a period of 25 h. No reaction was observed.

In a second experiment, 0.31 g (0.64 mmol) of II was dissolved in a mixture of 2 mL of C_6D_6 and 2 mL of MeOH in a quartz tube and the solution photolyzed under an argon atmosphere. The reaction was monitored by ^{29}Si NMR and IR spectroscopy. After 54 h no new products were observed. When the reaction was repeated under an O_2 atmosphere with O_2-saturated solvents, all the starting material had disappeared after 15 h and the ^{29}Si NMR spectrum exhibited a single new resonance at 12.4 ppm assignable to $FcSiMe_2OMe$ (V).

Photolysis of IX. A 0.12-g (0.49-mmol) sample of IX in a mixture of 1 mL of C_6D_6 and 1 mL of MeOH in a quartz tube was photolyzed over a period of 32 h under scrupulously O_2-free conditions. IR and ^{29}Si NMR monitoring of the reaction showed that no reaction occurred. When the reaction was repeated in an O_2 atmosphere complete conversion to V was observed.

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Photochemical Conversion of a Disilanylene-Bridged Iron Dimer to Silylene-Bridged Iron Dimers

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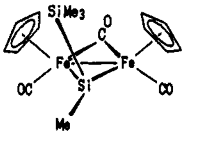
Photolysis of $FpSiMe_2SiMe_2Fp$ (Fp = $CpFe(CO)_2$; Cp = η-C_5H_5) in C_6D_6 afforded, in the early stages of photolysis, a mixture of the geometric isomers $[CpFe(CO)]_2(\mu\text{-}CO)\{\mu\text{-}SiMe(SiMe_3)\}$ and $[CpFe(CO)]_2(\mu\text{-}SiMe_2)_2$ (a small amount). Prolonged photolysis gave *cis*- and *trans*-$[CpFe(CO)]_2(\mu\text{-}SiMe_2)_2$ almost quantitatively. The structure of a geometric isomer of $[CpFe(CO)]_2(\mu\text{-}CO)\{\mu\text{-}SiMe(SiMe_3)\}$ was determined by X-ray crystallography. Crystal data: $C_{17}H_{22}Fe_2O_3Si_2$, triclinic in the space group $P\bar{1}$, a = 9.555 (3) Å, b = 14.031 (3) Å, c = 7.986 (2) Å, α = 91.18 (3)°, β = 113.56 (3)°, γ = 101.72 (3)°, V = 955.5 (4) Å^3, Z = 2, and $R(F_o)$ = 0.040 for 3792 reflections with $|F_o| > 3\sigma(F_o)$.

Introduction

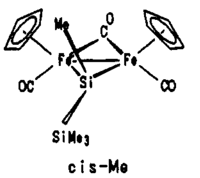
Photolytic rearrangement of disilanyliron complexes $FpSi_2R_5$ (Fp = $CpFe(CO)_2$; Cp = η-C_5H_5; R = alkyl, aryl) has been proposed to proceed via a mechanism containing silyl(silylene)iron complexes $CpFe(CO)(SiR_3)({=}SiR_2)$ as key intermediates.[1,2] Rapid alkyl or aryl scrambling (1,3-alkyl or -aryl migration) occurs in the silyl(silylene)iron intermediates. The proposed mechanism has been strongly supported by the observation that isomeric $FpSi_2R_5$ compounds produce the same relative proportions of $FpSiR_3$[1d,e] and also our recent study on the photolysis of alkoxy-substituted disilanyliron complexes $FpSi_2R_{5-m}(OR)_m$, which leads to the formation of the alkoxy-bridged bis(silylene)iron complexes.[3] This paper describes the photolysis of $FpSiMe_2SiMe_2Fp$ (1).[4] Judging from the findings accumulated for the photolysis of disilanyliron complexes, several products had been expected to be formed as follows: $FpSiMe_3$, $FpSiMe_2Fp$, and/or $[CpFe(CO)]_2(\mu\text{-}CO)(\mu\text{-}SiMe_2)$. However, actual photolysis did *not* give these products *at all* but $[CpFe(CO)]_2(\mu\text{-}CO)\{\mu\text{-}SiMe(SiMe_3)\}$ (**2**) and $[CpFe(CO)]_2(\mu\text{-}SiMe_2)_2$ (**3**).

