as having localized C—N(imino) double bonds and two C—C double bonds. Molecular orbital calculations made with the CNDO program (kindly provided by Professor P. G. Perkins) support this view, giving average bond indices (Armstrong, Perkins & Stewart, 1973) of 1·65 for the C—N and C—C double bonds, 1·23 for the C—C single bond and 1·14 for the C—N(amino) single bond.

Intramolecular hydrogen bonding does not appear to be an important factor in determining the orientation of the two rings; O—H(4) and H(7)—N(2), the two pertinent contacts, are 2·40 and 2·19 Å respectively. The asymmetric disposition of the rings adopted about the C=O bond would appear to alleviate an otherwise unfavourable lone-pair repulsion between N(2) and N(4).

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Ferrocenyldiphenylsilane

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Abstract. $C_{22}H_{20}FeSi$, $M_r = 368·34$; monoclinic; $P2_1/c$, $a = 10·797(3)$, $b = 24·639(7)$, $c = 7·707(3)$ Å, $\beta = 116·95(2)°$; $Z = 4$, $D_m = 1·33(1)$, $D_c = 1·34$ g cm^{-3}, $U = 1828(1)$ Å^3, μ(Mo $K\alpha$) $= 9·1$ cm^{-1}. The two planar cyclopentadienyl rings in the ferrocenyl moiety are tilted 2·7° from parallel and twisted approximately 13·3° from the fully eclipsed configuration. The distance between the cyclopentadienyl ring centroids is 3·28 Å. The mean silicon–phenyl carbon bond length is 1·871(4) Å; the silicon–cyclopentadienyl carbon bond length is 1·856(4) Å.

Introduction. Crystals of the title compound were prepared by the method reported for ferrocenyltriphenylsilane (Benkeser, Goggin & Schroll, 1954). A crystal, $0·22 \times 0·15 \times 0·12$ mm, was mounted on a Syntex $P2_1$ diffractometer and data were collected to a 2θ of 55° with Mo $K\alpha$ ($\lambda = 0·71069$ Å) radiation

(graphite monochromator) and the θ–2θ scan technique at 2° min^{-1}. Backgrounds were measured at each end of the scan (2θ scan range: 2θMo $K\alpha_1 - 1·0°$ to 2θMo $K\alpha_2 + 1·0°$) for a total time equal to one-half the scan time. Systematic absences were $h0l$, l odd; $0k0$, k odd, uniquely determining the space group $P2_1/c$. Fifteen reflections with 2θ between 7 and 24° were centered with a programmed centering routine; cell parameters were obtained by least-squares refinement of these angles. During data collection, the intensities of three standard reflections were measured every 97 reflections with no significant variation in intensity observed. The data were reduced to F^2 and $\sigma(F^2)$ by procedures similar to those described previously (Schmonsees, 1974). Standard deviations were assigned as follows: $\sigma(I) = [\sigma_{counter}(I)^2 + (0·03I)^2]^{1/2}$, where $\sigma_{counter} = (I + K^2B)^{1/2}$, $I =$ net intensity, $B =$ total background count, and $K =$ ratio of scan time to background time. No corrections for absorption were made. The 2160 data with $F_o^2 > 3\sigma(F_o^2)$ from 4632 data

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Table 1. *Final positional* (×10⁴) *and anisotropic thermal* (×10⁴) *parameters with estimated standard deviations in parentheses*

