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1,1,1,3,3,3-Hexamethyltrisilanylene-bridged bis(cyclopentadienyl)tetracarbonyldiiron and its derivatives: Synthesis, properties and molecular structures

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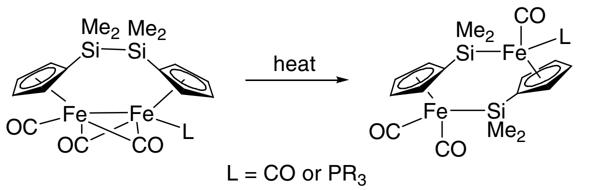
Abstract

The title ring-bridged bis(cyclopentadienyl)diiron complexes [η^5,η^5-C_5H_4–Si($SiMe_3$)$_2$–C_5H_4]Fe_2(CO)L(μ-CO)$_2$ [L = CO (**1**), P$(OPh)_3$ (**2**), P$(OMe)_3$ (**3**), PPh_3 (**4**), PMe_3 (**5**)] that contain exocyclic Si–Si bonds attached to the bridging silicon atom have been synthesized. The Si–Si bonds were found to be stable to the intramolecular iron centers under both thermal and photochemical conditions, in sharp contrast to the facile cleavage of the Si–Si bond in 1,1,2,2-tetramethyldisilanylene-bridged analogous complexes. The stability of the Si–Si bonds in the present cases may be attributed to the fact that these Si–Si bonds are spatially unapproachable by the intramolecular coordinatively unsaturated iron centers. Molecular structures of **1** and **2** have been determined by X-ray diffraction methods. An obvious conformational change due to substitution of CO for P$(OPh)_3$ was observed.

Keywords: Silicon; Iron; Cyclopentadienyl; Phosphorus; Carbonyl; X-ray diffraction

1. Introduction

Ring-bridged bis(cyclopentadienyl)tetracarbonyldiiron complexes (η^5,η^5-C_5H_4–X–C_5H_4)$Fe_2(CO)_2$(μ-CO)$_2$, in which the bridging group –X– can be either carbon- or silicon-containing segments (e.g., –CH_2– [1], –CMe_2– [2], –CH_2CH_2– [3], –CMe_2CMe_2– [4], –($CHNMe_2CHNMe_2$)– [5], –$(CH_2)_3$– [6], –$SiMe_2$– [7,8], –$Me_2SiCH_2CH_2SiMe_2$– [9] and –$Me_2SiOSiPh_2OSiMe_2$– [10]) have been extensively studied owing to their unique structural and reactive properties in the past. Of particular interest is the introduction of the Si–Si bond-containing group –$Me_2SiSiMe_2$– as the bridge, which has resulted in the unexpected discovery of a novel rearrangement reaction involving metathesis of the intramolecular Si–Si and Fe–Fe bonds under thermal conditions (Eq. (1)) [11–23].



(1)

Since the first report of this rearrangement by us in 1993 [11], much effort has been devoted to explore its scope and mechanistic details [12–23]. It was not until very recently, however, that the nature of this reaction has been recognized to involve activation of the Si–Si bond via oxidative addition to an intramolecular coordinatively unsaturated iron center (i.e. the "oxidative addition mechanism") [13,15] rather than through interaction with iron-centered free radicals (i.e. the "free radical mechanism") [24].

One of the significant features of this rearrangement is the regiospecific cleavage of the bridging Si–Si bond in the cyclic structure. This has been demonstrated previously with complexes containing additional non-bridging Si–Si

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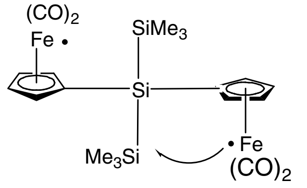


Fig. 1. Iron-centered free radicals from Fe–Fe bond homolysis of **1**.

bonds attached to the Cp rings [16,17], which furnished strong evidence to rule out the stepwise pathway of the free radical mechanism [24]. To further examine the regiospecific feature, a study on systems containing non-bridging Si–Si bonds attached to other positions, such as at the bridge, is desirable. In this aspect, we have in a recent study [25] occasionally isolated the silicon-bridged complex [η^5,η^5-C_5H_4–Si(Me)($SiMePh_2$)–C_5H_4]$Fe_2(CO)_2(\mu\text{-}CO)_2$ that contains a non-bridging exocyclic Si–Si bond at the silicon bridge. Unfortunately, the complex has been obtained as a by-product in a too small amount to study its chemistry.

