N1	0.3395 (8)	0.5473 (3)	0.1086 (5)	3.5 (3)
N2	0.5122 (8)	0.4933 (4)	0.2972 (5)	4.3 (3)
N3	0.5748 (8)	0.6454 (3)	0.0969 (5)	3.4 (3)
N4	0.7448 (8)	0.5933 (3)	0.2768 (5)	3.4 (3)
C1	0.3367 (9)	0.5765 (4)	0.0144 (6)	3.4 (3)
C2	0.6530 (11)	0.4792 (5)	0.3552 (6)	4.5 (4)
C3	0.4677 (10)	0.6365 (4)	−0.0017 (6)	3.5 (3)
C4	0.7927 (10)	0.5344 (5)	0.3554 (6)	4.4 (4)
C5	0.2096 (11)	0.5487 (6)	−0.0712 (7)	5.2 (5)
C6	0.6754 (13)	0.4069 (6)	0.4134 (9)	6.3 (5)
C7	0.3715 (13)	0.7070 (5)	−0.0354 (8)	5.3 (4)
C10	0.8162 (15)	0.5732 (7)	0.4687 (7)	6.9 (6)
C11	0.7413 (11)	0.6850 (5)	0.0792 (7)	4.6 (4)
C12	0.8977 (10)	0.6356 (5)	0.2408 (7)	4.4 (4)
C13	0.8403 (10)	0.7041 (5)	0.1821 (7)	4.1 (4)
C14	0.9989 (12)	0.7578 (5)	0.1671 (9)	5.6 (5)
C15	0.7507 (12)	0.7614 (5)	0.2547 (8)	5.0 (4)
C16	0.9218 (14)	0.8081 (7)	0.2519 (12)	8.1 (7)

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

Tc—O3	1.664 (5)	C1—C3	1.479 (11)
Tc—N1	2.075 (6)	C1—C5	1.504 (10)
Tc—N2	2.087 (7)	C2—C4	1.446 (13)
Tc—N3	1.917 (6)	C2—C6	1.512 (12)
Tc—N4	1.917 (6)	C3—C7	1.528 (11)
O1—N1	1.353 (8)	C4—C10	1.588 (13)
O2—N2	1.330 (9)	C11—C13	1.511 (12)
N1—C1	1.290 (10)	C12—C13	1.507 (12)
N2—C2	1.290 (10)	C13—C14	1.545 (12)
N3—C3	1.465 (9)	C13—C15	1.539 (12)
N3—C11	1.452 (10)	C14—C16	1.518 (16)
N4—C4	1.493 (10)	C15—C16	1.531 (14)
N4—C12	1.449 (10)		
O3—Tc—N1	109.4 (2)	N1—C1—C3	115.2 (6)
O3—Tc—N2	108.3 (3)	C3—C1—C5	124.1 (7)
O3—Tc—N3	111.3 (3)	N2—C2—C4	116.0 (7)
O3—Tc—N4	111.3 (3)	N2—C2—C6	121.8 (8)
N1—Tc—N2	86.1 (3)	C4—C2—C6	122.1 (7)
N1—Tc—N3	77.7 (2)	N3—C3—C1	108.6 (6)
N1—Tc—N4	139.2 (2)	N3—C3—C7	112.6 (7)
N2—Tc—N3	140.2 (3)	C1—C3—C7	110.7 (6)
N2—Tc—N4	78.2 (3)	N4—C4—C2	109.6 (6)
N3—Tc—N4	90.8 (3)	N4—C4—C10	106.6 (7)
Tc—N1—O1	122.9 (4)	C2—C4—C10	112.4 (7)
Tc—N1—C1	117.5 (5)	N3—C11—C13	113.1 (7)
O1—N1—C1	119.2 (6)	N4—C12—C13	112.0 (6)
Tc—N2—O2	123.0 (5)	C11—C13—C12	110.7 (7)
Tc—N2—C2	116.7 (6)	C11—C13—C14	113.9 (7)
O2—N2—C2	120.2 (7)	C11—C13—C15	116.5 (7)
Tc—N3—C3	119.3 (5)	C12—C13—C14	111.9 (7)
Tc—N3—C11	127.8 (5)	C12—C13—C15	113.5 (7)
C3—N3—C11	112.5 (6)	C14—C13—C15	88.7 (6)
Tc—N4—C4	117.3 (5)	C13—C14—C16	90.2 (7)
Tc—N4—C12	128.4 (5)	C13—C15—C16	89.9 (7)
C4—N4—C12	113.9 (6)	C14—C16—C15	90.0 (7)
N1—C1—C5	120.7 (7)		