Anisotropic thermal parameters are in the form: $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Fe(1)	6381 (1)	3592·0 (2)	6652 (1)	99 (1)	13·2 (1)	148 (1)	4·5 (3)	58 (1)	7·1 (4)
Si(2)	3490 (1)	3697·2 (5)	2081 (2)	101 (2)	14·2 (2)	163 (3)	1·2 (5)	53 (2)	0 (1)
C(3)	2283 (4)	3511 (2)	3101 (6)	97 (5)	15 (1)	163 (10)	−1 (2)	38 (6)	9 (2)
C(4)	1729 (5)	2985 (2)	2846 (6)	134 (7)	18 (1)	200 (12)	−3 (2)	57 (8)	1 (3)
C(5)	808 (6)	2840 (2)	3555 (8)	158 (8)	25 (1)	261 (15)	−21 (3)	73 (9)	10 (4)
C(6)	430 (5)	3211 (2)	4575 (7)	134 (8)	29 (1)	240 (14)	−5 (3)	90 (9)	22 (4)
C(7)	987 (5)	3728 (2)	4891 (7)	132 (7)	26 (1)	241 (13)	10 (2)	89 (8)	11 (3)
C(8)	1878 (5)	3881 (2)	4143 (7)	133 (7)	19 (1)	250 (13)	−1 (2)	103 (8)	−1 (3)
C(9)	2697 (4)	4256 (2)	238 (5)	84 (5)	14 (1)	143 (10)	−3 (2)	33 (6)	−3 (2)
C(10)	3487 (4)	4516 (2)	−535 (6)	86 (6)	21 (1)	202 (11)	0 (2)	45 (7)	8 (3)
C(11)	2956 (5)	4930 (2)	−1874 (7)	113 (6)	22 (1)	206 (12)	−6 (2)	58 (8)	13 (3)
C(12)	1608 (5)	5103 (2)	−2471 (7)	131 (7)	20 (1)	190 (12)	0 (2)	35 (8)	16 (3)
C(13)	793 (4)	4856 (2)	−1742 (7)	99 (6)	19 (1)	231 (12)	6 (2)	52 (7)	8 (3)
C(14)	1333 (4)	4436 (2)	−402 (6)	102 (6)	18 (1)	182 (11)	2 (2)	54 (7)	5 (3)
C(15)	5220 (4)	3926 (2)	3953 (6)	98 (6)	14 (1)	157 (10)	−1 (2)	49 (6)	12 (2)
C(16)	6542 (5)	3747 (2)	4174 (6)	109 (6)	22 (1)	173 (10)	4 (2)	77 (7)	14 (3)
C(17)	7605 (5)	4024 (2)	5784 (7)	96 (6)	27 (1)	209 (12)	−8 (2)	62 (7)	20 (3)
C(18)	6962 (5)	4377 (2)	6580 (7)	121 (7)	15 (1)	217 (12)	−8 (2)	35 (8)	6 (3)
C(19)	5496 (5)	4318 (2)	5465 (6)	129 (7)	12 (1)	218 (12)	0 (2)	64 (8)	6 (3)
C(20)	6767 (10)	3446 (2)	9428 (8)	323 (14)	26 (2)	194 (14)	−4 (4)	156 (13)	9 (3)
C(21)	7666 (6)	3144 (3)	8969 (9)	146 (8)	29 (1)	260 (17)	8 (3)	24 (10)	47 (4)
C(22)	6896 (8)	2805 (2)	7416 (9)	260 (12)	16 (1)	286 (16)	25 (3)	149 (12)	24 (3)
C(23)	5497 (7)	2885 (2)	6914 (9)	179 (10)	20 (1)	335 (18)	−16 (3)	64 (11)	39 (4)
C(24)	5414 (8)	3275 (3)	8139 (12)	251 (13)	28 (2)	520 (26)	30 (4)	290 (16)	67 (5)

Table 2. *Final hydrogen atom positional* (×10³) *and thermal parameters*

The hydrogen atom bonded to Si(2) was included in the least-squares refinement. All other hydrogen atoms were included at calculated positions with C–H distances of 1·0 Å and fixed thermal parameters 10% greater than the equivalent B of the atom bonded to the hydrogen atom.

	x	y	z	B (Å²)
H(Si2)	373 (3)	325 (1)	125 (5)	0·9 (8)
H(C4)	201	271	214	5·0
H(C5)	41	247	333	5·9
H(C6)	−24	311	508	5·6
H(C7)	74	399	567	5·5
H(C8)	224	426	434	5·0
H(C10)	447	440	−10	4·5
H(C11)	354	510	−241	4·8
H(C12)	122	541	−343	5·0
H(C13)	−18	498	−218	4·8
H(C14)	73	426	11	4·3
H(C16)	670	347	334	4·3
H(C17)	863	398	627	4·7
H(C18)	745	462	772	4·7
H(C19)	478	452	570	4·3
H(C20)	703	373	1047	6·5
H(C21)	870	317	965	6·0
H(C22)	728	255	677	5·8
H(C23)	469	269	585	5·8
H(C24)	454	341	812	6·3

scanned were used in the structure solution and refinement.

The structure was solved by the heavy-atom method. The Fe and Si atoms were located from a Patterson map, and the remaining non-hydrogen atoms were found from subsequent Fourier maps. Least-squares refinement (Busing, Martin & Levy, 1962) of the non-hydrogen atoms with isotropic thermal parameters gave a discrepancy value R_1 (= $\Sigma||F_o| - |F_c||/\Sigma|F_o|$) of 0·087. The H bonded to Si was located from a difference map and included in the refinement with an isotropic thermal parameter. Refinement was continued with anisotropic thermal parameters for the non-hydrogen atoms; the H atoms bonded to C were included in the calculations at ideal locations (Zalkin, 1974) with fixed H distances of 1·0 Å and isotropic thermal parameters 10% larger than the equivalent isotropic B of the atom to which they are bonded. Tables 1 and 2 give final positional and thermal parameters with associated estimated standard deviations. Final discrepancy values were $R_1 = 0·041$ and $R_2 = [\Sigma(w|F_o| - |F_c|)^2/\Sigma wF_o^2]^{1/2} = 0·041$. The largest parameter shift in the final cycle of full-matrix refinement was less than 5% of its standard deviation; the error of fit was 1·39. Atomic scattering factors and real and imaginary anomalous dispersion corrections for Fe and Si were taken from *International Tables for X-ray Crystallography* (1974). The highest residual electron density in the final difference map was 0·28 e Å⁻³.*