In this paper, we report the first designed synthesis of such complexes by choosing the 1,1,1,3,3,3-hexamethyltrisilanylene-bridged complex [η^5,η^5-C_5H_4–Si($SiMe_3$)$_2$–C_5H_4]$Fe_2(CO)_2(\mu\text{-}CO)_2$ (**1**) and its CO ligand substitution derivatives [η^5,η^5-C_5H_4–Si($SiMe_3$)$_2$–C_5H_4]Fe_2(CO)L(μ-CO)$_2$ [L = $P(OPh)_3$ (**2**), $P(OMe)_3$ (**3**), PPh_3 (**4**), PMe_3 (**5**)] that contain two exocyclic Si–Si bonds at the silicon bridge as the target molecules. Examination of the chemical properties of these complexes under various conditions demonstrated that these Si–Si bonds could not be cleaved by the intramolecular iron centers, providing further evidence to rule out the possibility of activation of Si–Si bonds by the iron-centered free radicals from homolysis of the Fe–Fe bond (Fig. 1). On the contrary, the inertness of the Si–Si bonds towards the metal centers could be rationally explained in terms of the oxidative addition mechanism of activation of Si–Si bonds [26,27].

2. Experimental

2.1. General

All the reactions were carried out under a nitrogen or argon atmosphere by using vacuum line and Schlenk techniques. THF, *p*-xylene and hexane were dried and distilled from sodium/benzophenone. IR spectra were recorded with a Bio-Rad FTS 135 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded with a Varian UNITY Plus-400 or a Bruker AC-P200 spectrometer. Elemental analysis was performed using an Elementar Vario EL instrument. $(Me_3Si)_2SiCl_2$ was prepared according to the literature procedures [28].

2.2. Synthesis of complex **1**

To a flask containing 1.9 g (29 mmol) of cyclopentadiene and 50 ml of THF was added 14 ml (1.77 M in hexane, 25 mmol) of *n*-butyllithium at −78 °C. The mixture was stirred at room temperature for 1 h, then, 3.0 g (12 mmol) of $Me_3SiSiCl_2SiMe_3$ was added dropwise at −78 °C. The mixture was allowed to warm to room temperature, stirred at this temperature overnight and then refluxed for 2 h. After cooling to room temperature, the mixture was hydrolyzed with a saturated solution of NH_4Cl, extracted with ether, washed with water and dried over anhydrous magnesium sulfate. Evaporation of the solvent under reduced pressure gave $Me_3SiSi(C_5H_5)_2SiMe_3$ as an oil, which was used directly, without further purification, for the next step of reaction.

To a flask containing the oil product obtained above and 50 ml of *p*-xylene was added 4.9 g of $Fe(CO)_5$ (25 mmol). The mixture was refluxed with stirring overnight. The solvent was removed under reduced pressure (unreacted $Fe(CO)_5$ should be collected in a liquid nitrogen trap) to give a solid residue, which was separated through a neutral alumina column. Elution with petroleum ether/dichloromethane (4:1) gave a red band, from which 1.2 g of **1** (28% yield) was obtained as a dark-red crystalline solid, m.p. 220 °C (dec). *Anal.* Calc. for $C_{20}H_{26}O_4Fe_2Si_3$: C, 45.63; H, 4.94. Found: C, 45.64; H, 5.00%. ^{1}H NMR ($CDCl_3$): δ 0.26 (s, 9H, SiMe), 4.99 (t, $J = 2.1$ Hz, 2H, Cp), 5.46 (t, $J = 2.1$ Hz, 2H, Cp). ^{13}C NMR ($CDCl_3$): δ 0.64 (SiMe), 86.45, 86.83, 101.38 (Cp), 209.83 (terminal CO), 272.65 (bridging CO). IR (KBr, cm^{-1}): ν_{co} 1993 (s), 1949 (s), 1774 (s).

