

Synthesis and structure of the iron-substituted silylacetylene [[η^5 -C₅H₅)Fe(CO)₂SiMe₂C]₂ and its cobalt hexacarbonyl derivative [[η^5 -C₅H₅)Fe(CO)₂SiMe₂C]₂ · Co₂(CO)₆

Eulalia Ramirez-Oliva ^a, Jorge Cervantes ^a, Francisco Cervantes-Lee ^b, Ramesh N. Kapoor ^b,
Keith H. Pannell ^{b,*}

^a Facultad de Química, Universidad de Guanajuato, Guanajuato, GTO 36050, Mexico

^b Department of Chemistry, University of Texas at El Paso, El Paso, TX 79968-0513, USA

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Abstract

A transition metal-substituted silylacetylene [(η^5 -C₅H₅)Fe(CO)₂SiMe₂C]₂, [FpMe₂SiC]₂ (**I**) was synthesized and characterized spectroscopically and structurally. **I** crystallized in the monoclinic space group *P*2₁/*n*, *a* = 13.011(3) Å, *b* = 12.912(3) Å, *c* = 13.175(5) Å, β = 94.95(2). The acetylene linkage is reactive toward Co₂(CO)₈ to form **I** · Co₂(CO)₆ (**II**) which was also characterized spectroscopically and by single crystal X-ray diffraction. **II** crystallized in the orthorhombic space group *Pbca*, *a* = 17.64(2) Å, *b* = 14.225(10) Å, *c* = 24.49(2) Å.

Keywords: Iron; Silylacetylene; Cobalt; Cyclopentadienyls; Carbonyls; X-ray diffraction structure

1. Introduction

The chemistry of silylacetylenes has been widely studied and the compounds have found a significant synthetic utility [1]. Among their properties the ability to function as ligands to various transition metal complexes and a range of derivatives of Fe, Co, and Mn carbonyls with silyl-mono- and di-ynes has long been established [2–4]. More recently the work of Vollhardt and coworkers has amply demonstrated the utility of (η^5 -C₅H₅)C α (CO)₂ as a catalyst of great synthetic utility in co-oligomerization of α - ω -diynes with silylacetylenes [5]. The stoichiometric reaction between the catalyst and silylacetylenes led to a wide range of condensed products [6]. We have found no reports in the literature concerning the chemistry of transition metal-substituted silylacetylenes, with the triple bond intact, and we report on the synthesis, structure and reactivity towards dicobaltoctacarbonyl of one such example, [(η^5 -C₅H₅)Fe(CO)₂SiMe₂C]₂, [FpMe₂SiC]₂ (**I**).

2. Experimental section

All reactions were performed under an N₂ atmosphere using dry, oxygen-free solvents; bis-chlorodimethylsilylacetylene [7] was purchased from Silar Inc. and redistilled prior to use.

2.1. Synthesis of [(η^5 -C₅H₅)Fe(CO)₂SiMe₂C]₂ (**I**)

In a 200 ml side-armed flask containing [(η^5 -C₅H₅)Fe(CO)₂]⁻Na⁺ prepared from 3.0 g (8.47 mmol) of [(η^5 -C₅H₅)Fe(CO)₂]₂, an excess of [ClMe₂SiC]₂, 2.0 g (9.5 mmol) was added slowly at 0°C. The reaction mixture was stirred for 16 h at room temperature, and the solvent was removed in vacuo. The red–orange oily residue was dissolved in the minimum of hexane/methylene chloride solvent mixture (70:30) and placed upon a silica-gel column, 2.5 × 10 cm. Elution with hexane produced a yellow band that was collected and, subsequent to recrystallization from hexane, yielded 2.0 g (4.0 mmol, 47%) of **I** as yellow needles. Analysis and spectral data are recorded in Table 1.

* Corresponding author.