Discussion. Ferrocenyldiphenylsilane is one of several compounds considered as possible precursors of the silicenium ion (Corey, Gust & Mislow, 1975; Bickart, Llort & Mislow, 1976).

The molecule and the numbering scheme are shown in Fig. 1 (Johnson, 1965). Table 3 gives the values of the interatomic bond lengths and angles (Busing, Martin & Levy, 1964). The title compound adopts a nearly regular tetrahedral geometry about Si. A stereoscopic view of the atoms (excluding H atoms bonded to C) in the asymmetric unit is shown in Fig. 2.

The Si—H bond length of 1·36 (3) Å is outside the range of 1·46–1·54 (5) Å reported for a variety of silanes in the gas phase (Ondik & Smith, 1962), and is less than the value of 1·65 (15) Å obtained by X-ray methods for [(C$_5$H$_5$)TiSiH$_2$]$_2$ (Hencken & Weiss, 1973). The C(cyclopentadienyl ring)—Si distance of 1·856 (3) Å is in good agreement with the mean value

Fig. 1. Numbering of atoms within the ferrocenyldiphenylsilane molecule.

Table 3. *Bond lengths* (Å) *and angles* (°)

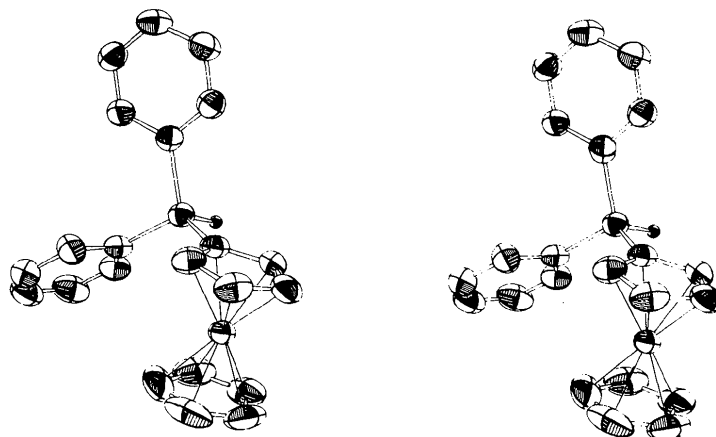|			
Fe(1)—C(15)	2·052 (4)	C(3)—Si(2)—C(9)	109·4 (2)
Fe(1)—C(16)	2·031 (4)	C(3)—Si(2)—C(15)	113·4 (2)
Fe(1)—C(17)	2·032 (4)	C(3)—Si(2)—H(Si2)	109 (1)
Fe(1)—C(18)	2·043 (4)	C(9)—Si(2)—C(15)	108·6 (2)
Fe(1)—C(19)	2·038 (4)	C(9)—Si(2)—H(Si2)	111 (1)
Fe(1)—C(20)	2·018 (5)	C(15)—Si(2)—H(Si2)	106 (1)
Fe(1)—C(21)	2·021 (5)	Si(2)—C(3)—C(4)	120·3 (3)
Fe(1)—C(22)	2·031 (5)	Si(2)—C(3)—C(8)	122·6 (3)
Fe(1)—C(23)	2·040 (5)	C(4)—C(3)—C(8)	117·1 (4)
Fe(1)—C(24)	2·025 (5)	C(5)—C(4)—C(3)	121·3 (5)
Si(2)—C(3)	1·860 (4)	C(6)—C(5)—C(4)	120·5 (5)
Si(2)—C(9)	1·882 (4)	C(7)—C(6)—C(5)	119·5 (5)
Si(2)—C(15)	1·856 (4)	C(8)—C(7)—C(6)	120·5 (5)
Si(2)—H(Si2)	1·36 (3)	C(3)—C(8)—C(7)	121·2 (4)
C(3)—C(4)	1·404 (4)	Si(2)—C(9)—C(10)	120·0 (3)
C(4)—C(5)	1·381 (6)	Si(2)—C(9)—C(14)	123·1 (3)
C(5)—C(6)	1·381 (7)	C(10)—C(9)—C(14)	116·9 (4)
C(6)—C(7)	1·384 (7)	C(11)—C(10)—C(9)	122·2 (4)
C(7)—C(8)	1·379 (6)	C(12)—C(11)—C(10)	119·8 (4)
C(8)—C(3)	1·408 (6)	C(13)—C(12)—C(11)	119·8 (4)
C(9)—C(10)	1·399 (6)	C(14)—C(13)—C(12)	120·0 (4)
C(10)—C(11)	1·376 (6)	C(9)—C(14)—C(13)	121·3 (4)
C(11)—C(12)	1·381 (6)	Si(2)—C(15)—C(16)	126·7 (3)
C(12)—C(13)	1·381 (6)	Si(2)—C(15)—C(19)	126·9 (3)
C(13)—C(14)	1·390 (6)	C(16)—C(15)—C(19)	106·4 (4)
C(14)—C(9)	1·397 (5)	C(15)—C(16)—C(17)	108·8 (4)
C(15)—C(16)	1·429 (6)	C(16)—C(17)—C(18)	108·2 (4)
C(16)—C(17)	1·425 (6)	C(17)—C(18)—C(19)	107·7 (4)
C(17)—C(18)	1·414 (6)	C(18)—C(19)—C(15)	108·9 (4)
C(18)—C(19)	1·426 (6)	C(24)—C(20)—C(21)	106·3 (5)
C(19)—C(15)	1·435 (6)	C(20)—C(21)—C(22)	109·1 (5)
C(20)—C(21)	1·390 (8)	C(21)—C(22)—C(23)	107·7 (5)
C(21)—C(22)	1·387 (7)	C(22)—C(23)—C(24)	107·9 (5)
C(22)—C(23)	1·393 (7)	C(23)—C(24)—C(20)	108·9 (6)
C(23)—C(24)	1·378 (8)		
C(24)—C(20)	1·408 (9)		