2.3. Synthesis of complexes **2–5**

2.3.1. Thermal reactions of **1** with $P(OPh)_3$ and $P(OMe)_3$

To a flask charged with 100 mg (0.19 mmol) of **1** and 10 ml of *p*-xylene was added 92 mg (0.74 mmol) of $P(OPh)_3$. The mixture was refluxed for 16 h with magnetic stirring. The solvent was removed under reduced pressure. The residue was separated through a neutral alumina column. Elution with petroleum ether/dichloromethane (4:1) developed a green band, from which 46 mg (36% yield) of **2** was afforded as black crystals. In a similar fashion, **3** was prepared (32% yield) from **1** and $P(OMe)_3$ after refluxing for 24 h.

For **2**: m.p. 210 °C (dec). *Anal.* Calc. for $C_{37}H_{41}O_6Fe_2PSi_3$: C, 54.95; H, 5.07. Found: C, 54.88; H, 5.08%. ^{1}H NMR ($CDCl_3$): δ 0.23 (s, 18H, SiMe), 4.29, 4.49, 4.97, 5.41 (br s, 2H:2H:2H:2H, Cp), 7.16–7.33 (m, 15H, Ph). ^{13}C NMR ($CDCl_3$): δ 0.69 (SiMe); 83.56, 85.68, 86.34, 88.32, 98.09, 99.82 (Cp); 121.75 (d, $J =$ 3.6 Hz, Ph), 124.70 (Ph), 129.59 (Ph), 151.51 (d, $J = 9.9$ Hz, Ph), 213.96 (terminal CO), 278.62 (d, $J = 21.5$ Hz, bridging CO). IR (KBr, cm^{-1}): ν_{co} 1993 (s), 1964 (s), 1777 (s), 1746 (s).

For **3**: m.p. 208 °C (dec). *Anal.* Calc. for $C_{22}H_{35}O_6Fe_2Si_3P$: C, 42.44; H, 5.63. Found: C, 42.48; H, 5.61%. ^{1}H NMR ($CDCl_3$): δ 0.26 (s, 18H, $SiMe_3$), 3.55 (d, $J = 11.1$ Hz, 9H, OCH_3), 4.81, 4.90, 4.91, 5.32 (br s, 2H:2H:2H:2H, Cp). ^{13}C NMR ($CDCl_3$): δ 0.75 (SiMe), 52.06 (d, $J = 4.93$ Hz, OCH_3), 82.63, 85.40, 85.76, 87.13, 99.11, 100.28 (Cp), 214.51 (terminal CO), 281.90 (d, $J = 24.65$ Hz, bridging CO). IR (KBr, cm^{-1}): ν_{co} 1995 (s), 1963 (s), 1774 (s), 1730 (s).

2.3.2. *Photochemical reactions of **1** with PPh_3 and PMe_3*

A solution containing 100 mg (0.19 mmol) of **1**, 199 mg (0.76 mmol) of PPh_3 and 20 ml benzene in a Pyrex tube was irradiated with a high-pressure mercury lamp (500 W) for 10 h at room temperature. The solvent was removed under reduced pressure and the residue was separated through a column (neutral alumina, petroleum ether/ether, 10:1). Collection of the dark green band gave black crystals of **4** (90 mg, 62% yield). In a similar fashion, **5** was obtained by using PMe_3 in place of PPh_3 as grey crystals in 53% yield.