Table 1
M.p., elemental analysis and spectroscopic data for complexes **I** and **II**^a

I , 35°C, Anal. Calc. (Found): C, 48.58 (48.05); H 4.45 (4.72)	
¹ H	0.5 (SiMe); 4.8 (C ₅ H ₅)
¹³ C	7.1 (SiMe); 84.5 (C ₅ H ₅); 120.1 (C ₂); 215.5 (CO)
²⁹ Si	17.5
ν(CO)	2000, 1949
MS(<i>m/e</i>) ^b	438[[M–2CO] (10)]; 414[[M–3CO] (6)]; 382 [[M–4CO] (36)]; 261 [[M–4CO–Fe(C ₅ H ₅)] (5)]; 121 [[Fe(C ₅ H ₅)] (31)]
II 82°C (decomp.), Anal. Calc. (Found): C, 40.03 (40.08); H 2.84(2.75)	
¹ H	0.85 (SiMe); 4.3 (C ₅ H ₅)
¹³ C	11.4 (SiMe); 83.9 (C ₅ H ₅); 103.0 (C ₂); 202.7, 203.7 (Co–CO; 215.5 (Fe–CO)
²⁹ Si	38.4
ν(CO)	2079, 2040, 2021 (Co(CO) ₃), 2001, 1951 (Fe(CO) ₂)

^a NMR data, ppm, recorded in C₆D₆, IR cm⁻¹ in hexane. Analyses performed by Galbraith Laboratories Inc.

^b *m/e* [[ion composition] (% of ion current)].

2.2. Synthesis of [(η⁵-C₅H₅)Fe(CO)₂SiMe₂C]₂Co₂(CO)₆ (**II**)

A 100 ml side-armed flask was charged with a solution of 0.30 g (0.87 mmol) of Co₂(CO)₈ in 20 ml of hexane. To this magnetically stirred solution was added an excess of **I**, 0.5 g (1.01 mmol) and the stirring was

continued for 22 h. The solvent was removed in vacuo and the dark brown–black solid was redissolved in hexane and filtered. The filtrate was concentrated to 4 ml and this solution was placed upon a silica-gel column, 2.5 × 8 cm, and eluted with hexane to develop a brown band that was collected. Removal of the solvent and recrystallization from hexane produced **II**, 0.56 g

Table 2
Crystal and collection data

	I	II
Empirical formula	C ₁₀ H ₁₁ FeO ₂ Si	C ₂₆ H ₂₂ Co ₂ Fe ₂ O ₁₀ Si ₂
Color; habit	yellow fragment	dark red–brown plate
Crystal size (mm)	0.44 × 0.24 × 0.40	0.40 × 0.24 × 0.18
Crystal system	monoclinic	orthorhombic
Space group	<i>P</i> 2 ₁ / <i>n</i>	<i>Pbca</i>
Unit cell dimensions	<i>a</i> = 13.011(3) Å <i>b</i> = 12.912(4) Å <i>c</i> = 13.175(5) Å <i>β</i> = 94.95(2) ^o	<i>a</i> = 17.64(2) Å <i>b</i> = 14.225(10) Å <i>c</i> = 24.49(2) Å
Volume	2205.1(12) Å ³	6145(9) Å ³
<i>Z</i>	8	8
Formula weight	247.1	780.2
Density (calc.)	1.489 Mg cm ⁻³	1.687 Mg m ⁻³
Absorption coefficient	1.444 mm ⁻¹	2.114 mm ⁻¹
<i>F</i> (000)	1016	3136
Index ranges	0 ≤ <i>h</i> ≤ 18, 0 ≤ <i>k</i> ≤ 15, -26 ≤ <i>l</i> ≤ 0	0 ≤ <i>h</i> ≤ 14, 0 ≤ <i>k</i> ≤ 13, -14 ≤ <i>l</i> ≤ 14
Reflections collected	4528	3205
Independent reflections	4048 (<i>R</i> _{int} = 0.00%)	2893 (<i>R</i> _{int} = 5.17%)
Observed reflections	2399 (<i>F</i> > 3.0σ(<i>F</i>))	2578 (<i>F</i> > 3.0σ(<i>F</i>))
Absorption correction	semi-empirical	N/A
Min/max transmission	0.1628/0.2257	
Number of parameters refined	254	380
Final <i>R</i> indices (observed data)	<i>R</i> = 3.88%, <i>R</i> _w = 5.08%	<i>R</i> = 5.36%, <i>R</i> _w = 4.72%
<i>R</i> indices (all data)	<i>R</i> = 4.29%, <i>R</i> _w = 5.20%	<i>R</i> = 10.18%, <i>R</i> _w = 5.52%
Goodness-of-fit	1.25	1.10
Largest and mean Δ/σ	0.098, 0.017	2.953, 0.399
Data-to-parameter ratio	10.1 : 1	6.3 : 1
Largest difference peak	0.65 eÅ ⁻³	0.58 eÅ ⁻³
Largest difference hole	-0.60 eÅ ⁻³	-0.51 eÅ ⁻³

(0.71 mmol, 82%) as dark brown shiny needles. Analytical and spectroscopic properties of **II** are presented in Table 1.