Results and Discussion

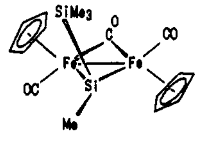
A C_6D_6 solution of 1 in a flame-sealed Pyrex NMR tube was irradiated by using a 450-W medium-pressure Hg lamp. The ^{1}H NMR spectral change upon the irradiation is shown in Figure 1. The spectrum obtained after 2 h irradiation (Figure 1b) shows that 1 disappears almost completely. In turn, all possible geometrical isomers for 2 (**2a–c**) are formed. Figure 1b also shows the formation of small amounts of **3a** and **3b**. Further irradiation results



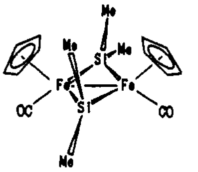
cis-SiMe$_3$
2a



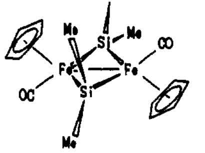
cis-Me
2b



trans
2c



cis
3a



trans
3b

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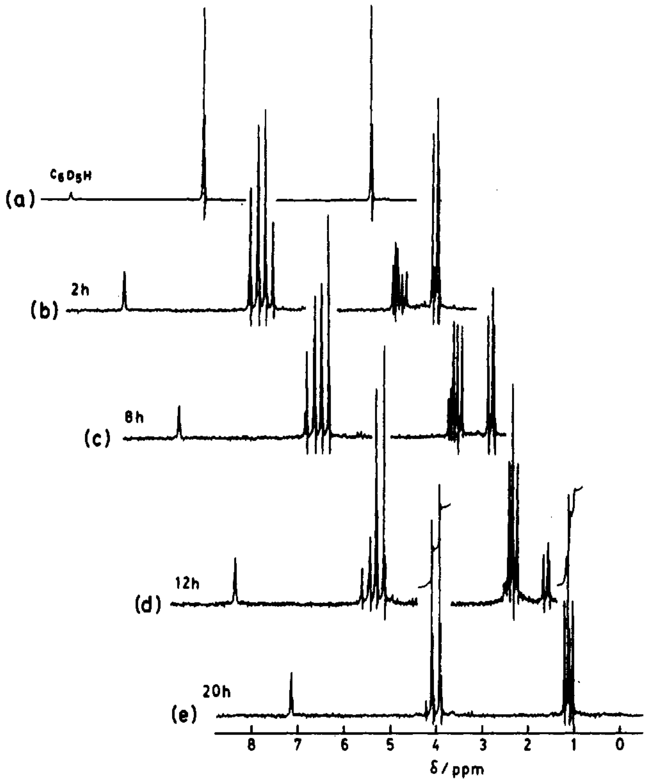

Figure 1. ^{1}H NMR spectral change for the photolysis of FpSiMe$_2$SiMe$_2$Fp (1, 25 mg) in 0.4 mL of C$_6$D$_6$: (a) before irradiation; (b) after 2 h of irradiation; (c) after 8 h of irradiation; (d) after 12 h of irradiation; (e) after 20 h of irradiation.

in the gradual decrease of 2 and increase of 3 (Figure 1b–e). The spectrum obtained after 20 h of irradiation (Figure 1e) shows the almost complete disappearance of 2 and the formation of a mixture of *cis-* and *trans-*[CpFe(CO)]$_2$(μ-SiMe$_2$)$_2$.

Figure 2 shows the ^{1}H NMR spectrum of the Cp signal region obtained at a very early stage of the photolysis. It should be noted that **3a,b** as well as **2a–c** are formed even at this stage.

