Monoclinic
$P2_1/n$
$a = 5.7790$ (10) Å
$b = 5.300$ (3) Å
$c = 35.811$ (5) Å
$\beta = 95.06$ (2)°
$V = 1092.5$ (7) Å^3
$Z = 4$
$D_x = 1.507$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 6$–13°
$\mu = 0.379$ mm^{-1}
$T = 293$ (2) K
Transparent plates
0.50 × 0.42 × 0.13 mm
Colourless
Crystal source: slow evaporation of aqueous solution at 295 K

Data collection

Enraf–Nonius CAD-4 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
1955 measured reflections
1923 independent reflections
1566 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0127$

$\theta_{\rm max} = 24.98$°
$h = -6 \rightarrow 6$
$k = 0 \rightarrow 6$
$l = 0 \rightarrow 42$
3 standard reflections monitored every 100 reflections
frequency: 180 min
intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0310$
$wR(F^2) = 0.0903$
$S = 1.180$
1920 reflections
172 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.4528P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.016$
$\Delta\rho_{\rm max} = 0.192$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.241$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Mg1	0	1/2	0	0.0254 (2)
Cl1	0.15111 (10)	0.00444 (11)	0.21921 (2)	0.0523 (2)
O1	−0.7609 (2)	1.0442 (3)	0.08599 (4)	0.0357 (3)
O2	−0.4738 (2)	1.2931 (3)	0.07147 (4)	0.0403 (4)
O3	0.2729 (2)	0.6395 (3)	0.03617 (4)	0.0338 (3)
O4	−0.0837 (2)	0.2423 (2)	0.03976 (4)	0.0309 (3)
O5	0.2375 (3)	0.2373 (3)	−0.01637 (4)	0.0388 (4)
C1	−0.5496 (3)	1.1040 (3)	0.08800 (5)	0.0284 (4)
C2	−0.3785 (3)	0.9429 (4)	0.11007 (5)	0.0327 (4)
C3	−0.4392 (3)	0.7761 (4)	0.13469 (5)	0.0342 (4)
C4	−0.2863 (3)	0.5975 (4)	0.15603 (5)	0.0332 (4)
C5	−0.3527 (3)	0.4943 (4)	0.18909 (6)	0.0402 (5)
C6	−0.2179 (4)	0.3156 (4)	0.20900 (6)	0.0427 (5)
C7	−0.0160 (3)	0.2370 (4)	0.19526 (5)	0.0371 (5)
C8	0.0562 (4)	0.3349 (4)	0.16263 (6)	0.0399 (5)
C9	−0.0794 (3)	0.5144 (4)	0.14326 (5)	0.0375 (5)

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

Mg1—O4$^{\rm i}$	2.0608 (14)	C1—C2	1.481 (3)
Mg1—O4	2.0610 (14)	C2—C3	1.317 (3)
Mg1—O5	2.0755 (15)	C3—C4	1.464 (3)
Mg1—O5$^{\rm i}$	2.0756 (15)	O3···O2$^{\rm ii}$	2.605 (2)
Mg1—O3	2.0860 (13)	O3···O1$^{\rm iii}$	2.808 (2)
Mg1—O3$^{\rm i}$	2.0860 (13)	O4···O2$^{\rm iv}$	2.626 (2)
Cl1—C7	1.744 (2)	O4···O1$^{\rm ii}$	2.604 (2)
O1—C1	1.257 (2)	O5···O4$^{\rm v}$	2.796 (2)
O2—C1	1.262 (2)		
O4—Mg1—O5	87.25 (7)	C3—C2—C1	122.6 (2)
O4—Mg1—O3	90.96 (6)	C2—C3—C4	126.8 (2)
O5—Mg1—O3	86.01 (6)	C5—C4—C3	120.0 (2)
O1—C1—O2	123.3 (2)	C9—C4—C3	122.1 (2)
O1—C1—C2	119.0 (2)	C6—C7—Cl1	119.4 (2)
O2—C1—C2	117.7 (2)	C8—C7—Cl1	119.2 (2)

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1+x, y-1, z$; (iii) $1+x, y, z$; (iv) $x, y-1, z$; (v) $-x, -y, -z$.

All non-H atoms were assigned anisotropic displacement parameters. The positions of the anion H atoms were fixed geometrically and the difference Fourier synthesis map revealed the locations of the water H atoms. Data collection and cell refinement: Enraf–Nonius CAD-4 software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1992). Molecular graphics: *DTMM* (Crabbe & Appleyard, 1991). Software used to prepare material for publication: *SHELXL*92.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1117). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). **C50**, 1667–1669

A Linear Oligo(ferrocenylsilane)

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(*Received* 25 *April* 1994; *accepted* 7 *June* 1994)