For **4**: m.p. 220 °C (dec). *Anal.* Calc. for $C_{37}H_{41}Fe_2O_3PSi_3$: C, 58.42; H, 5.43. Found: C, 58.41; H, 5.40%. ^1H NMR ($CDCl_3$): δ 0.27 (s, 18H, SiMe), 4.19, 4.90, 4.91, 5.12 (br s, 2H:2H:2H:2H, Cp), 7.24–7.53 (m, 15H, Ph).^{13}C NMR ($CDCl_3$): δ 0.77 (SiMe); 82.00, 86.36, 88.84, 90.00, 97.72, 98.61 (Cp); 128.65 (d, $J = 9.9$ Hz, Ph), 129.77 (Ph), 134.21 (d, $J = 9.2$ Hz, Ph), 135.84 (d, $J = 41.7$ Hz, Ph), 212.88 (terminal CO), 284.11 (d, $J = 14.8$ Hz, bridging CO). IR: (KBr, cm^{-1}): ν_{co} 1955 (s), 1766 (s), 1730 (s).

For **5**: m.p. 230 °C (dec) *Anal.* Calc. for $C_{22}H_{35}Fe_2O_3PSi_3$: C, 46.0; H, 6.14. Found: C, 45.87; H, 5.90%. ^1H NMR ($CDCl_3$): δ 0.26 (s, 18H, SiMe), 1.00 (d, $J = 9.0$ Hz, 9H, CH_3), 4.77, 4.88, 4.94, 5.29 (br s, 2H:2H:2H:2H, Cp). ^{13}C NMR ($CDCl_3$): δ 0.76 (SiMe), 18.78 (d, $J = 28.2$ Hz, CH_3); 80.89, 85.60, 85.81, 88.57, 99.24 (Cp); 217.20 (terminal CO), 283.80 (d, $J = 16.0$ Hz, bridging CO). IR (KBr, cm^{-1}): ν_{co} 1933 (s), 1767 (s), 1729 (s).

Table 1
Crystallographic data of **1** and **2**

	1	**2**
Formula	$C_{20}H_{26}Fe_2O_4Si_3$	$C_{37}H_{41}Fe_2O_6PSi_3$
Formula weight	526.38	808.64
Temperature (K)	293(2)	293(2)
Crystal size (mm)	0.20 × 0.16 × 0.10	0.24 × 0.22 × 0.18
Crystal system	orthorhombic	monoclinic
Space group	*Pnma*	*P*2(1)/*n*
a (Å)	21.295(8)	13.167(4)
b (Å)	12.203(4)	17.500(5)
c (Å)	9.363(3)	17.057(5)
α (°)	90	90
β (°)	90	99.681(5)
γ (°)	90	90
V ($Å^3$)	2433.2(15)	3874.5(18)
Z	4	4
D_{cal} ($g\ cm^{-3}$)	1.437	1.386
Instrument	Bruker Smart 1000	Bruker Smart 1000
Wavelength (Å)	0.71073	0.71073
$F(000)$	1088	1680
θ Range (°)	1.91–26.37	1.81–25.00
N_{total}	13304	19358
$N_{independent}$	2612	6714
$N_{observed}$	1693	3131
Parameters	157	442
Restrains	8	0
Goodness-of-fit	1.036	0.926
R	0.0471	0.0566
R_w	0.0836	0.0819