The structure was solved by Patterson methods and Fourier synthesis, and refined by full-matrix least squares. H atoms were located by a difference Fourier method and refined isotropically.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX*. Program(s) used to refine structure: *NRCVAX*. Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1986). Software used to prepare material for publication: *NRCVAX*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: KH1061). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[$Fe_2(CO)_4(PMe_3)_4(\mu\text{-}H)(\mu\text{-}SiCl_2)$]-[$Fe(CO)_4(SiCl_3)$]

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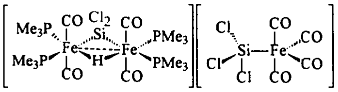
Abstract

The structure of tetracarbonyl-μ-(dichlorosilylene)-μ-hydrido-tetrakis(trimethylphosphine)diiron(1+) tetracarbonyl(trichlorosilyl)ferrate(1−), has been determined at 119 K. The cation has an Fe—Fe distance of 2.947 (2) Å and an average Fe—Si distance of

2.249 (3) Å. A bridging hydride was located from a difference map with Fe—H distances of 1.77 and 1.63 Å. The anion has an Fe—Si distance of 2.238 (3) Å.

Comment

In an attempt to find a one-step synthesis of phosphine-substituted iron–silicon complexes, the reaction of $Fe(CO)_5$, $HSiCl_3$ and PMe_3 in a 1:1:1 ratio was studied. Combination of these reagents resulted in a vigorous redistribution reaction and the known compound $SiCl_4(PMe_3)_2$ (Beattie & Ozin, 1969) precipitated. The mixture, which presumably contained the redistribution product SiH_2Cl_2, was photolysized. After removal of $SiCl_4(PMe_3)_2$ by filtration, the title complex, (I), was isolated from the solution. The formation of (I) can be viewed as the protonation of the neutral complex $Fe_2(CO)_4(PMe_3)_4(\mu_2\text{-}SiCl_2)$ by the strong Brönsted acid $HFe(CO)_4(SiCl_3)$ (Jetz & Graham, 1971). We report herein the crystal structure of (I).



(I)

Characterization of (I) by spectral methods proved difficult and an X-ray structure determination was undertaken to establish its identity. The crystal structure of the anion of (I) has been obtained previously (Olie, Oskam & Jansen, 1975) and our results are within experimental error of those of the previous determination. Fig. 1 shows a displacement ellipsoid plot of the cation of (1).

The cation contains two Fe atoms which are doubly bridged by an H atom and an $SiCl_2$ ligand. The two Fe atoms and the Si atom define a plane from which H(1) deviates by only 0.085 Å. The distorted octahedral geometry at each Fe atom is completed by two PMe_3 and two carbonyl ligands. The carbonyl ligands have an average C≡O bond distance of 1.14 (2) Å and the PMe_3 ligands have an average P—C bond distance of 1.81 (1) Å. The Fe—Fe distance [2.947 (2) Å] of the cation of (I) is long when compared with other Fe—Fe distances. Typical Fe—Fe distances in complexes bridged by one silyl unit are 2.614 (1)–2.626 (2) Å (Ogino, Tobita & Kawano, 1991, 1992), but distances as long as 2.721 (2) Å have been reported (Ogino *et al.*, 1994). Supported μ_2-bridging hydride ligands have been found to exert both lengthening and shortening influences on metal–metal bond distances (Churchill, DeBoer & Rotella, 1976). The long Fe—Fe distance for the cation of (I) is most probably the result of complexation of a proton by the Fe—Fe bond of the neutral complex $Fe_2(CO)_4(PMe_3)_4(\mu_2\text{-}SiCl_2)$. Even in neutral complexes, a similar description of the supported bridging metal hydrides has been given (Mingos & Wales, 1990; Shriver, Kaesz & Adams, 1990). The bridging hydride was located from the difference Fourier map at distances of 1.77 and 1.63 Å from Fe(1) and Fe(2), respectively. Asymmetric bridging hydrides between two metal atoms have been described before (Bau, Teller, Kirtley & Koetzle, 1979).