2.3. X-ray structural determination

The structures of both **I** and **II** were determined using a Siemens R3m/V diffractometer with MoK α radiation ($\lambda = 0.71073 \text{ \AA}$) at room temperature (**II**) and 173 K (**I**). Refinement of the structures was accomplished using the SHELXTL PLUS (VMS) software package. Three standard reflections were measured every 97 reflections and neither crystal exhibited any decomposition. Crystal and collection data are recorded in Table 2, atomic coordinates are recorded in Table 3, and selected bond lengths and angles are presented in Table 4.

3. Results and discussion

The synthesis of $[(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2\text{SiMe}_2\text{C}]_2$ (**I**) was readily achieved via the standard salt-elimination

Table 3(a)
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$) of **I**

	x	y	z	U_{eq}
Fe(1)	2353(1)	780(1)	8845(1)	24(1)
Fe(2)	2665(1)	1585(1)	3708(1)	22(1)
Si(1)	3773(1)	1093(1)	7952(1)	23(1)
Si(2)	2683(1)	-18(1)	4490(1)	21(1)
O(1)	3129(2)	-1325(2)	8965(2)	48(1)
O(2)	3575(2)	1583(2)	10620(2)	43(1)
O(3)	1550(2)	638(3)	1944(2)	54(1)
O(4)	836(2)	1953(2)	4776(2)	44(1)
C(1)	3517(3)	479(3)	6679(3)	25(1)
C(2)	3224(3)	178(3)	5836(3)	24(1)
C(3)	1475(3)	1516(3)	7641(3)	36(1)
C(4)	1110(3)	502(3)	7775(3)	40(1)
C(5)	772(3)	430(4)	8762(3)	46(2)
C(6)	948(3)	1388(3)	9258(3)	43(1)
C(7)	1382(3)	2057(3)	8565(3)	39(1)
C(8)	2828(3)	-479(3)	8929(3)	31(1)
C(9)	3100(3)	1256(3)	9913(3)	28(1)
C(10)	5016(3)	576(3)	8582(3)	33(1)
C(11)	3994(3)	2511(3)	7716(3)	37(1)
C(12)	3725(3)	2426(3)	4659(3)	32(1)
C(13)	3305(3)	3082(3)	3862(3)	35(1)
C(14)	3537(3)	2664(3)	2932(3)	34(1)
C(15)	4113(3)	1746(3)	3129(3)	31(1)
C(16)	4234(3)	1610(3)	4209(3)	30(1)
C(17)	1991(3)	1015(3)	2647(3)	32(1)
C(18)	1556(3)	1816(3)	4354(3)	30(1)
C(19)	3513(3)	-995(3)	3886(3)	29(1)
C(20)	1380(3)	-620(3)	4571(3)	3(1)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 3(b)

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$) of **II**

	x	y	z	U_{eq}
Fe(1)	-1085(1)	3034(1)	874(1)	50(1)
Fe(2)	1112(1)	2536(1)	3090(1)	48(1)
Co(1)	1660(1)	2867(1)	876(1)	42(1)
Co(2)	1667(1)	1355(1)	1372(1)	41(1)
Si(1)	-185(1)	1849(2)	949(1)	40(1)
Si(2)	1334(1)	3293(2)	2258(1)	36(1)
O(1)	-2146(5)	1509(6)	695(4)	96(4)
O(2)	-1192(5)	3005(8)	2050(4)	112(5)
O(3)	1937(5)	4068(6)	3586(4)	87(4)
O(4)	2438(5)	1385(6)	2926(4)	95(4)
O(5)	3300(4)	3148(6)	960(4)	90(4)
O(6)	1525(5)	2273(7)	-262(3)	88(4)
O(7)	1326(5)	4867(5)	803(4)	79(4)
O(8)	1058(4)	16(5)	2151(3)	73(3)
O(9)	3230(4)	1391(5)	1798(4)	82(4)
O(10)	1785(5)	149(6)	403(4)	89(4)
C(1)	1204(5)	2560(7)	1622(4)	33(3)
C(2)	761(5)	2209(6)	1233(4)	34(3)
C(3)	-56(6)	1292(8)	266(4)	75(5)
C(4)	-515(6)	848(7)	1401(5)	73(5)
C(5)	-544(6)	4310(8)	771(6)	74(6)
C(6)	-366(7)	3748(8)	328(5)	64(5)
C(7)	-1060(10)	3526(10)	82(6)	93(7)
C(8)	-1653(8)	3973(11)	361(8)	111(8)
C(9)	-1324(8)	4461(8)	779(7)	100(7)
C(10)	-1728(7)	2095(8)	774(5)	62(5)
C(11)	-1145(7)	3015(9)	1580(6)	73(5)
C(12)	2311(5)	3788(7)	2216(4)	57(4)
C(13)	673(5)	4315(6)	2168(4)	47(4)
C(14)	71(7)	2028(13)	2823(6)	85(6)
C(15)	-48(6)	2845(11)	3128(7)	83(6)
C(16)	226(7)	2674(9)	3657(5)	73(5)
C(17)	501(7)	1775(9)	3668(5)	77(5)
C(18)	408(8)	1356(10)	3160(7)	91(6)
C(19)	1619(7)	3478(8)	3385(5)	63(5)
C(20)	1914(7)	1865(7)	2983(5)	62(5)
C(21)	2669(7)	3037(7)	947(4)	56(4)
C(22)	1568(6)	2491(9)	178(5)	64(5)
C(23)	1435(6)	4082(8)	829(5)	52(4)
C(24)	1301(6)	541(7)	1847(4)	45(4)
C(25)	2620(6)	1412(7)	1640(5)	54(4)
C(26)	1734(7)	602(8)	780(5)	63(4)5

