

An oxygen-bridged bis(sila[1]ferrocenophane),
[Fe(η -C₅H₄)₂Si(Me)]₂OAndrea Berenbaum, Alan J.
Lough* and Ian MannersDepartment of Chemistry, University of Toronto,
Toronto, Ontario, Canada M5S 3H6Correspondence e-mail:
alough@chem.utoronto.ca

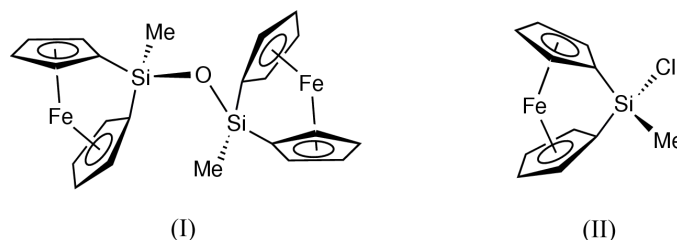
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ (C–C) = 0.004 Å
R factor = 0.029
wR factor = 0.069
Data-to-parameter ratio = 17.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, [Fe(C₅H₄)₂(CH₃Si)]₂O, the cyclopentadienyl rings in both ferrocenophane moieties are tilted towards the bridging Si atoms, with tilt angles of 19.7 (2) and 20.7 (2)°; the central Si—O—Si angle is 143.25 (12)°.

Comment

The title compound was isolated in trace amounts as an impurity from aged samples of the silicon-bridged [1]ferrocenophane, (II), (Zechel *et al.*, 1996), which possesses a hydrolytically sensitive Si—Cl moiety. The bis(sila[1]ferrocenophane), (I), is of potential utility as a crosslinking agent in the polymerization of other sila[1]ferrocenophanes (MacLachlan *et al.*, 1996, 2000; Kulbaba *et al.*, 2001; Kulbaba & Manners, 2001); however, attempts to produce it on a large scale have been unsuccessful to date.



In (I), the cyclopentadienyl (Cp) rings of both of the ferrocenophane groups are tilted towards the bridging Si atoms. The angle between the least-squares planes of the Cp rings bonded to Fe1 is 19.7 (2)°, and the angle between the Cp rings bonded to Fe2 is 20.7 (2)°. The tilt of the Cp rings is also reflected in the variation of the Fe—C distances in each of the ferrocene groups. The Fe—C distances in the ferrocene group containing Fe1 range from 2.010 (2) to 2.082 (3) Å, and in the ferrocene group containing Fe2, the Fe—C distances range from 2.009 (3) to 2.087 (3) Å. The shortest Fe—C distances are for the C atoms which are also bonded to the bridging Si atoms, namely C1, C6, C11 and C16 (see Table 1). The overall conformation of the molecule is twisted, such that the ferrocene groups are rotated about the Si—O bonds so that they are approximately perpendicular to each other (see Fig. 1). The degree of rotation can be described by a torsion angle calculated using the non-bonded atoms Fe1···Si1···Si2···Fe2, which gives a value of 75.07 (5)°. The Si1—O1—Si2 angle is 143.25 (12)°.

Experimental

Crystals of (I) were obtained at 243 K in trace amounts from the hexanes-soluble fraction of a reaction mixture which initially contained (II). No other hexanes-soluble products were formed in

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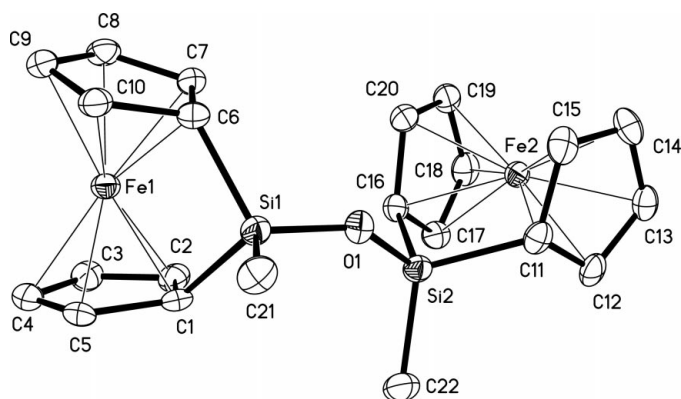


Figure 1
View of molecule (I), with displacement ellipsoids drawn at the 50% probability level.