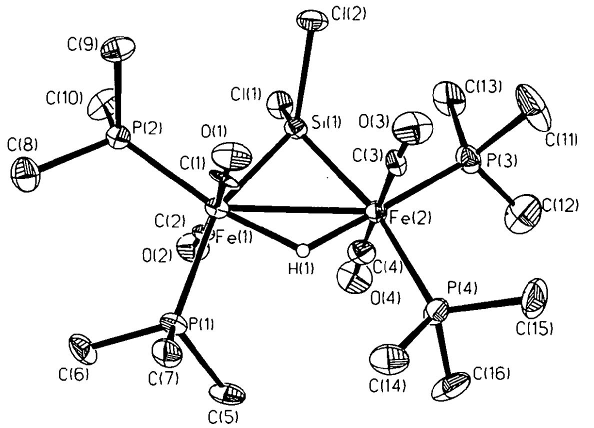


Fig. 1. Structure of the cation of $[Fe_2(CO)_4(PMe_3)_4(\mu_2\text{-}H)(\mu_2\text{-}SiCl_2)][Fe(CO)_4(SiCl_3)]$. The displacement ellipsoids are drawn at the 50% probability level. All H atoms except the bridging hydride have been omitted for clarity.

Experimental

Crystals of the title compound suitable for X-ray analysis were obtained from a saturated hexane solution.

Crystal data

$[Fe_2H(SiCl_2)(CO)_4(C_3H_9P)_4][Fe(SiCl_3)(CO)_4]$
$M_r = 930.4$
Monoclinic
$P2_1$
$a = 8.411 (2)$ Å
$b = 14.454 (3)$ Å
$c = 15.800 (3)$ Å
$\beta = 91.32 (3)°$
$V = 1920.3 (7)$ Å^3
$Z = 2$
$D_x = 1.609$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–$15°$
$\mu = 1.733$ mm^{-1}
$T = 119$ K
Plate
0.4 × 0.2 × 0.15 mm
Red

Data collection

Syntex $P2_1$ diffractometer
ω scans
Absorption correction: ψ scans (North, Phillips & Matthews, 1968)
$T_{min} = 0.242$, $T_{max} = 1.000$
4299 measured reflections
3524 independent reflections
2972 observed reflections [$F > 4\sigma(F)$]

$R_{int} = 0.0145$
$\theta_{max} = 25°$
$h = -9 \rightarrow 1$
$k = 0 \rightarrow 17$
$l = -18 \rightarrow 18$
3 standard reflections monitored every 97 reflections
intensity decay: 0.86%