When the solution containing predominantly 2 was stored at 1 °C, red-violet crystals of **2a** were obtained exclusively. The X-ray crystal structure analysis was carried out for the crystal. The solved structure is shown in Figure 3. Selected bond distances and angles are listed in Table I. The two Cp rings are mutually cis, and the Cp rings and the bulky Me$_3$Si group are located on the same side with respect to the four-membered ring defined by Fe(1), Si(1), Fe(2), and C(13). The Fe–Si bonds (2.294 (1) and 2.301 (1) Å) of **2a** are longer than those of cis-H isomer [CpFe(CO)]$_2$(μ-CO)(μ-SiHBu^t) (4) (2.270 (1) and 2.272 (1) Å).[5] The Fe–Fe bond (2.622 (1) Å) is also longer than those of 4 (2.614 (1) Å),[5] [CpFe(CO)]$_2$(μ-CO)$_2$ (2.531 (2) Å),[6] and [CpFe(CO)]$_2$(μ-CO)(μ-CHMe) (2.520 (2) Å)[7] but slightly shorter than that of [CpFe(CO)]$_2$(μ-CO)(μ-GeMe$_2$) (2.628 (1) Å).[8] The angle between the Fe$_2$Si(1) plane and the Si(1)–Si(2) bond (133.4°) is larger than that required for the tetrahedral configuration around Si(1) atom (125.3°). In contrast to this, the angle between the Fe$_2$Si(1) plane and Si(1)–C(14) (127.3°) is close to the ideal value. The distortion may be attributed to the steric repulsion between SiMe$_3$ and the Cp rings.

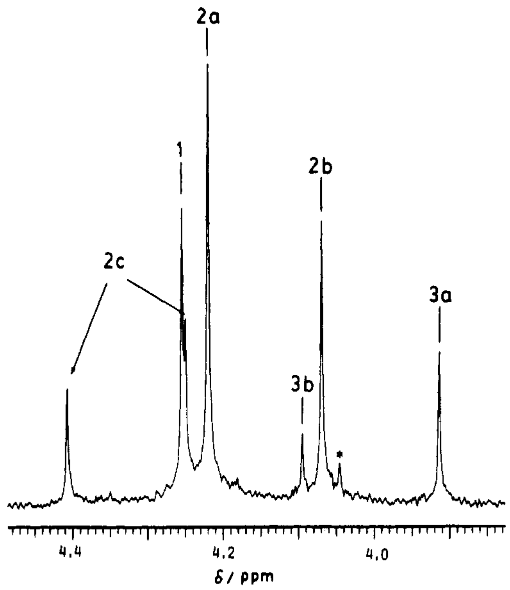

Figure 2. ^{1}H NMR spectrum of the Cp signal region obtained after 6 min of irradiation of C$_6$D$_6$ solution (0.4 mL) containing 1 (ca. 10 mg). The peak marked by an asterisk is due to an impurity.

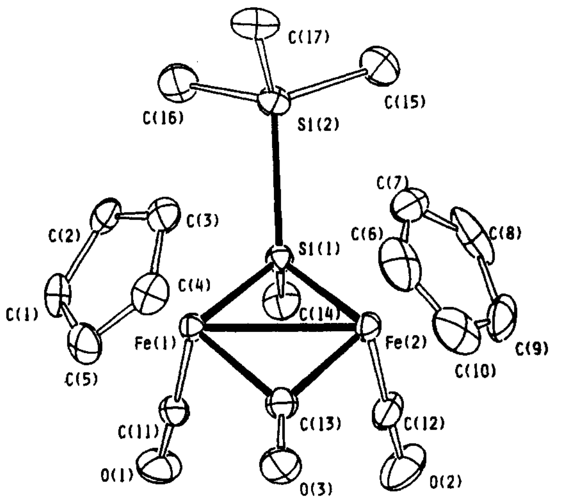

Figure 3. ORTEP view of **2a** with thermal ellipsoids at the 30% probability level.

Table I. Selected Bond Distances (Å) and Angles (deg) for [CpFe(CO)]$_2$(μ-CO){μ-SiMe(SiMe$_3$)} (2a)

Distances			
Fe(1)–Fe(2)	2.622 (1)	Fe(1)–Si(1)	2.294 (1)
Fe(2)–Si(1)	2.301 (1)	Fe(1)–C(11)	1.729 (4)
Fe(2)–C(12)	1.737 (5)	Fe(1)–C(13)	1.936 (3)
Fe(2)–C(13)	1.915 (4)	Si(1)–Si(2)	2.371 (1)
Si(1)–C(14)	1.904 (4)	Si(2)–C(15)	1.876 (5)
Si(2)–C(16)	1.876 (4)	Si(2)–C(17)	1.872 (6)
O(1)–C(11)	1.154 (6)	O(2)–C(12)	1.153 (6)
O(3)–C(13)	1.180 (4)		

