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Synthesis and Structure of [Fe(η^5-C_9Me_6)(η^5-C_5H_4)$SiMe_2$]: A Mixed-Ring [1]Ferrocenophane

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Summary: A new bridged ligand incorporating the hexamethylindenyl moiety has been synthesized and used to prepare the corresponding ansa ferrocene, [Fe(η^5-C_9Me_6)(η^5-C_5H_4)$SiMe_2$], ***10****, which is the first mixed-ring [1]ferrocenophane to be structurally characterized. Comparison of the crystal structure of* ***10*** *with those of [Fe(η^5-C_5H_4)$_2SiMe_2$],* ***1****, and rac-[Fe(η^5-C_9Me_6)$_2SiMe_2$],* ***5a****, shows many structural features, such as the ring tilt of 17°, to be intermediate between those of the two model compounds, while the average Si−C(ipso) bond length is much closer to that in* ***5a****.*

Introduction

One of the most interesting and useful features of [1]-ferrocenophanes is their ability to undergo stoichiometric ring-opening reactions. These reactions have been used to obtain high molecular weight organometallic polymers,[1−3] which have been studied for applications ranging from electrochromic films[4] to precursors for new ceramics,[5] and to derivatize surfaces[6] and the walls of mesoporous solids.[7−9] As part of an effort to understand the relationship between the electronic and structural features of these compounds, we have reported studies of a series of ring-methylated $SiMe_2$-bridged [1]ferrocenophanes (**1**−**4**, Figure 1) using ^{57}Fe Mössbauer, UV−vis and UV−photoelectron spectroscopy, crystallography, electrochemistry, and density functional theory.[10,11] We have also reported the synthesis and structure of the first dibenzo[1]ferrocenophane (i.e., the first bridged bis(indenyl)iron complex): [Fe(η^5-C_9Me_6)$_2SiMe_2$], **5** (Figure 1).[12] We found a correlation between the electrochemical oxidation potential, $E_{1/2}$, and the crystallographically determined ring tilt, α (defined by Figure 2); both $E_{1/2}$ and α decrease along the series **1**, **2**, **4**, **5**, the decrease in α being compensated by an increase in the structural distortion of the bridging group. This correlation can be understood in terms of the greater energy cost calculated to be associated with ring tilt in more electron-rich methylated metallocenes.[11] We were interested in investigating what structural features would be found in a compound such as **3**, in which the two coordinated rings differed markedly in their electronic properties. However, crystals of **3** suitable for a structure determination have not been obtained. Hence, we report here on the synthesis, structure, and polymerization behavior of [Fe(η^5-C_9Me_6)(η^5-C_5H_4)$SiMe_2$], **10**,

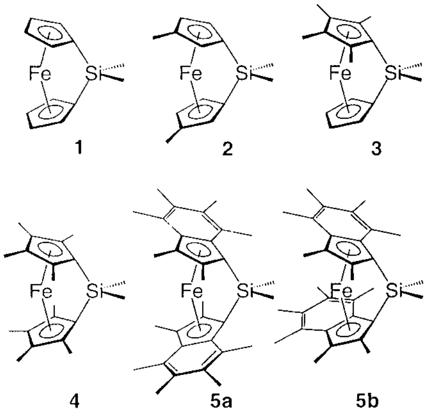

Figure 1. Some $SiMe_2$-bridged [1]ferrocenophanes.

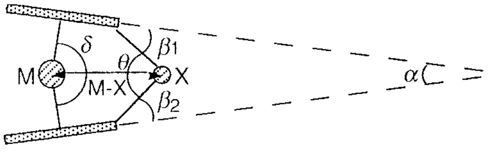

Figure 2. Some parameters useful for the discussion of the structures of [1]ferrocenophanes.