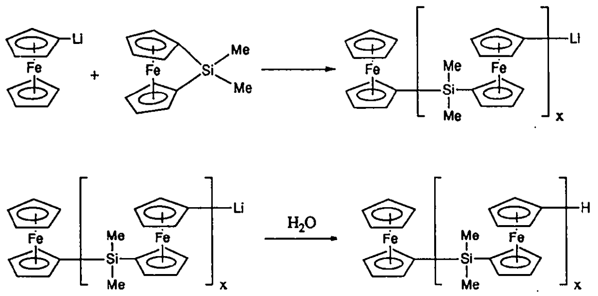
Abstract

The title compound 1,1′-bis(ferrocenyldimethylsilyl)-ferrocene, $[\{C_5H_5\text{-Fe-}C_5H_4\text{-Si(Me}_2)\text{-}C_5H_4\}_2Fe]$ {or bis-[1,3(η^5)-cyclopentadienyl][μ-1(η^5):2(η^5)-dimethylsilanediyldicyclopentadienyl][μ-2(η^5):3(η^5)-dimethylsilanedi-

yldicyclopentadienyl]triiron, $[Fe_3(C_5H_5)_2(C_{12}H_{14}Si)_2]\}$- is an oligomer containing three ferrocene units. The intramolecular Fe···Fe distance is 6.188 (1) Å.

Comment

Polymers with transition metal atoms in the main chain are of considerable current interest (Sheats, Carraher & Pittman, 1985; Manners, 1994). We have previously shown that silicon-bridged [1]ferrocenophanes undergo ring-opening polymerization to yield high molecular weight poly(ferrocenylsilanes) which possess an unusual polymer backbone consisting of ferrocene units and Si atoms (Foucher, Tang & Manners, 1992). Short-chain oligomers are of interest as models of the high polymers with respect to structure and physical properties. We report here the structure of the linear oligo(ferrocenylsilane) (I) with three ferrocenyl groups.



$x = 1–5$

(I) $x = 2$

The molecule (Fig. 1) lies on a crystallographic inversion centre which is at the site of Fe2. The central ferrocene unit is almost perpendicular to the two outer ferrocene groups. This geometry can be described by the angle between the least-squares planes containing C1–C5 and C11–C15, which is 84.32 (16)°. The conformation of the title compound is similar to that of the five-ferrocene unit oligomer which was recently characterized by X-ray crystallography (Rulkens, Lough & Manners, 1994).

The intramolecular Fe1···Fe2 distance, which is important for modelling high polymers, is 6.188 (1) Å in the title compound. As might be expected, the terminal unsubstituted cyclopentadienyl rings are affected more by thermal displacement than the rings to which the Si atoms are bonded (see Fig. 1).

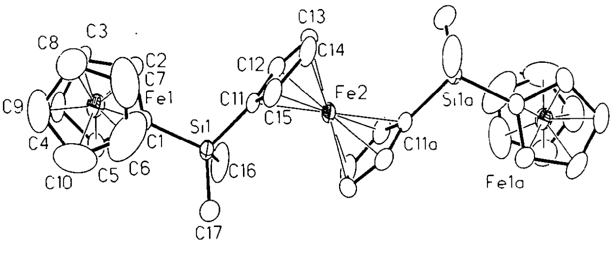


Fig. 1. View of the molecule with the crystallographic numbering scheme; displacement ellipsoids are drawn at the 25% probability level. H atoms have been omitted for clarity.

Experimental

We recently reported the synthesis of a range of oligo(ferrocenylsilanes) (Rulkens, Lough & Manners, 1994) (see scheme above). The title compound was separated from the mixture of oligomeric products by column chromatography. Suitable crystals were obtained from a solution of hexanes.

Crystal data

$[Fe(C_{17}H_{19}FeSi)_2]$
$M_r = 670.37$
Monoclinic
$P2_1/c$
$a = 10.071$ (1) Å
$b = 14.937$ (1) Å
$c = 11.173$ (1) Å
$\beta = 115.03$ (1)°
$V = 1522.9$ (2) Å^3
$Z = 2$
$D_x = 1.462$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 6.5–18.2°$
$\mu = 1.511$ mm^{-1}
$T = 293$ (2) K
Block
0.3 × 0.2 × 0.2 mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: semi-empirical
$T_{min} = 0.705$, $T_{max} = 0.941$
3492 measured reflections
3308 independent reflections
2142 observed reflections $[I > 2\sigma(I)]$

$R_{int} = 0.0295$
$\theta_{max} = 26.99°$
$h = -12 \rightarrow 0$
$k = 0 \rightarrow 19$
$l = -12 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity variation: 12% (linear correction)

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0431$
$wR(F^2) = 0.0990$
$S = 1.084$
3304 reflections
197 parameters
H atoms refined as riding, C—H 0.96 Å
$w = 1/[\sigma^2(F_o^2) + (0.0400P)^2 + 1.6454P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.008$
$\Delta\rho_{max} = 0.363$ e Å^{-3}
$\Delta\rho_{min} = -0.321$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Fe1	0.86355 (6)	0.89113 (4)	0.13348 (6)	0.0545 (2)
Fe2	3/2	1	1/2	0.0566 (2)
Si1	1.23016 (12)	0.83955 (8)	0.34691 (11)	0.0629 (3)
C1	1.0637 (4)	0.8307 (2)	0.1885 (4)	0.0493 (9)
C2	1.0306 (4)	0.8863 (3)	0.0760 (4)	0.0546 (10)