Angles			
Si(1)–Fe(1)–C(11)	86.9 (2)	Si(1)–Fe(1)–C(13)	100.04 (9)
C(11)–Fe(1)–C(13)	89.6 (2)	Si(1)–Fe(2)–C(12)	87.2 (2)
Si(1)–Fe(2)–C(13)	100.5 (1)	C(12)–Fe(2)–C(13)	89.4 (2)
Fe(1)–Si(1)–Fe(2)	69.59 (3)	Fe(1)–Si(1)–Si(2)	123.18 (5)
Fe(1)–Si(1)–C(14)	120.4 (1)	Fe(2)–Si(1)–Si(2)	125.45 (5)
Fe(2)–Si(1)–C(14)	119.3 (1)	Si(2)–Si(1)–C(14)	99.4 (1)
Fe(1)–C(11)–O(1)	177.8 (4)	Fe(2)–C(12)–O(2)	177.5 (5)
Fe(1)–C(13)–Fe(2)	85.8 (1)	Fe(1)–C(13)–O(3)	135.8 (3)
Fe(2)–C(13)–O(3)	138.3 (3)		

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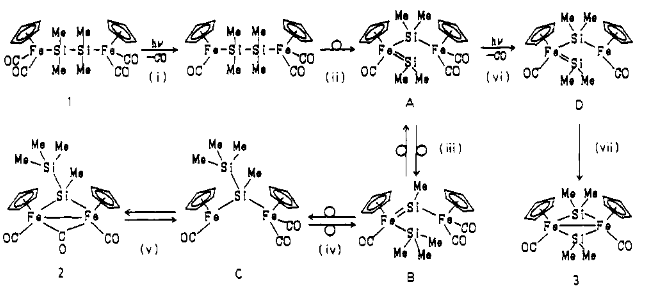
Scheme I. One Possible Mechanism for the Photolysis of $FpSiMe_2SiMe_2Fp$ (1)



In $[CpFe(CO)]_2(\mu\text{-}CO)(\mu\text{-}SiHR)$ (R = Me,[9] Bu^t[5,10]) or $[CpFe(CO)]_2(\mu\text{-}CO)(\mu\text{-}SiMeR)$ (R = Bu^t[10]), a sterically crowded substituent R on a bridging silicon atom tends to be located away from the Cp ring(s). Therefore, one may expect that the isomer **2b** is the most stable among **2a–c**. Contrary to this expectation, however, the isomer **2a** was found to be thermodynamically the most stable. When crystals of **2a** are dissolved in benzene, a mixture of **2a–c** results. The isomerization reaction equilibrates after 10 h at room temperature. The molar ratio of the isomers was cis-$SiMe_3$:cis-Me:trans = 41:32:27. The isomer distribution may indicate that the steric hindrance between the $SiMe_3$ group and the Cp rings in **2a** is smaller than that between the methyl group on the Si(1) atom and Cp rings in **2b**. This curious phenomenon arises from the fact that the Si–Si bond in **2a** (2.371 (1) Å) is longer than the Si–C bond in **2a** (1.904 (4) Å).

We propose one possible mechanism reasonably explaining the photochemical conversion of 1 to 2 in Scheme I:[11] (i) photoinduced CO loss to generate a coordinatively unsaturated 16-electron intermediate; (ii) 1,2-shift of the $SiMe_2Fp$ group to form the (Fp-substituted silyl)silyleneiron intermediate A; (iii) 1,3-shift of Me group from the Fp–silyl group to the silylene ligand to give the silyl(Fp-substituted silylene)iron intermediate B; (iv) 1,2-shift of the silyl group to the unsaturated silicon atom to form the intermediate C containing bridging $Si(Me)SiMe_3$; (v) bridging of a terminal CO ligand in the Fp fragment to produce 2.