2.4. *X-ray molecular structure determination of **1** and **2***

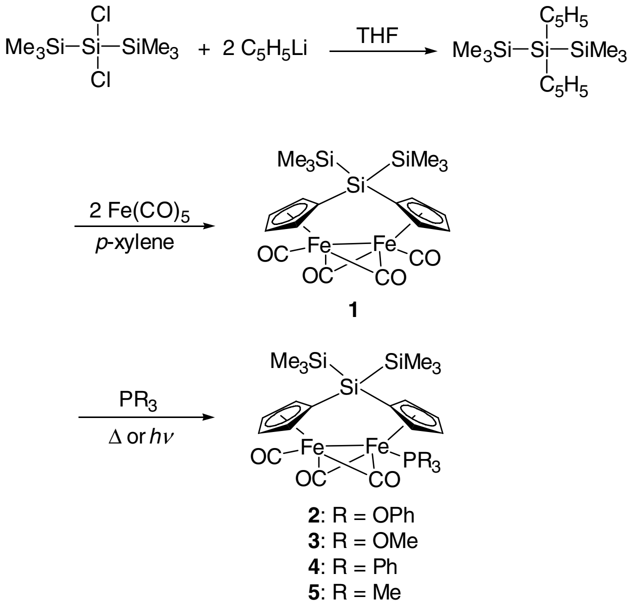
Single crystals of **1** and **2** suitable for X-ray diffraction analyses were obtained from *n*-hexane/dichloromethane solutions. Data collections were performed on a BRUKER SMART 1000 diffractometer at room temperature using $2\theta/\omega$ scan techniques with Mo Kα radiation ($\lambda = 0.71073$ Å). The structures were solved by direct methods using the SHELX-90 program and refined by least square methods on F^2. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added theoretically and included in the refinement processes in an isotropic manner. Crystal data and selected experimental information have been listed in Table 1.

3. Results and discussion

3.1. *Synthesis of complexes*

Synthesis of complex **1** was accomplished through the reaction of $(Me_3Si)_2Si(C_5H_5)_2$, prepared from $(Me_3Si)_2SiCl_2$ and two equivalents of C_5H_5Li in THF, with $Fe(CO)_5$ in refluxing *p*-xylene in a total yield of 28% (Scheme 1).

When **1** was heated in refluxing *p*-xylene in the presence of $P(OPh)_3$ or $P(OMe)_3$, CO-substituted derivatives **2** and **3** were obtained, respectively. The CO substitution reaction with PPh_3, which did not occur under thermal conditions



Scheme 1. Synthesis of the complexes.

due to steric hindrance, could be carried out under photochemical conditions, to provide complex **4**. The same kind of reaction of **1** with PMe_3, which is too volatile to be used at high temperatures, could be effected photochemically at room temperature to afford complex **5** (Scheme 1).

Complexes **1**–**5** were characterized by elemental analyses and spectroscopic methods. The ^{1}H NMR spectrum of **1** showed a singlet at 0.26 ppm for the SiMe protons and two sets of pseudotriplets at 4.99 and 5.46 ppm for protons of the Cp groups. After substitution of CO by a phosphite or phosphine ligand, the singlet due to the SiMe groups remained little changed, whereas the signals due to the Cp protons split into four sets, demonstrating successful substitution of CO by a phosphorus ligand. Signals due to PR_3 were also observed. The ^{13}C NMR spectrum of **1** exhibited one signal at 0.64 ppm for the SiMe carbons, three signals at 86.45, 86.83 and 101.38 ppm for Cp carbons, and two signals at 209.83 and 272.65 ppm for terminal and bridging CO carbons, respectively. The spectra of the PR_3-substituted derivatives showed generally one signal for SiMe carbons, six signals due to Cp carbons, a singlet and a doublet for terminal and bridging CO, respectively, along with those attributable to the PR_3 ligands. The doublet signal for the bridging CO was due to coupling with the adjacent phosphorus nucleus. IR spectra of these complexes showed absorption bands for both terminal and bridging CO groups, just as expected.

3.2. Stability of the Si–Si bonds

It was found that the Si–Si bonds of **1** were stable under the conditions of refluxing in *p*-xylene. This is in sharp contrast to the case of the $–Me_2SiSiMe_2–$ bridged complexes [11,12], in which the bridging Si–Si bond could be readily broken under the same conditions. Moreover, the Si–Si bonds of **1** were also stable under UV irradiation. The stability of the Si–Si bonds has allowed the CO substitution reactions of **1** to be operated under either thermal or photochemical conditions. The Si–Si bonds in the ligand substituted derivatives **2**–**5** were also found to be stable under both thermal and photochemical conditions. Since it has been well established that the phosphorus ligands can considerably accelerate the Si–Si bond activation in the Si–Si bond-bridged systems [12], the present results could further strengthen the conclusion that these exocyclic Si–Si bonds could not be activated by the intramolecular transition metal centers.