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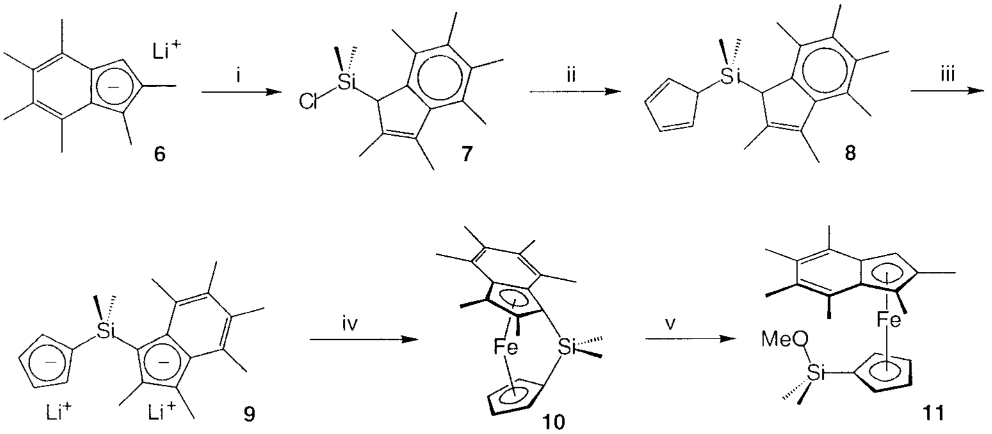
Scheme 1[a]



[a] Legend: (i) Me_2SiCl_2, petroleum ether (bp 40−60 °C); (ii) NaCp or $MgCp_2$, THF; (iii) ^nBuLi, TMEDA, petroleum ether (bp 40−60 °C); (iv) $FeCl_2 \cdot 1.5THF$ or $FeBr_2 \cdot 2THF$, THF; (v) MeOH, THF.

which combines the ligated rings from the two extreme members of the series **1**−**5**.

Results and Discussion

The synthesis of [Fe(η^5-C_9Me_6)(η^5-C_5H_4)$SiMe_2$], **10**, is outlined in Scheme 1. Reaction of lithium 1,2,4,5,6,7-hexamethylindenide, **6**,[12] with dichlorodimethylsilane gave [(C_9Me_6H)$SiMe_2Cl$], **7**, which can be converted to [(C_9Me_6H)(C_5H_5)$SiMe_2$], **8**, with either sodium cyclopentadienide or magnesocene. Reaction of doubly lithiated **8** (**9**) with either $FeCl_2 \cdot 1.5THF$ or $FeBr_2 \cdot 2THF$ in THF gave modest yields of [Fe(η^5-C_9Me_6)(η^5-C_5H_4)$SiMe_2$], **10**. ^1H and ^{13}C NMR spectroscopy, mass spectrometry, and X-ray crystallography (vide infra) all show **10** to be a strained monomeric species (rather than a dimer or higher oligomer). The large spread in C_5H_4 ^1H chemical shifts is typical of ring-tilted ferrocenes. The ^{13}C signals corresponding to the ipso bridgehead carbons of **10** occur at chemical shifts (δ in C_6D_6) of 21.0 and 33.9 ppm. These values, although very low δ for formally sp^2 carbons, are typical for the ipso carbons of strained ferrocenophanes, reflecting the nonplanar geometry around these atoms (which is seen in their crystal structures); the corresponding resonances for **1**, **5a**, and **5b** are seen at 33.5, 21.7, and 20.3 ppm, respectively.[12]

Single crystals of **10** were obtained by slow cooling of a pentane solution. The crystal structure was solved and refined in the centrosymmetric space group $P\bar{1}$, with one molecule in the asymmetric unit. Figure 3 shows the molecular structure, while Figure 2 defines the parameters used in the following discussion. The Fe−C bond lengths are similar to those in other silicon-bridged [1]ferrocenophanes;[10,12−19] the longest Fe−C bonds are those to the carbons at the indenyl ring junction, as is found for other η^5-indenyl compounds (for examples see refs 20−27[16]) including **5a**.[12] The ring tilt (α) for **10** (17.2°) is similar to the average (17.1°) of those for **1** (20.8°)[14] and **5a** (average 13.4° for two inequivalent molecules),[12] consistent with **10** being electronically intermediate between **1** and **5a**. The angle (β_1) between the cyclopentadienyl ring of **10** and the ipso carbon−silicon (C1−Si) bond is 39.2°, while the corresponding angle, β_2, for the permethylindenyl side of the molecule is 41.2°. The angles, β_{av}, for **1** and **5a** are 37.0° and 42.2°,