C3	0.8968 (5)	0.8568 (4)	−0.0274 (4)	0.0715 (13)
C4	0.8479 (4)	0.7830 (3)	0.0185 (5)	0.0747 (14)
C5	0.9482 (4)	0.7661 (3)	0.1504 (5)	0.0648 (12)
C6	0.8616 (13)	0.9594 (12)	0.2881 (10)	0.188 (7)
C7	0.8493 (10)	1.0156 (6)	0.1968 (12)	0.149 (4)
C8	0.7270 (8)	0.9981 (5)	0.0837 (7)	0.096 (2)
C9	0.6599 (6)	0.9236 (6)	0.1079 (8)	0.113 (2)
C10	0.7464 (12)	0.8997 (7)	0.2423 (12)	0.146 (4)
C11	1.3132 (3)	0.9501 (2)	0.3513 (3)	0.0445 (8)
C12	1.4196 (5)	0.9704 (4)	0.3038 (4)	0.0744 (14)
C13	1.4554 (7)	1.0610 (5)	0.3250 (6)	0.127 (3)
C14	1.3731 (8)	1.0989 (4)	0.3807 (7)	0.123 (3)
C15	1.2852 (5)	1.0329 (3)	0.3990 (4)	0.0720 (13)
C16	1.3637 (7)	0.7520 (4)	0.3495 (9)	0.146 (4)
C17	1.1861 (9)	0.8235 (7)	0.4892 (5)	0.163 (4)

Table 2. *Selected geometric parameters* (Å, °)

Fe1—C9	2.007 (5)	Fe2—C13	2.029 (6)
Fe1—C6	2.013 (8)	Fe2—C15	2.032 (4)
Fe1—C7	2.016 (7)	Fe2—C14	2.038 (5)
Fe1—C10	2.025 (6)	Fe2—C12	2.038 (4)
Fe1—C8	2.026 (5)	Fe2—C11	2.052 (3)
Fe1—C5	2.028 (4)	Si1—C17	1.838 (6)
Fe1—C4	2.028 (4)	Si1—C11	1.843 (4)
Fe1—C3	2.029 (4)	Si1—C1	1.858 (3)
Fe1—C2	2.040 (4)	Si1—C16	1.867 (6)
Fe1—C1	2.051 (4)		
C17—Si1—C11	111.5 (3)	C5—C1—Si1	128.6 (3)
C17—Si1—C1	111.4 (2)	Si1—C1—Fe1	129.1 (2)
C11—Si1—C1	107.8 (2)	C12—C11—Si1	126.1 (3)
C17—Si1—C16	109.3 (5)	C15—C11—Si1	129.1 (3)
C11—Si1—C16	108.2 (3)	C12—C11—Fe2	69.2 (2)
C1—Si1—C16	108.6 (2)	C15—C11—Fe2	68.9 (2)
C2—C1—Si1	125.4 (3)	Si1—C11—Fe2	126.7 (2)

Data collection and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *XCAD*4 (Siemens, 1993). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93.

This research was supported by NSERC Canada and the University of Toronto.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: FG1011). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). **C50**, 1669–1671

Tricarbonyl(η^6-pyridine)chromium(0)

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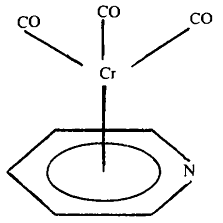
(*Received* 23 *February* 1994; *accepted* 13 *June* 1994)

Abstract

The molecular structure of tricarbonyl(η^6-pyridine)chromium(0), [Cr(CO)$_3$(C$_5$H$_5$N)], is presented here. The Cr atom is bound to the pyridine ring *via* the π-electron system and is situated directly below the heteroarene ring centre. The pyridine ring is slightly distorted compared to free pyridine and the distance from the Cr atom to the plane of the pyridine ring is 1.698 (2) Å. The tricarbonyl moiety adopts an eclipsed conformation with respect to the pyridine ring atoms.

Comment

The title compound, (I), consists of an unsubstituted pyridine ring with a tricarbonylchromium unit bonded to the π-electron system of the heterocycle.



(I)

This compound is analogous to the [Cr(CO)$_3$(η^6-C$_6$H$_6$)] compound in which the π-bonded benzene ring exhibits no significant distortion from the sixfold D_{6h} symmetry of free benzene (Bailey & Dahl, 1965). No such symmetry exists for pyridine, however, as is evident from the results calculated from microwave measurements by Bak, Hansen & Rastrup-Andersen (1954), which indicate that the internal angles in the heteroaromatic ring vary from 116.7 (1)° at the N atom to