Since iron-centered free radicals could easily be produced under the thermal and photochemical conditions [29,30], the stableness of the Si–Si bonds means that these radicals are not able to activate the Si–Si bonds. Also formed under these conditions could be the coordinatively unsaturated iron species from dissociation of the appropriate ligands [30–32]. Although these species are believed to be responsible for activation of the bridging Si–Si bond as concluded in the previous paper [15], they are apparently not able to cleave the non-bridging exocyclic Si–Si bonds in the present systems. This should be attributed to the fact that these Si–Si bonds are located in a position such that they could not be approached by the intramolecular iron center, due to restriction of the cyclic structure. Such spatial restriction, however, does not exist in the structure of the iron-centered free radical (Fig. 1), because the cyclic structure has been opened after breaking the Fe–Fe bond.

3.3. Molecular structures

The molecular structures of **1** and **2** have been determined by X-ray diffraction methods. The molecule of **1** (Fig. 2) contains a disordered Me_3Si group that has two rotational positions (each at 50% occupancy). The overall molecule has C_s symmetry. The symmetrical plane goes through three silicon atoms and the center of the Fe–Fe

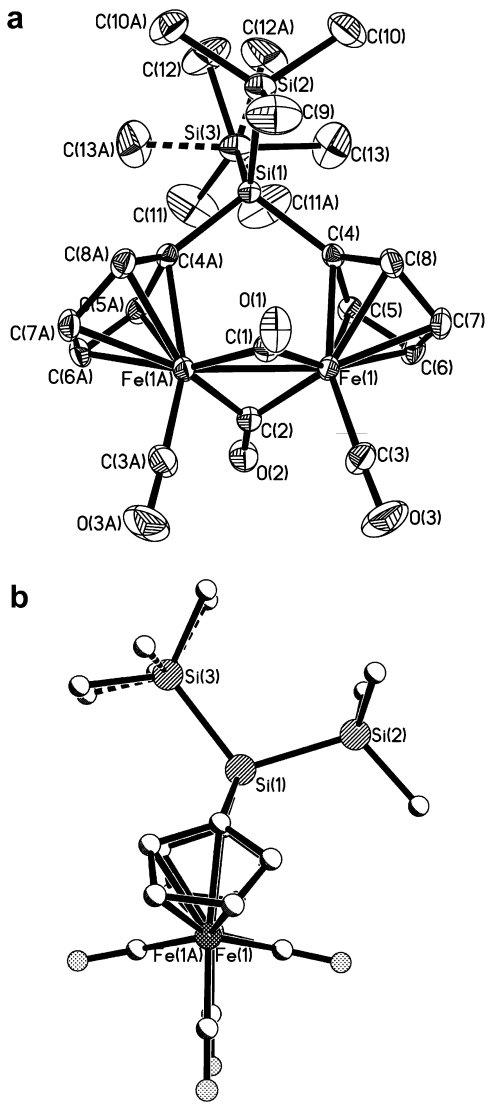


Fig. 2. (a) ORTEP drawing of $[\eta^5,\eta^5\text{-}C_5H_4\text{–}Si(Me_3Si)_2\text{–}C_5H_4]Fe_2(\mu\text{-}CO)_2(CO)_2$ (**1**). Thermal ellipsoids are at the 30% probability level. Hydrogen atoms are omitted for clarity. (b) Side view of **1**.