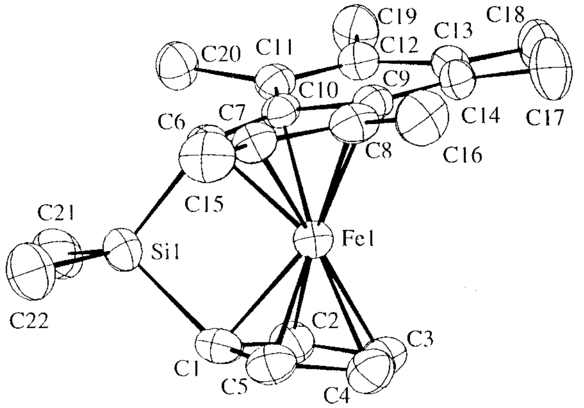


Figure 3. Molecular structure of **10** (50% thermal probability ellipsoids, H atoms excluded for clarity). Selected bond lengths: Fe(1)−C(1) 2.010(4) Å, Fe(1)−C(2) 2.017(5) Å, Fe(1)−C(3) 2.063(5) Å, Fe(1)−C(4) 2.069(5) Å, Fe(1)−C(5) 2.015(5) Å, Fe(1)−C(6) 2.010(4) Å, Fe(1)−C(7) 2.032(4) Å, Fe(1)−C(8) 2.056(4) Å, Fe(1)−C(9) 2.094(4) Å, Fe(1)−C(10) 2.093(4) Å.

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respectively. The Fe−Si distance of 2.668(1) Å and the angles δ (167.5°), θ (97.4(2)°) and Me−Si−Me (106.8(3)°) are also intermediate between the corresponding values for **1** and **5a**. However, while the average Si−C(ipso) bond lengths steadily increase from 1.858(9)° for **1** to 1.905(8)° for **5a** as α decreases, both the Si−C(ipso) bonds in **10** are rather long: Si−C1 (cyclopentadienyl) and Si−C6 (permethylindenyl) distances for **10** are 1.886(5)° and 1.927(4)°, respectively.

Attempts to compare the electrochemistry of **10** to that of **1**−**5** were complicated by the appearance of additional features in the cyclic voltammogram, and of a precipitate, after several scans. Thus, $\mathbf{10}^+$ is rather less stable than the cations of **1**−**5**; the reasons for this are unclear. We estimate the half-wave potential for the $\mathbf{10}^+$/**10** couple to be −150 mV vs ferrocenium/ferrocene. It is surprsing that this value is much closer to that for **1** (0 mV) than for **5** (−690 mV). The value for **3** (−210 mV), by contrast, is close to the average of the values for **1** and **4** (−390 mV).

Differential scanning calorimetric (DSC) studies of **10** showed endotherms at 50 and 160 °C, with no evidence for any polymerization exotherm below 300 °C (the highest temperature examined). Gel permeation chromatography (GPC) showed no high molecular weight products from a bulk polymerization attempt at 260 °C (8 h), although ^{1}H NMR spectroscopy showed that no **10** remained, thus suggesting that the higher of the two endotherms relates to decomposition of **10**. Attempts to thermally ring-opening polymerize **5** also result in decomposition.[28] [1]Ferrocenophanes have also been polymerized at room temperature using late transition metal catalysts;[29,30] therefore, we attempted to polymerize **10** using platinum(II) chloride in C_6D_6. After 5 days, ^{1}H NMR spectroscopy showed only unchanged **10** and GPC showed no high molecular weight products.

The reaction of **10** with methanol gave a single ring-opened product, [Fe(η^5-C_9Me_6H)(η^5-$C_5H_4SiMe_2OMe$)], **11**, with no evidence for the formation of [Fe(η^5-$C_9Me_6SiMe_2OMe$)(η^5-C_5H_5)]. The observed regiospecificity suggests that this ring-opening may proceed via protonation of the most electron-rich ipso carbon, rather than by nucleophilic attack at silicon followed by ring opening to give the most stable carbanion. The same regioselectivity was observed when **10** was ring-opened to functionalize the pores of the mesoporous silica FSM-16.[9]

Experimental Details

General Procedures. Elemental analyses were performed by the analytical department of the Inorganic Chemistry Laboratory. NMR spectra were recorded using a Bruker AM 300 or a Varian Unity Plus 500. Spectra were referenced relative to tetramethylsilane using the residual protio-solvent signal. Electron impact mass spectra were recorded using a VG 70-250-S instrument. Cyclic voltammograms were recorded using a platinum working and auxiliary and silver wire pseudo-reference electrode. Measurements were made under nitrogen on deoxygenated dry dichloromethane solutions ca. 10^{-2} M in sample and 0.1 M in $[Bu_4N]^+[PF_6]^-$. Ferrocene was used as an internal reference. Oxygen- or water-sensitive materials were handled using standard Schlenk techniques or a Vacuum Atmospheres glovebox. Where necessary, solvents were dried by distillation under nitrogen from potassium (THF, benzene-d_6) or sodium−potassium alloy (pentane, petroleum ether (bp 40−60 °C), diethyl ether). Reagents were either synthesized according to the references given or were purchased from Aldrich and used without further purification, unless stated otherwise.

