
Z	2
F_w	658.2
Cryst. shape	irregular
Cryst. size (mm)	minimum 0.60, maximum 0.92
μ (Mo- K_α) (mm ⁻¹)	1.60
Scan method	ω
Range, 2θ (°)	3.0–60.4
No. of unique data	1446
No. of data with $ F_o^2 > 3\sigma F_o^2 $	1090
R_1	0.027
R_2	0.026
No. of variables	122
Goodness of fit	1.38

TABLE 3

FRACTIONAL ATOMIC COORDINATES ($\times 10^4$) AND EQUIVALENT ISOTROPIC THERMAL PARAMETERS FOR NON-HYDROGEN ATOMS IN CRYSTALLINE $\text{CH}_3\text{Si}[(\eta^5\text{-C}_5\text{H}_4)\text{Fe}(\text{CO})_2(\text{C}_2\text{H}_5)]_3$ ^a

Atom ^b	x	y	z	B (Å ²) ^c
Fe	-2157(1)	1377(1)	1084(1)	3.3
Si ^d	0(-) ^d	0(-) ^d	0(-) ^d	3.4
O(1)	-3887(4)	-753(3)	-542(4)	7.9
O(2)	-2131(4)	574(4)	3288(3)	8.0
C(1)	-3192(4)	75(4)	107(4)	4.8
C(2)	-2152(4)	895(4)	2419(4)	4.7
C(3)	-3675(4)	2033(4)	1257(4)	5.0
C(4)	-4672(4)	1262(4)	1625(5)	6.4
Cp(1)	-540(3)	1386(2)	492(3)	3.5
Cp(2)	-250(4)	2135(3)	1680(4)	4.0
Cp(3)	-853(4)	3086(4)	1691(4)	4.8
Cp(4)	-1499(4)	2949(3)	506(4)	4.6
Cp(5)	-1328(3)	1917(3)	-237(3)	4.0
C _m	-1201(4)	-1201(4)	-1201(4)	4.5

^a The numbers in parentheses are the estimated standard deviations in the last significant digit. ^b Atoms are labeled in agreement with Fig. 1. ^c This is one-third of the trace of the orthogonalized B_{ij} tensor.

^d This atom was used to fix the origin of the unit cell; its coordinates are therefore listed without estimated standard deviations.

(CDCl_3) 216.0 (CO), 92.7, 89.6, 81.9 (C_5H_4), 47.4, 27.4, 11.7 (CH_2 's), -22.5 (FeCH_3); IR (CH_2Cl_2) 2010 and 1953 cm^{-1} . Anal. Found: C, 47.84; H, 4.04. $\text{C}_{27}\text{H}_{27}\text{ClFe}_3\text{O}_6\text{Si}$ calc: C, 47.79; H, 4.01%.

[*m*- and *p*-(CH_2Cl) C_6H_4]-(CH_2)₂-Si[$\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{CH}_3$]₃ (5). ¹H NMR (CDCl_3) 7.35–7.10 (m, 4H), 4.92 (brs, 12H), 4.58, 4.56 (s and shoulder, 2H), 3.16–2.92 (m, 2H), 1.73–1.52 (m, 2H), 0.16 (s, 9H); ¹³C NMR (CDCl_3); 216.7 (CO), 144.2, 137.6, 128.9, 127.9, 127.7, 126.3 (phenyl, major isomer), 92.7, 89.7, 82.1 (C_5H_5), 46.4, 29.9, 16.4 (CH_2 's), -22.1 (FeCH_3). Anal. Found: C, 52.47; H, 4.18. $\text{C}_{33}\text{H}_{31}\text{ClFe}_3\text{O}_6\text{Si}$ calc: C, 52.38; H, 4.13%.

Crystal and molecular structure determination of $\text{Me}[\eta^5\text{-C}_5\text{H}_4\text{Fe}(\text{CO})_2\text{CH}_2\text{CH}_3]_3$ (1c)

Single crystals of 1c were grown from a saturated CH_2Cl_2 solution at -25 °C. A summary of the X-ray study is presented in Table 2 and a listing of final atomic positions for non-hydrogen atoms can be found in Table 3. The anisotropic thermal parameters for non-hydrogen atoms, atomic coordinates for hydrogen atoms, and complete details of the structure determination can be found in Tables 4 and 5, and the crystal structure analysis report, respectively, located in the supplemental material.

Acknowledgement

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