bond. The bridging silicon atom, however, is not located on the top of the molecule but moves away from the plane defined by two iron and two bridged-head Cp carbon atoms by a distance of 0.58 Å (see Fig. 2b). This results in the five-membered ring composed of the bridging silicon, two iron and two the bridged-head Cp carbon atoms taking an envelope-like conformation. This type of conformation has previously been observed in other silicon- and carbon-bridged complexes [2,7,8]. Interestingly, in complex **2** the bridging silicon atom has moved onto the top of the molecule (Fig. 3). Thus, the molecule has C_1 symmetry in the crystalline state. The five-membered cyclic skeleton is basically planar, with a mean deviation of only 0.011 Å (Fig. 3b). This is, to our knowledge, the first example of a silicon-bridged complexes to have such a conformation.

The shift of the bridge onto the top of the molecule is likely caused by the steric effect of the $P(OPh)_3$ ligand. Meanwhile, the bulkiness of $P(OPh)_3$ makes the Fe–Fe bond slightly lengthened from 2.5227 Å for **1** to 2.5311 Å for **2**. The Cp–Si–Cp angle is also increased from 103.7° for **1** to 105.3° for **2**. Interestingly, the bridging Si–Cp bonds have been slightly shortened from 1.886 Å for **1** to 1.870/1.880 Å for **2**, implying that the original strain that may exist in the five-membered cyclic structure may have been released to some extent. The lengths of the Si–Si bonds are 2.362/2.361 Å for **1** and 2.355/2.351 Å for **2**, which are rather close to one another. Other selected bond lengths and angles for **1** and **2** have been listed in Tables 2 and 3, respectively.