Refinement

Refinement on F
$R = 0.0398$
$wR = 0.0432$
$S = 1.03$
2972 reflections
380 parameters
H atoms: see below
$w = 1/[\sigma^2(F) + 0.0008F^2]$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.52$ e Å^{-3}
$\Delta\rho_{\min} = -0.50$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	$U_{\rm eq}$
Fe(1)	0.33209 (12)	0.89647	0.56639 (7)	0.0132 (3)
Fe(2)	0.37144 (13)	0.85382 (11)	0.74807 (6)	0.0156 (3)
Fe(3)	0.93271 (14)	0.29936 (11)	0.84637 (7)	0.0231 (4)
P(1)	0.1493 (2)	1.01008 (16)	0.54853 (13)	0.0173 (6)
P(2)	0.4221 (2)	0.86635 (16)	0.43725 (12)	0.0174 (6)
P(3)	0.4589 (3)	0.75213 (17)	0.84443 (14)	0.0256 (7)
P(4)	0.2824 (3)	0.96590 (17)	0.83475 (13)	0.0227 (6)
Cl(1)	0.3885 (3)	0.64469 (15)	0.60299 (13)	0.0243 (6)
Cl(2)	0.7109 (2)	0.76178 (17)	0.62933 (13)	0.0257 (6)
Cl(3)	1.1359 (3)	0.4963 (2)	0.75624 (16)	0.0512 (9)
Cl(4)	1.3649 (3)	0.33440 (19)	0.83313 (17)	0.0420 (8)
Cl(5)	1.1842 (3)	0.47446 (19)	0.95710 (14)	0.0372 (7)
Si(1)	0.4629 (2)	0.78063 (17)	0.63383 (13)	0.0160 (6)
Si(2)	1.1420 (3)	0.39491 (19)	0.84892 (14)	0.0229 (7)
O(1)	0.5988 (7)	1.0268 (4)	0.5842 (4)	0.0262 (19)
O(2)	0.0679 (7)	0.7686 (5)	0.5330 (4)	0.030 (2)
O(3)	0.6781 (7)	0.9489 (5)	0.7622 (4)	0.034 (2)
O(4)	0.0713 (7)	0.7491 (5)	0.7429 (4)	0.037 (2)
O(5)	0.7474 (8)	0.4656 (6)	0.8795 (5)	0.053 (3)
O(6)	1.0238 (8)	0.2694 (6)	0.6712 (4)	0.047 (3)
O(7)	1.0826 (8)	0.1927 (5)	0.9834 (4)	0.050 (3)
O(8)	0.6647 (8)	0.1693 (6)	0.8371 (5)	0.055 (3)
C(1)	0.4940 (9)	0.9761 (6)	0.5760 (5)	0.018 (2)
C(2)	0.1698 (10)	0.8170 (6)	0.5474 (5)	0.019 (2)
C(3)	0.5591 (9)	0.9113 (6)	0.7549 (5)	0.019 (2)
C(4)	0.1887 (10)	0.7896 (6)	0.7442 (5)	0.024 (3)
C(5)	−0.0138 (9)	1.0035 (7)	0.6219 (5)	0.029 (3)
C(6)	0.0353 (10)	1.0173 (7)	0.4498 (5)	0.030 (3)
C(7)	0.2255 (9)	1.1263 (6)	0.5585 (5)	0.021 (2)
C(8)	0.3533 (12)	0.9395 (6)	0.3503 (5)	0.031 (3)
C(9)	0.6357 (9)	0.8754 (6)	0.4271 (5)	0.026 (3)
C(10)	0.3817 (10)	0.7515 (6)	0.3959 (5)	0.026 (3)
C(11)	0.6447 (13)	0.7803 (7)	0.8972 (7)	0.056 (4)
C(12)	0.3258 (14)	0.7308 (7)	0.9294 (7)	0.052 (4)
C(13)	0.4999 (14)	0.6353 (6)	0.8117 (6)	0.042 (3)
C(14)	0.2806 (11)	1.0815 (6)	0.7904 (6)	0.034 (3)
C(15)	0.3981 (13)	0.9887 (8)	0.9312 (5)	0.044 (4)
C(16)	0.0813 (11)	0.9541 (8)	0.8748 (6)	0.045 (4)
C(17)	0.8182 (10)	0.3993 (7)	0.8673 (5)	0.031 (3)
C(18)	0.9904 (9)	0.2810 (7)	0.7395 (6)	0.029 (3)
C(19)	1.0256 (10)	0.2368 (6)	0.9307 (5)	0.029 (3)
C(20)	0.7673 (11)	0.2212 (7)	0.8407 (6)	0.032 (3)

