

SHORT-FORMAT PAPERS

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Structure of Tetracarbonyl[tris(trimethylsilyl)phosphine]iron(0)

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Abstract. $[\text{Fe}\{(\text{CH}_3)_3\text{Si}\}_3\text{P}(\text{CO})_4]$, $M_r = 418.4$, monoclinic, Cc , $a = 9.548$ (2), $b = 15.052$ (3), $c = 15.552$ (6) Å, $\beta = 98.12$ (2)°, $V = 2212.7$ Å³, $Z = 4$, $D_x = 1.256$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.921$ mm⁻¹, $F(000) = 880$, $T = 296$ (1) K, final $R = 0.0590$ for 1204 unique observed diffractometer data and 199 variables. Coordination about Fe is trigonal bipyramidal with the phosphine occupying an axial site and an Fe-P bond length of 2.338 (4) Å.

Experimental. Title compound prepared according to the literature method (Schumann & Stelzer, 1968). Orange crystals from toluene on cooling to 278 K. Crystals mounted under argon in thin-walled glass capillaries. A prism with dimensions 0.24 × 0.33 × 0.62 mm used for X-ray work. Lattice parameters determined by least-squares fitting of setting angles of reflections, $12 < \theta < 15^\circ$, automatically centred on CAD-4 diffractometer. Intensities collected with graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\omega/2\theta$ scan mode, scan width $(0.8 + 0.35\tan\theta)^\circ$, aperture setting 4 mm, $1.5 \leq \theta \leq 25^\circ$, 2163 reflections measured, 2076 unique, 842 unobserved [$I \geq 3\sigma(I)$], index range $0 \leq h \leq 11$, $0 \leq k \leq 17$, $-18 \leq l \leq 18$ where $h + k = 2n$. Two intensity control reflections, monitored after every hour of data collection, showed only slight variation; Lorentz-polarization correction and absorption correction via ψ scans, max. = 99.55, min. = 77.12%. No extinction correction was applied. Structure was solved by Patterson synthesis. Full-matrix least-squares anisotropic refinement of positions of non-hydrogen atoms (no H's were refined). A non-Poisson-contribution weighting scheme was used with an experimental instability factor of $P = 0.06$ used in the calculation of $\sigma(I)$ to downweight intense reflections. The function $\sum w(|F_o| - |F_c|)^2$ was minimized where $w = 4(F_o)^2 / [\sum (F_o)^2]^2$, where $[\sum (F_o)^2]^2$

Table 1. Fractional coordinates and equivalent isotropic temperature factors (Å²) with e.s.d.'s in parentheses

	x	y	z	B_{eq}
Fe	0.080	0.3693 (1)	0.080	4.09 (4)
P	0.0095 (4)	0.2442 (3)	-0.0042 (3)	3.71 (7)
Si1	0.0686 (7)	0.1144 (3)	0.0673 (3)	5.3 (1)
Si2	-0.2304 (5)	0.2400 (4)	-0.0448 (3)	4.7 (1)
Si3	0.1058 (5)	0.2385 (4)	-0.1312 (3)	5.5 (1)
O1	0.016 (2)	0.4925 (8)	-0.0640 (8)	7.8 (4)
O2	0.182 (2)	0.5152 (9)	0.1926 (8)	9.3 (5)
O3	0.376 (1)	0.302 (1)	0.1155 (8)	7.5 (4)
O4	0.365 (1)	0.159 (1)	0.6906 (9)	11.1 (5)
C1	0.041 (2)	0.440 (1)	-0.011 (1)	5.3 (4)
C2	0.144 (2)	0.456 (1)	0.148 (1)	6.0 (4)
C3	0.260 (2)	0.324 (1)	0.101 (1)	5.1 (4)
C4	-0.055 (2)	0.349 (1)	0.144 (1)	7.2 (5)
C11	0.038 (2)	0.129 (1)	0.185 (1)	7.6 (6)
C12	0.262 (2)	0.090 (1)	0.067 (1)	5.9 (5)
C13	-0.043 (2)	0.022 (1)	0.017 (1)	6.9 (5)
C21	-0.278 (2)	0.163 (1)	-0.140 (1)	6.1 (5)
C22	-0.291 (2)	0.356 (1)	-0.074 (1)	6.0 (5)
C23	-0.315 (2)	0.195 (1)	0.051 (1)	6.5 (5)
C31	0.006 (2)	0.321 (1)	-0.209 (1)	7.3 (5)
C32	0.091 (2)	0.119 (1)	-0.176 (1)	6.8 (5)
C33	0.297 (2)	0.275 (2)	-0.106 (1)	8.9 (6)

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $\frac{1}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + abc\cos\gamma B(1,2) + accos\beta B(1,3) + bccos\alpha B(2,3)]$.