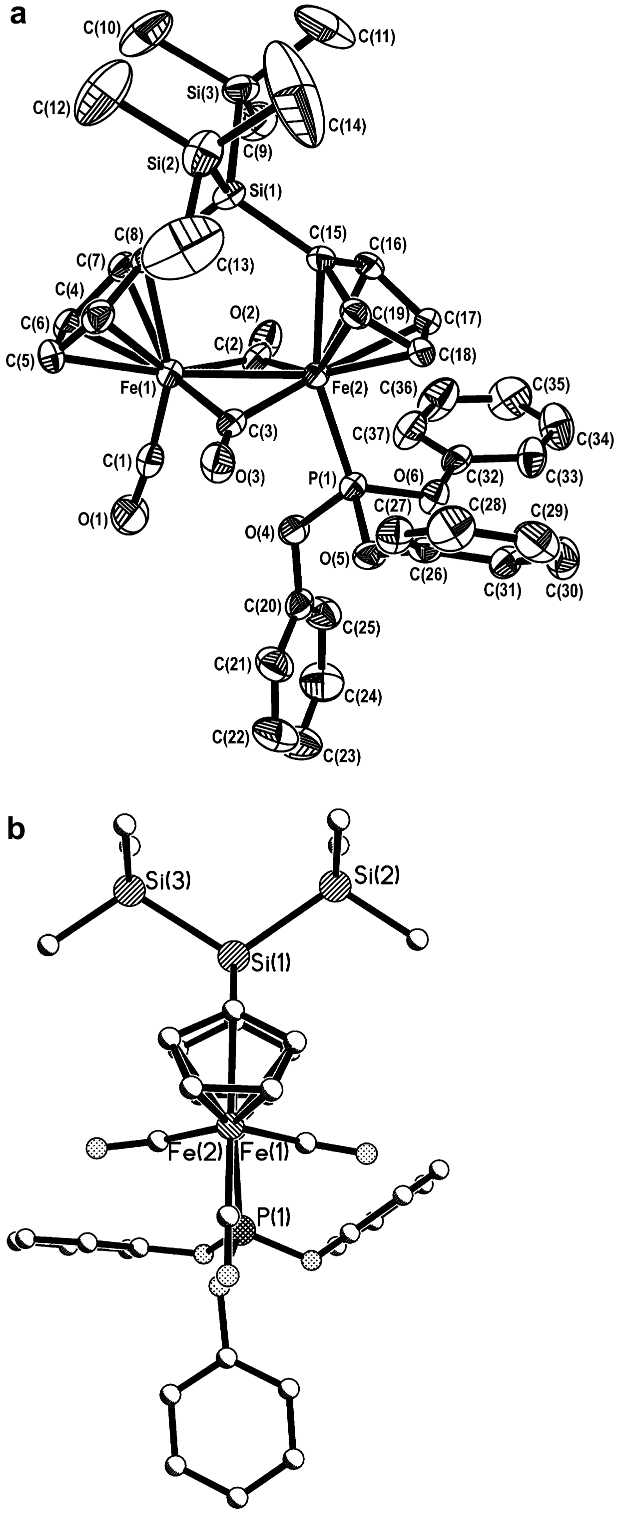


Fig. 3. (a) ORTEP drawing of $[\eta^5,\eta^5\text{-}C_5H_4\text{–}Si(Me_3Si)_2\text{–}C_5H_4]Fe_2(\mu\text{-}CO)_2(CO)P(OPh)_3$ (**2**). Thermal ellipsoids are at the 30% probability level. Hydrogen atoms are omitted for clarity. (b) Side view of **2**.

Table 2
Selected bond lengths (Å) and angles (°) for **1**

Fe(1)–Fe(1A)	2.523(1)	Si(1)–C(4)	1.886(4)
Si(1)–Si(2)	2.361(2)	Si(1)–Si(3)	2.362(2)
Fe(1)–C(4)	2.143(4)	Fe(1)–C(3)	1.752(5)
Fe(1)–C(2)	1.939(5)	Fe(1)–C(1)	1.930(4)
C(4)–Si(1)–C(4A)	103.7(2)	Si(2)–Si(1)–Si(3)	110.4(1)
C(4)–Si(1)–Si(2)	111.3(1)	C(4)–Si(1)–Si(3)	110.0(1)
Si(1)–C(4)–Fe(1)	121.2(2)	C(4)–Fe(1)–Fe(1A)	95.9(1)

Table 3
Selected bond lengths (Å) and angles (°) for **2**

Fe(1)–Fe(2)	2.531(1)	Fe(2)-P(1)	2.123(2)
Si(1)–Si(2)	2.351(2)	Si(1)–Si(3)	2.355(2)
Si(1)–C(8)	1.870(5)	Si(1)–C(15)	1.880(5)
Fe(1)–C(8)	2.146(5)	Fe(2)–C(15)	2.138(5)
Fe(1)–C(1)	1.735(7)	Fe(1)–C(2)	1.922(6)
Fe(1)–C(3)	1.933(5)	Fe(2)–C(2)	1.915(6)
Fe(2)–C(3)	1.906(5)		
Si(2)–Si(1)–Si(3)	113.0(1)	C(8)–Si(1)–C(15)	105.3(2)
C(8)–Si(1)–Si(2)	109.1(2)	C(15)–Si(1)–Si(2)	111.0(2)
C(8)–Si(1)–Si(3)	108.6(2)	C(15)–Si(1)–Si(3)	109.6(2)
C(8)–Fe(1)–Fe(2)	97.2(1)	C(15)–Fe(2)–Fe(1)	94.9(1)
P(1)–Fe(2)–Fe(1)	105.6(1)	Si(1)–C(15)–Fe(2)	122.3(2)
Si(1)–C(8)–Fe(1)	120.2(2)		

4. Summary

According to the results observed above, it seems that an appropriate position of the Si–Si bonds with respect to the transition metal centers should be an important factor that may control the intramolecular Si–Si bond activation. Without such a position that is approachable by the intramolecular coordinatively unsaturated transition metal centers, the Si–Si bond would remain stable, although Si–Si bonds should be highly susceptible towards oxidative addition to intramolecular transition metal centers [33,34].

Acknowledgements

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Appendix A. Supplementary material

CCDC 280002 and 280003 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2006.10.026.

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