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

Fe(1)—Fe(2)	2.947 (2)	Fe(2)—P(4)	2.262 (3)
Fe(1)—P(1)	2.265 (3)	Fe(2)—Si(1)	2.242 (3)
Fe(1)—P(2)	2.235 (3)	Fe(2)—C(3)	1.786 (8)
Fe(1)—Si(1)	2.255 (3)	Fe(2)—C(4)	1.795 (9)
Fe(1)—C(1)	1.789 (8)	Cl(1)—Si(1)	2.115 (4)
Fe(1)—C(2)	1.800 (9)	Cl(2)—Si(1)	2.107 (3)
Fe(2)—P(3)	2.228 (3)		
Fe(2)—Fe(1)—P(1)	109.5 (1)	Fe(1)—P(1)—C(5)	113.7 (3)
Fe(2)—Fe(1)—P(2)	144.7 (1)	Fe(1)—P(1)—C(6)	119.5 (3)
P(1)—Fe(1)—P(2)	105.6 (1)	P(4)—Fe(2)—C(4)	95.6 (3)
Fe(2)—Fe(1)—Si(1)	48.9 (1)	Fe(1)—P(1)—C(7)	115.4 (3)
P(1)—Fe(1)—Si(1)	156.9 (1)	Si(1)—Fe(2)—C(4)	92.1 (3)
P(2)—Fe(1)—Si(1)	96.7 (1)	P(3)—Fe(2)—C(4)	87.3 (4)
Fe(2)—Fe(1)—C(1)	89.0 (3)	Fe(1)—P(2)—C(8)	118.1 (3)
P(1)—Fe(1)—C(1)	93.3 (3)	Fe(1)—P(2)—C(9)	115.1 (3)
P(2)—Fe(1)—C(1)	85.9 (3)	Fe(2)—C(4)—O(4)	178.6 (9)
Si(1)—Fe(1)—C(1)	94.2 (3)	Fe(1)—P(2)—C(10)	116.1 (3)
Fe(2)—Fe(1)—C(2)	95.6 (3)	Fe(2)—P(3)—C(11)	115.8 (4)
P(1)—Fe(1)—C(2)	86.2 (3)	Fe(2)—P(3)—C(12)	114.9 (4)
P(2)—Fe(1)—C(2)	89.7 (3)	Fe(2)—P(3)—C(13)	118.8 (3)
Si(1)—Fe(1)—C(2)	88.0 (3)	Fe(2)—P(4)—C(14)	115.3 (3)
C(1)—Fe(1)—C(2)	175.3 (4)	Fe(2)—P(4)—C(15)	117.3 (4)
Fe(1)—Fe(2)—P(3)	146.3 (1)	Fe(2)—P(4)—C(16)	117.9 (4)
Fe(1)—Fe(2)—P(4)	114.1 (1)	Fe(1)—Si(1)—Fe(2)	81.9 (1)
P(3)—Fe(2)—P(4)	99.7 (1)	Fe(1)—Si(1)—Cl(1)	116.5 (2)
Fe(1)—Fe(2)—Si(1)	49.2 (1)	Fe(2)—Si(1)—Cl(1)	121.2 (1)
P(3)—Fe(2)—Si(1)	97.3 (1)	Fe(1)—Si(1)—Cl(2)	123.6 (1)
P(4)—Fe(2)—Si(1)	161.7 (1)	Fe(2)—Si(1)—Cl(2)	116.5 (1)
Fe(1)—Fe(2)—C(3)	92.4 (3)	Cl(1)—Si(1)—Cl(2)	99.2 (2)
P(3)—Fe(2)—C(3)	89.3 (3)	Fe(1)—C(1)—O(1)	178.4 (7)
P(4)—Fe(2)—C(3)	86.1 (3)	Fe(1)—C(2)—O(2)	177.7 (8)
Si(1)—Fe(2)—C(3)	87.2 (3)	Fe(2)—C(3)—O(3)	177.8 (7)
Fe(1)—Fe(2)—C(4)	89.8 (3)		

H(1) was refined with isotropic U; all remaining H atoms were refined using a riding model with fixed isotropic U values. For the absolute structure refinement with inverted coordinates, $R = 0.0421$, $wR = 0.0475$ and $S = 0.60$. All calculations were performed using *SHELXTL-Plus* (Sheldrick, 1991).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1051). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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