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

reaction used to produce Fp-silyl complexes:

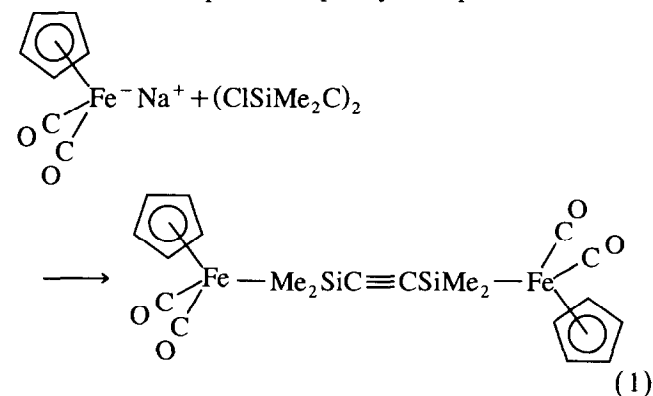


Table 4
Selected bond lengths (Å) and bond angles (°)

<i>Bond lengths (I)</i>			
Fe(1)–Si(1)	2.310(1)	Fe(2)–Si(2)	2.311(1)
Si(1)–C(1)	1.859(4)	Si(2)–C(2)	1.868(4)
C(1)–C(2)	1.207(5)	Si(1)–C(10)	1.877(4)
Fe(1)–C(9)	1.750(4)	C(9)–O(2)	1.152(5)
<i>Bond angles (I)</i>			
Si1–C1–C2	170.1(3)	Si2–C2–C1	168.7(3)
C10–Si1–C11	106.3(2)	C20–Si2–C19	107.5(2)
C11–Si1–C1	106.5(2)	C19–Si2–C2	108.0(2)
Fe1–Si1–C1	107.3(1)	Fe1–Si1–C10	114.2(1)
C8–Fe1–C9	96.2(2)		
<i>Bond lengths (II)</i>			
Fe(1)–Si(1)	2.322(4)	Fe(2)–Si(2)	2.338(4)
Si(1)–C(2)	1.879(9)	Si(2)–C(1)	1.887(10)
C(1)–C(2)	1.330(13)	Co(1)–Cα(2)	2.470(3)
Co(1)–C(2)	2.038(9)	Co(2)–C(1)	1.995(9)
Fe(1)–C(11)	1.733(15)	Co(1)–C(22)	1.797(13)
<i>Bond angles (II)</i>			
Si1–C2–C1	152.7(7)	Si2–C1–C2	150.5(7)
C3–Si1–C2	109.8(4)	C4–Si1–C3	104.0(5)
Co2–C1–C2	72.4(6)	Co2–Co1–C2	52.6(3)
Fe1–Si1–C2	116.0(3)	Fe1–Si1–C3	108.8(4)
C19–Fe2–C20	93.8(6)		