Recently, Pannell and his colleagues reported interesting observations that the nature of the photolytic products derived from oligosilanyliron complexes $FpSi_nR_m$ changes dramatically depending on the number of the silicon atoms n.[2] For $n = 2$, the silylene ligand is lost from the silyl(silylene)iron intermediate to form the monosilyliron complex $FpSiR_3$, while in the case of $n > 3$, skeletal redistribution occurs without "SiR_2" expulsion to give the branched oligosilanyliron complex(es). The photochemical behavior of the disilanylene-bridged iron dimer 1 can be categorized formally with that of oligosilanyliron complexes containing more than three silicon atoms ($n > 3$).

Our early detection of 3 from 1 upon irradiation may be explained by the sequence of the processes shown in Scheme I: (vi) photoinduced CO loss from the intermediate A to give a coordinatively unsaturated intermediate D; (vii) cyclization to form the complex 3. However, photolysis of 2 was found also to give 3 quantitatively. Therefore, 2 is the "dead-end"[12] intermediate in the photolysis of 1 to the ultimate product 3. This means that the processes iii–v must be reversible under the experimental conditions.

Table II. Crystal Data for $[CpFe(CO)]_2(\mu\text{-}CO)\{\mu\text{-}SiMe(SiMe_3)\}$ (2a)

formula	$C_{17}H_{22}Fe_2O_3Si_2$
fw	442.23
cryst system	triclinic
space group	$P\bar{1}$
a/Å	9.555 (3)
b/Å	14.041 (3)
c/Å	7.986 (2)
α/deg	91.18 (3)
β/deg	113.56 (3)
γ/deg	101.72 (3)
V/Å^3	955.5 (4)
Z	2
d_{calcd}/g cm^{-3}	1.54
μ(Mo Kα)/cm^{-1}	16.9
cryst size/mm	0.33 × 0.10 × 0.09
radiation	Mo Kα (λ = 0.71073 Å)
monochrometer	graphite
temp/°C	21.5
type of diffractometer	Rigaku AFC-6A
reflcns measd	$\pm h,\pm k,+l$
2θ range/deg	3–60
scan mode	ω–2θ
ω-scan width/deg	1.0 + 0.35 tan θ
no. of unique data	5574
no. of data used with $\lvert F_o\rvert > 3\sigma(\lvert F_o\rvert)$	3792
R^a	0.040
$R_w{}^b$	0.038

$^a R = \sum\lvert\lvert F_o\rvert - \lvert F_c\rvert\rvert/\sum\lvert F_o\rvert$. $^b R_w = [\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2/\sum w\lvert F_o\rvert^2]^{1/2}$; $w = [\sigma^2(\lvert F_o\rvert) + aF_o{}^2]^{-1}$, where a = 0.00015.

Experimental Section

General Procedures. All manipulations were carried out under a nitrogen or argon atmosphere or high vacuum. Reagent grade pentane and toluene were distilled under a nitrogen atmosphere from sodium benzophenone ketyl immediately before use. Benzene-d_6 was dried over a potassium mirror, and dichloromethane-d_2 was dried over 4-Å molecular sieves. These two solvents were transferred to reaction vessels under vacuum before use. $FpSiMe_2SiMe_2Fp$ (1) was prepared according to the literature.[13] IR spectra were recorded on a JASCO IR-810 spectrophotometer using solution cells of 0.1-mm path length with sodium chloride windows. ^1H NMR spectra were recorded on a Varian XL-200 or a Varian EM-390 spectrometer. ^{13}C NMR spectra were recorded on the Varian XL-200 spectrometer. ^{29}Si NMR spectra were obtained on a Bruker AC-300 spectrometer using the DEPT pulse sequence. Mass spectra were recorded with a JEOL JMS-HX110 or a Hitachi M-52 mass spectrometer. High-resolution mass spectra (HRMS) were recorded with the JEOL JMS-HX110 mass spectrometer.