this reaction, and an investigation of the sample of (II) utilized in the synthesis revealed the presence of trace (< 5%) amounts of (I). For (I): ^1H NMR (400 MHz, C_6D_6 , 298 K): $\delta = 4.46\text{--}4.44$ (*m*, 4H, Cp), 4.42–4.39 (*m*, 8H, Cp), 3.87–3.86 (*m*, 4H, Cp), 0.58 (*s*, 6H, Me); $^{13}\text{C}\{^1\text{H}\}$ NMR (100.4 MHz, C_6D_6 , 298 K): $\delta = 78.4, 77.8, 75.9, 74.5$ (Cp), 38.7 (*ipso*-Cp), -0.6 (Me); $^{29}\text{Si}\{^1\text{H}\}$ NMR (79.3 MHz, C_6D_6 , 298 K): $\delta = -13.4$.

Crystal data

$[\text{Fe}(\text{C}_5\text{H}_4)_2(\text{CH}_3\text{Si})_2\text{O}]$
 $M_r = 470.28$
Orthorhombic, $P2_12_12_1$
 $a = 7.5203$ (1) Å
 $b = 11.4213$ (3) Å
 $c = 22.2986$ (6) Å
 $V = 1915.26$ (8) Å³
 $Z = 4$
 $D_x = 1.631$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 19591 reflections
 $\theta = 2.9\text{--}27.5^\circ$
 $\mu = 1.65$ mm⁻¹
 $T = 100$ (1) K
Plate, orange
 $0.35 \times 0.32 \times 0.10$ mm

Data collection

Nonius Kappa-CCD diffractometer
 φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)
 $T_{\min} = 0.596, T_{\max} = 0.853$
19591 measured reflections

4381 independent reflections
3810 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.075$
 $\theta_{\max} = 27.5^\circ$
 $h = -9 \rightarrow 9$
 $k = -14 \rightarrow 14$
 $l = -28 \rightarrow 28$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.069$
 $S = 1.00$
4381 reflections
246 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.48$ e Å⁻³
Absolute structure: (Flack, 1983),
1871 Friedel pairs
Flack parameter = -0.009 (13)

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-----------------|-------------|-------------|-------------|
| Fe1—C1 | 2.010 (2) | Fe2—C15 | 2.028 (3) |
| Fe1—C6 | 2.016 (2) | Fe2—C17 | 2.030 (2) |
| Fe1—C2 | 2.025 (3) | Fe2—C14 | 2.079 (3) |
| Fe1—C7 | 2.032 (3) | Fe2—C13 | 2.080 (3) |
| Fe1—C5 | 2.032 (3) | Fe2—C18 | 2.080 (3) |
| Fe1—C10 | 2.037 (3) | Fe2—C19 | 2.087 (3) |
| Fe1—C4 | 2.068 (3) | Si1—O1 | 1.6308 (18) |
| Fe1—C9 | 2.076 (3) | Si1—C21 | 1.851 (3) |
| Fe1—C8 | 2.076 (3) | Si1—C6 | 1.882 (3) |
| Fe1—C3 | 2.082 (3) | Si1—C1 | 1.885 (3) |
| Fe2—C11 | 2.009 (3) | Si2—O1 | 1.6377 (19) |
| Fe2—C16 | 2.020 (3) | Si2—C22 | 1.847 (3) |
| Fe2—C20 | 2.025 (3) | Si2—C16 | 1.871 (3) |
| Fe2—C12 | 2.027 (3) | Si2—C11 | 1.879 (3) |
| O1—Si1—C6 | 111.18 (11) | O1—Si2—C11 | 110.11 (11) |
| O1—Si1—C1 | 111.21 (11) | C16—Si2—C11 | 96.72 (11) |
| C6—Si1—C1 | 96.82 (11) | Si1—O1—Si2 | 143.25 (12) |
| O1—Si2—C16 | 110.51 (11) | | |
| Fe1—Si1—Si2—Fe2 | 75.07 (5) | | |

All H atoms were included in calculated positions, with distances of 1.00 Å (for Cp C—H) and 0.98 Å (for methyl C—H). In the refinement H atoms were included in riding-motion approximation, with $U_{\text{iso}} = 1.2U_{\text{eq}}$ (1.5 U_{eq} for methyl) of the carrier atom.

Data collection: *COLLECT* (Nonius 1997–2001); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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