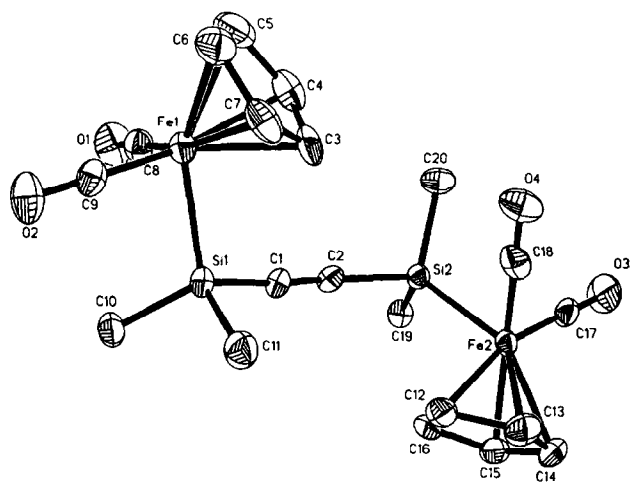
The spectral properties of **I** are in accord with expectation. The ^{29}Si NMR resonance at 17.5 ppm exhibits a 36.6 ppm downfield shift from the permethylated $\text{Me}_3\text{SiC}_2\text{SiMe}_3$ at -19.1 ppm. This represents a typical $\Delta\delta$ ($\delta \text{Fp-SiR}_3 - \delta \text{Me-SiR}_3$) shift for the silicon atom attached directly to Fe in Fp–Si complexes [8]. The mass spectrum of **I** exhibited an $[\text{M}-2\text{CO}]^+$ ion at m/e 438, with the base ion of m/e 382 representing a CO-free species $[\text{M}-4(\text{CO})]^+$. The complete data are presented in Table 1.

The structure of **I** is illustrated in Fig. 1. The Si–C–C–Si linkage is almost linear (Si–C–C = $170.1(3)^\circ$ and $168.7(3)^\circ$) with Si–Csp bond lengths of 1.868(4) Å and 1.859(4) Å and a C–C triple bond length of 1.207(5) Å. These values are in accord with other silylacetylene structures as noted in Table 5. When viewed along the Si–C–C–Si axis the groups attached to the silicon atoms are seen to be completely eclipsed (Fig. 2) with the two Fp units each being eclipsed by a methyl group. The Fe–Si bond lengths of 2.310(1) Å and 2.311(1) Å

Table 5
Structural parameters of bis-silylacetylenes and metal complexes ^a

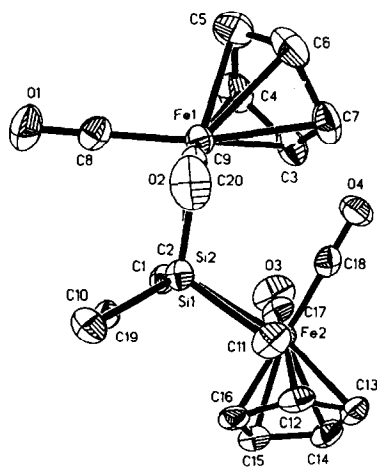
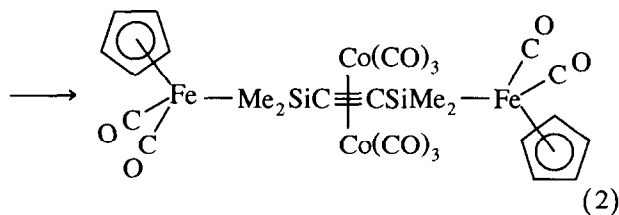
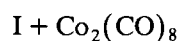
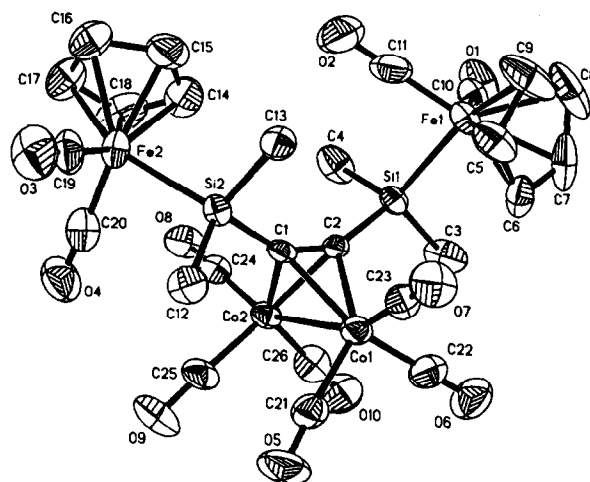
	Compound	C ₂ bond (Å)	Si–Csp bond (Å)	Reference
1	(PhH ₂ Si) ₂ C ₂	1.196	1.831	[9]
2	(Me ₁₁ Si ₆) ₂ C ₂	1.211	1.833	[10]
3	I	1.207	1.859, 1.868	This work
4	BTSANi(PPh ₃) ₂	1.255	1.864	[11]
5	BTSAOs(CO) ₄	1.273	1.845, 1.829	[12]
6	BTSACp ₂ (R)Nb	1.281	1.848, 1.844	[13]
7	[BTSAMo(H)Cp ₂] ⁺	1.249	1.857, 1.863	[14]
8	BTSACe(C ₅ Me ₄ R)	1.249	1.849, 1.848	[15]
9	B TSA ₄ Fe ₂	1.288(3)		[16]
10	B TSA ₄ Fe ₂	1.314(3)	1.857, 1.854	[16]
11	(B TSAWCl ₄) ₂	1.333	1.883, 1.868	[17]
12	B TSA Mo ₂ Cp ₂ (CO) ₄	1.358	1.854	[18]
13	B TSA WFe ₂ Cp(CO) ₇ R	1.43	1.865, 1.849	[19]
14	Me ₃ SiC ₂ PhCo ₃ (CO) ₁₀	1.337	1.834	[20]
15	II	1.330	1.887, 1.879	This work