Photolysis of $FpSiMe_2SiMe_2Fp$ (1). The complex 1 was placed in a Pyrex NMR sample tube (5-mm o.d. × 3-mm i.d.) connected to a vacuum line and then dissolved in C_6D_6 transferred by the conventional trap-to-trap method. The sample tube was then flame-sealed under vacuum. Irradiation was carried out externally at room temperature with a 450-W medium-pressure Hg lamp (Ushio UV-450) placed in a water-cooled quartz jacket. The sample tube was immersed in water during the irradiation. The distance from the lamp to the sample tube was about 4 cm. The reaction was followed by taking ^1H NMR spectra periodically.

Preparation of $[CpFe(CO)]_2(\mu\text{-}CO)\{\mu\text{-}SiMe(SiMe_3)\}$ (2). A Pyrex NMR tube was charged with 52 mg of 1 and about 0.4 mL of C_6D_6. The solution was irradiated for 40 min. When the reaction mixture was stored at 1 °C, red-violet crystals of **2a** were obtained exclusively. The crystals were collected and washed with toluene (1 mL) and pentane (2 × 2 mL). Yield: 26 mg (54%). ^1H NMR (C_6D_6):[14] δ 4.41 (s, 5 H, Cp($SiMe_3$), **2c**), 4.25 (s, 5 H, Cp(Me), **2c**), 4.22 (s, 10 H, Cp, **2a**), 4.07 (s, 10 H, Cp, **2b**), 1.35 (s, 3 H, Si*Me*, **2a**), 1.29 (s, 3 H, Si*Me*, **2c**), 1.23 (s, 3 H, Si*Me*, **2b**),

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[11] The geometric configurations are not specified.

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[14] Some signals were assigned by means of the NOE technique. Details will be reported elsewhere.

Table III. Final Atomic Parameters and Temperature Factors of the Non-Hydrogen Atoms of $[CpFe(CO)]_2(\mu\text{-}CO)\{\mu\text{-}SiMe(SiMe_3)\}$ (2a)[a]

atom	*x*	*y*	*z*	B_{eqv}
Fe(1)	6695.9 (5)	1727.1 (3)	2687.5 (6)	2.3
Fe(2)	6863.7 (5)	3608.9 (3)	3120.5 (6)	2.6
Si(1)	4782.3 (10)	2492.3 (6)	944.0 (11)	2.4
Si(2)	2190.9 (10)	2101.2 (7)	768.9 (13)	3.2
O(1)	7527 (3)	1628 (2)	−396 (4)	5.1
O(2)	7800 (4)	4248 (2)	229 (4)	6.8
O(3)	9793 (3)	2981 (2)	4712 (4)	5.1
C(1)	6180 (5)	208 (2)	2658 (5)	3.9
C(2)	5006 (4)	561 (2)	2926 (5)	3.6
C(3)	5720 (4)	1199 (2)	4561 (5)	3.6
C(4)	7337 (4)	1230 (3)	5318 (5)	4.1
C(5)	7628 (4)	620 (3)	4153 (5)	4.2
C(6)	7068 (7)	3843 (3)	5847 (6)	6.5
C(7)	5577 (6)	3806 (3)	4649 (7)	6.4
C(8)	5563 (6)	4539 (4)	3523 (6)	7.1
C(9)	7172 (7)	5039 (3)	4149 (7)	6.6
C(10)	8013 (5)	4595 (4)	5551 (7)	7.1
C(11)	7197 (4)	1687 (2)	837 (4)	3.1
C(12)	7409 (5)	3974 (2)	1360 (5)	4.0
C(13)	8425 (4)	2853 (2)	3864 (4)	3.3
C(14)	4325 (5)	2575 (3)	−1591 (5)	4.0
C(15)	1243 (5)	3146 (3)	−77 (7)	6.1
C(16)	1104 (4)	1018 (3)	−1025 (5)	4.6
C(17)	1904 (5)	1762 (3)	2884 (6)	5.0

[a] Positional parameters are multiplied by 10^4. Thermal parameters are given by the equivalent temperature factors ($Å^2$).