Synthesis. [(C_9Me_6H)$SiMe_2Cl$], 7. A solution of dimethyldichlorosilane (freshly distilled from magnesium turnings, 34 mL, 280 mmol) in petroleum ether (bp 40−60 °C) (500 mL) was added dropwise to a stirred slurry of [C_9Me_6HLi],[12] **6**, (53.0 g, 257 mmol) in petroleum ether (bp 40−60 °C) (100 mL) at room temperature. After stirring the reaction mixture for a further 18 h, the mixture was filtered through Celite, concentrated under reduced pressure, and cooled to −80 °C. [$C_9Me_6HSiMe_2Cl$], **7** (14.5 g, 49 mmol, 66%), was obtained as white needles. Anal. Found (calcd): C, 69.9 (69.7); H, 9.0, (8.6). ^{1}H NMR (C_6D_6): δ −0.09 (s, 3H), 0.20 (s, 3H), 2.06 (s, 3H), 2.12 (apparent s, 6H), 2.13 (s, 3H), 2.15 (s, 3H), 2.42 (s, 3H), 3.48 (s, 1H). ^{13}C NMR (C_6D_6): δ −0.5, 3.21, 15.2, 15.3, 16.2, 16.3, 16.4, 19.2, 49.7, 126.5, 127.5, 130.4, 133.1, 134.4, 137.9, 139.7, 142.1. MS (EI): m/z 293 (M^+, 71%), 258 ($M^+ - Cl$, 96), 200 ($M^+ - SiMe_2Cl$, 50).

[(C_9Me_6H)(C_5H_4)$SiMe_2$], 8. Method A. A solution of sodium cyclopentadienide[31] (1.36 g, 15.4 mmol) in THF (50 mL) was added dropwise to a solution of **7** (4.5 g, 15.4 mmol) in THF (75 mL) at 0 °C. The reaction mixture was then allowed to warm to room temperature and stirred for a further 20 h. Excess solid ammonium chloride was then added to the reaction mixture, the solvent was removed under reduced pressure, and the residue was extracted with pentane. Evaporation of the pentane extracts under reduced pressure afforded a white solid (3.27 g, 9.86 mmol, 60%).

Method B. A solution of **7** (45.0 g, 154 mmol) in THF (600 mL) was added dropwise to a solution of magnesocene[32] (13.0 g, 84 mmol) in THF (200 mL) at 0 °C. The reaction mixture was then allowed to warm to room temperature and stirred for a further 16 h. The reaction was worked up in a fashion analogous to method A (45.3 g, 140 mmol, 91%). In each case **8** was converted to **9** without further characterization owing to its poor thermo- and photostability.

[(C_9Me_6Li)(C_5H_3Li)$SiMe_2$], 9. A hexane solution of n-butyllithium (112 mL of a 2.74 M solution, 307 mmol) was added dropwise to a solution of **8** (45.3 g, 140 mmol) and N,N,N',N'-tetramethylethylenediamine (TMEDA, distilled from calcium hydride, 46 mL, 310 mmol) in petroleum ether (bp 40−60 °C) (800 mL) at 0 °C. The reaction mixture was then allowed to warm to room temperature and stirred for a further 12 h, after which time the precipitate was collected on a frit, washed with diethyl ether (100 mL) and petroleum ether (bp 40−60 °C) (100 mL), and dried in vacuo to give **9** as a white air-sensitive powder (46.4 g, 139 mmol, 99% assuming no TMEDA incorporation).