^a BTSA = bis-trimethylsilylacetylene, Me₃SiC₂SiMe₃.

Fig. 1. Structure of **I**.

are shorter than usual for Fp complexes of the type FpSiMe_2R , $\text{R} = \text{alkyl, aryl, silyl}$, which are in the range 2.325(2)–2.365(3) Å [21]. This change represents the presence of the sp hybridized C atom on silicon, which is significantly more electronegative than the sp^3 C atoms present in the other complexes. Electronegative substituents on silicon lower the Fe–Si bond distance, possibly by increasing some Fe(d)–Si(d) interactions, e.g. FpSiCl_3 , $d(\text{Fe}–\text{Si}) = 2.215$ Å [22].

A typical reaction of acetylenes, including silylacetylenes, is their reactivity with cobalt octacarbonyl $\text{Co}_2(\text{CO})_8$, and **I** readily reacted to form the expected bridging complex **II**:

Fig. 2. Conformation of **I** viewed along SiCCSi axis.Fig. 3. Structure of **II**.

The diagnostic ^{29}Si resonance appeared at 38.4 ppm, a downfield shift of 20.9 ppm from **I**. This shift is the same as previously noted for silylacetylene- $\text{Co}_2(\text{CO})_6$ complexes, e.g. $\text{Me}_3\text{SiC}_2\text{SiMe}_3 \cdot \text{Co}_2(\text{CO})_6$ exhibited a 20.3 ppm downfield shift from that of $\text{Me}_3\text{SiC}_2\text{SiMe}_3$ [23].

The structure of **II** is presented in Fig. 3. The bond angles about the central π bond Si–C–C are now restricted to $150.5(7)^\circ$ and $152.7(7)^\circ$, and the C–C bond distance is 1.330(13) Å. These data indicate a significant change from the sp hybridization of the acetylene C atoms, however, they are still considerably deformed from a tetrahedral sp^3 hybridization that could be expected upon coordination to a bimetallic substrate. In Table 5 we present a range of structural data for silylacetylenes and silylacetylene complexes to note the systematic changes occurring upon coordination, and it is apparent that not all metals have the same effect upon the structural parameters of the silylacetylene portion of the resulting complexes. Monometallic complexes (4–9), for example $\text{Os}(\text{CO})_4(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$, have acetylenic C–C bond lengths in the range 1.249–1.281 Å, whereas bimetallic complexes (9–15), such as **II** and $(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_2(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ result in a significantly longer acetylenic linkage, in the range 1.330–1.43 Å. This is precisely documented by the structure of $(\text{Me}_3\text{SiC}_2\text{SiMe}_3)_4\text{Fe}_2$ which contains both a singly coordinated $\text{Me}_3\text{SiC}_2\text{SiMe}_3$ (C₂ bond = 1.288(3) Å) as well as a bridging unit (C₂ bond = 1.314 Å) [16]. Similar results have been noted for cyclic acetylene transition metal complexes [24].

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