0.48 (s, 9 H, Si*Me*$_3$, **2b**), 0.36 (s, 9 H, Si*Me*$_3$, **2c**), 0.33 (s, 9 H, Si*Me*$_3$, **2a**). ^{13}C NMR (CD_2Cl_2): δ 278.9 (μ-CO), 277.6 (μ-CO), 214.0 (CO), 213.4 (CO), 85.5 (Cp), 85.0 (Cp), 84.6 (Cp), 84.1 (Cp), 12.4 (Me), 10.8 (Me), 8.8 (Me), 1.8 (Me), 1.1 (Me), 0.6 (Me). ^{29}Si NMR (DEPT, CD_2Cl_2): δ 245.5 (μ-Si), 242.7 (μ-Si), 232.1 (μ-Si, **2a**), −3.7 ($SiMe_3$, **2a**), −4.9 ($SiMe_3$), −6.3 ($SiMe_3$). IR (C_6D_6): ν(CO) 1965, 1927, 1766 cm^{-1}. MS (DEI, 70 eV) [*m/z* (assignment, relative intensity)]: 442 (M^+, 30.1), 414 (M^+ − CO, 6.0), 386 (M^+ − 2CO, 100), 358 (M^+ − 3CO, 61.7). HRMS: calcd for $C_{17}H_{22}Fe_2O_3Si_2$, *m/z* 441.9807; found, *m/z* 441.9786. Anal. Found (calcd for $C_{17}H_{22}Fe_2O_3Si_2$): C, 45.78 (46.17); H, 4.96 (5.02).

Preparation of $[CpFe(CO)]_2(\mu\text{-}SiMe_2)_2$ (3). A Pyrex glass tube (10-mm o.d.) was charged with 60 mg of 1 and 4.3 mL of toluene. After the solution was irradiated for 10 h, the reaction mixture was concentrated to half-volume. Cooling the solution to −20 °C gave a mixture of red crystals of **3a**,**b**. The crystals were collected and washed with pentane (4 × 2 mL). Yield: 25 mg (46%). ^1H NMR (C_6D_6): δ 4.09 (s, 10 H, Cp, **3b**), 3.91 (s, 10 H, Cp, **3a**), 1.24 (s, 6 H, Me, **3a**), 1.14 (s, 12 H, Me, **3b**), 1.03 (s, 6 H, Me, **3a**). ^{13}C NMR (CD_2Cl_2): δ 215.7 (CO, **3a**), 214.5 (CO, **3b**), 81.9 (Cp, **3a**), 80.8 (Cp, **3b**), 15.9 (Me, **3a**), 15.4 (Me, **3b**), 13.1 (Me, **3a**). ^{29}Si NMR (DEPT, CD_2Cl_2): δ 243.8, 229.5. IR (C_6D_6): ν(CO) 1930, 1900 cm^{-1}. MS (EI, 25 eV) [*m/z* (assignment, relative intensity)]: 414 (M^+, 43.2), 386 (M^+ − CO, 100), 358 (M^+ − 2CO, 80.3). HRMS: calcd for $C_{16}H_{22}Fe_2O_2Si_2$, *m/z* 413.9857; found, *m/z* 413.9855. Anal. Found (calcd for $C_{16}H_{22}Fe_2O_2Si_2$): C, 45.88 (46.40); H, 5.10 (5.35).

X-ray Crystal Structure Determination of 2a. Diffraction measurements were made on a Rigaku AFC-6A four-circle diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) at 21.5 °C. Crystallographic data are listed in Table II. The reflection data were corrected for Lorentz and polarization factors but not for absorption. The structure was solved by the heavy-atom method and refined by the block-diagonal least-squares method. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located on a difference Fourier synthesis and refined with isotropic temperature factors. Atomic scattering factors for non-hydrogen atoms and hydrogen atoms were taken from refs 15 and 16, respectively. Final atomic parameters of non-hydrogen atoms are listed in Table III.

The calculations were performed on a Nippon Electric Co. ACOS-2000 computer system at the Computer Center of Tohoku University by using the Universal Crystallographic Computation Program System UNICS III.[17]

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Supplementary Material Available: For **2a**, tables of atomic positional and thermal parameters and bond distances and angles and an ORTEP figure (6 pages); a listing of observed and calculated structure factors (17 pages). Ordering information is given on any current masthead page.

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(15) *International Tables for X-ray Crystallography*; Kynoch: Birmingham, England, 1974; Vol. IV: Table 2.2A, pp 72–92; Table 2.3.1.1, pp 149–150.

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