[Fe(η^5-C_9Me_6)(η^5-C_5H_4)$SiMe_2$], 10. A solution of **9** (1.19 g, 3.46 mmol) in THF (50 mL) was added dropwise over 1.5 h to a slurry of $FeCl_2 \cdot 1.5THF$[33] (0.85 g, 4.26 mmol) in THF (50 mL) at 0 °C. The reaction mixture was then allowed to warm to room temperature and stirred for a further 3 h; the solvent was then removed under reduced pressure, and the residue was extracted with pentane. The pentane extracts were filtered through Celite, concentrated under reduced pressure, and cooled to −30 °C to give air-sensitive dark red crystals of **10** (0.365 g, 28%). In another reaction, $FeBr_2 \cdot 2THF$[33] was used in place of $FeCl_2 \cdot 1.5THF$, and the reaction mixture was only

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stirred for 0.5 h at 0 °C before solvent removal (yield, 35%). Anal. Found (calcd): C, 69.9 (70.2); H, 7.7 (7.5); Fe, 14.8 (14.5). ^{1}H NMR (C_6D_6): δ 0.75 (s, 3H), 0.90 (s, 3H), 1.83 (s, 3H), 1.99 (s, 3H), 2.06 (s, 3H), 2.35 (s, 3H), 2.41 (s, 3H), 2.57 (s, 3H), 3.12 (m, 1H), 3.30 (m, 1H), 3.74 (m, 1H), 4.39 (m, 1H). ^{13}C NMR (C_6D_6): δ 2.2, 4.8, 15.2, 15.4, 16.5, 16.9, 17.5, 21.0, 22.2, 33.9, 76.4, 78.3, 79.1, 79.2, 84.5, 92.8, 93.7, 100.6, 129.4, 130.8, 132.6, 133.4. MS (EI): *m*/*z* 376 (M^+, 100), 361 ($M^+ -$ Me, 55).

Reaction of 10 with Methanol. Methanol (1 mL) was added to a solution of **10** (100 mg) in THF (30 mL) at −78 °C. The reaction mixture was then allowed to warm to room temperature and stirred for a further 12 h; the solvent was then removed under reduced pressure, and the residue was extracted into benzene-d_6. ^{1}H NMR (C_6D_6): δ 0.30 (s, 3H), 0.36 (s, 3H), 1.85 (s, 3H), 2.07 (s, 3H), 2.09 (s, 3H), 2.33 (s, 3H), 2.41 (s, 3H), 2.58 (s, 3H), 3.28 (s, 3H), 3.50 (m, 1H), 3.52 (m, 1H), 3.61 (m, 1H), 4.03 (m, 1H), 4.63 (s, 1H). ^{13}C NMR (C_6D_6): δ −1.7, −1.6, 13.9, 14.9, 16.4, 16.6, 16.8, 16.9, 50.0, 61.6, 70.2, 72.6, 73.8, 73.9, 74.0, 75.8, 85.0, 86.5, 87.6, 129.1, 129.9, 130.0, 131.3.

Crystal Structure of 10. Single crystals of $C_{22}H_{28}FeSi$, fw = 376.40, were grown by slow cooling of a pentane solution. A dark red plate (0.09 × 0.54 × 0.62 mm^3) was mounted on an Enraf Nonius CAD-4 diffractometer, using Mo Kα radiation and found to be triclinic with $a = 8.753(4)$ Å, $b = 9.251(2)$ Å, $c = 12.052(3)$ Å, $\alpha = 92.05(2)°$, $\beta = 105.17(2)°$, $\gamma = 109.21(2)°$, $V = 963(1)$ Å^3, $Z = 2$, and $\mu = 0.880$ mm^{-1}; 2985 (2570 independent, 1954 with $I > 2\sigma(I)$) reflections were measured over the range 0−23.0° at 298 K using $\omega-2\theta$ scans. The structure was solved in the space group $P\bar{1}$ using SIR92;[34] all non-hydrogen atoms were revealed by the direct methods solution, while hydrogen atoms were placed in geometrically idealized positions and given isotropic thermal parameters. An absorption correction was applied using DIFABS.[35] A Chebyshev weighting scheme was applied in the refinement.[36] Corrections for anomalous dispersion and isotropic extinction were made via an overall extinction parameter.[37] Full refinement (positional and anisotropic displacement parameters of non-hydrogen atoms, positional and isotropic displacement parameters of hydrogen atoms) on F converged with $R = 0.0480$, $R_w = 0.0567$, $S = 1.090$, $\Delta\rho_{max} = 0.47$ e Å^{-3}, and $\Delta\rho_{min} = -0.50$ e Å^{-3}. All crystallographic calculations were performed using CRYSTALS[38] running on a Silicon Graphics Indigo R3000.

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Supporting Information Available: Tables of crystal structure solution and refinement details, atomic coordinates and equivalent isotropic displacement parameters, bond lengths and angles, anisotropic displacement parameters, hydrogen coordinates, and isotropic displacement parameters for **10**. This material is available free of charge via the Internet at http://pubs.acs.org.

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