Fig. 2. A stereoscopic view of ferrocenyldiphenylsilane. The atoms are represented by 50% probability thermal ellipsoids.
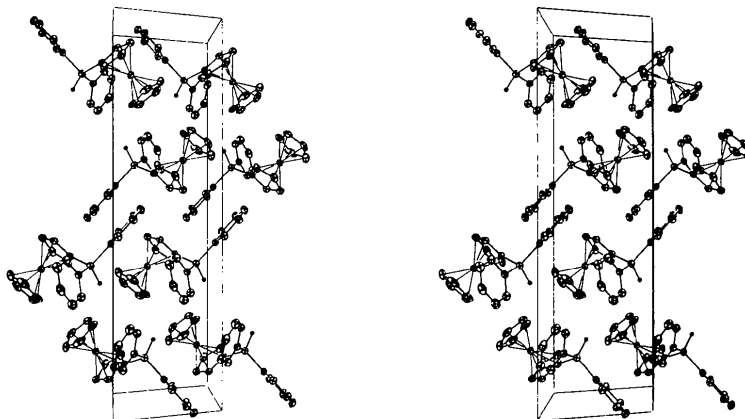
Fig. 3. A stereoscopic view of the unit-cell contents, excluding hydrogen atoms bonded to carbon, viewed down the a axis.

of 1·86 (3) Å reported for 1,1'-bis(pentamethyl-disilanyl)ferrocene (Hirotsu, Higuchi & Shimada, 1968).

The observed Fe—C distances and dihedral angle (2·7°) between the two cyclopentadienyl rings agree well with the values reported for non-bridged, substituted ferrocenes (Krukonis, Silverman & Yannoni, 1972 and references therein). The displacements of the Fe atom from the least-squares planes of the C atoms in the cyclopentadienyl groups are 1·64 and 1·65 Å for the Si substituted and unsubstituted rings respectively. The relative rotational orientation of the cyclopentadienyl rings was defined as the dihedral angle between a ring C, the two ring centroids, and the C atom on the opposite ring. This twist angle ranged from 12·9 to 13·9° with an average of 13·3° (0° = eclipsed, 36° = staggered) and is within the range of 0–17° reported for the majority of substituted, non-bridged ferrocenes (Churchill & Wormald, 1969; Krukonis, Silverman & Yannoni, 1972).

The packing of the molecules of the title compound in the crystal is shown in a stereoscopic drawing, Fig. 3. There are no intermolecular contacts less than van der Waals distances. The closest non-hydrogen intermolecular distance is 3·505 (8) Å between C(19) and C(19) related by the center of symmetry at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$.

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