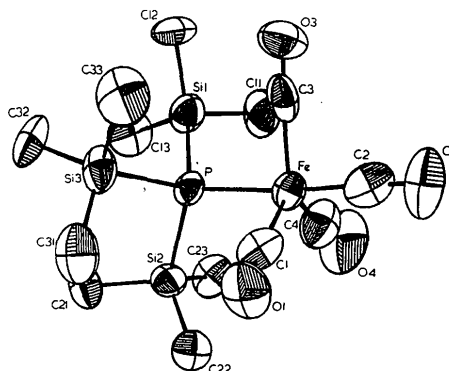


Fig. 1. The molecular structure of the title compound.

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Table 2. Bond lengths (Å) and angles (°)

Fe—P	2.338 (4)	Si2—C21	1.88 (2)
Fe—C1	1.77 (2)	Si2—C22	1.87 (2)
Fe—C2	1.74 (2)	Si2—C23	1.92 (2)
Fe—C3	1.84 (2)	Si3—C31	1.89 (2)
Fe—C4	1.77 (2)	Si3—C32	1.93 (2)
P—Si1	2.279 (6)	Si3—C33	1.89 (2)
P—Si2	2.289 (6)	O1—C1	1.14 (2)
P—Si3	2.294 (6)	O2—C2	1.15 (2)
Si1—C11	1.91 (2)	O3—C3	1.15 (2)
Si1—C12	1.89 (2)	O4—C4	1.123 (12)
Si1—C13	1.86 (2)		
P—Fe—C1	91.7 (5)	C11—Si1—C12	107.8 (9)
P—Fe—C2	174.9 (6)	C11—Si1—C13	110. (1)
P—Fe—C3	89.2 (5)	C12—Si1—C13	110.9 (9)
P—Fe—C4	90.0 (7)	P—Si2—C21	110.9 (7)
C1—Fe—C2	92.5 (8)	P—Si2—C22	107.6 (6)
C1—Fe—C3	116.9 (8)	P—Si2—C23	107.9 (6)
C1—Fe—C4	117.9 (9)	C21—Si2—C22	110.3 (8)
C2—Fe—C3	86.2 (8)	C21—Si2—C23	108.3 (9)
C2—Fe—C4	91. (1)	C22—Si2—C23	111.9 (9)
C3—Fe—C4	125.2 (8)	P—Si3—C31	107.3 (7)
Fe—P—Si1	112.6 (2)	P—Si3—C32	109.1 (7)
Fe—P—Si2	112.0 (2)	P—Si3—C33	107.7 (6)
Fe—P—Si3	113.2 (2)	C31—Si3—C32	112.2 (8)
Si1—P—Si2	106.3 (3)	C31—Si3—C33	109. (1)
Si1—P—Si3	106.6 (3)	C32—Si3—C33	111. (1)
Si2—P—Si3	105.6 (2)	Fe—C1—O1	174. (2)
P—Si1—C11	108.0 (7)	Fe—C2—O2	177. (2)
P—Si1—C12	110.0 (6)	Fe—C3—O3	174. (2)
P—Si1—C13	110.5 (7)	Fe—C4—O4	173. (1)

$= [S^2(C + R^2B) + \{P(F_0)^2\}^2]/Lp^2$, where S = scan rate, C = total integrated peak count, R = ratio of scan time to background counting time, B = total background count, Lp = Lorentz-polarization factor. Final $R = 0.0590$, $wR = 0.0659$. $\Delta/\sigma(\max.) = 0.127$, the largest peak in final ΔF map ± 0.4 (3) e Å⁻³. Scattering-factor data from *International Tables for X-ray Crystallography* (1974). Final atomic parameters are listed in

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Structure of Di[bis(triphenylphosphine)iminium] Hexachlororhenate(IV)

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Abstract. $[(C_6H_5)_3PNP(C_6H_5)_3]_2[ReCl_6]$, $M_r = 1476.1$, orthorhombic, $Pbca$ (D_{2h}^{15}), $a = 16.59$ (1), $b = 19.49$ (1), $c = 19.88$ (1) Å, $V = 6428$ (3) Å³, $Z = 4$, $D_x = 1.53$ g cm⁻³, $Mo K\alpha$, $\lambda(K\alpha_1) = 0.7093$ Å, $\mu = 23.1$ cm⁻¹, $F(000) = 2970$ (including anomalous dispersion), $T = 123$ K, $R(F^2) = 0.109$ for 4127 independent reflections. The $ReCl_6^{2-}$ anion has crystallographically imposed symmetry $\bar{1}$, the Re—Cl distances are 2.351 (3), 2.355 (3) and 2.376 (3) Å, and

Table 1.* Computer programs used: *SDP-Plus* (Frenz, 1981), *ORTEPII* (Johnson, 1976). The molecule and the numbering scheme are shown in Fig. 1. Bond lengths and angles are given in Table 2.

Related literature. Bennett, Neustadt, Parry & Cagle (1978), Kilbourn, Raeburn & Thompson (1969), Pickardt, Rösch & Schumann (1976).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44595 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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the Cl—Re—Cl angles are 89.72 (11), 91.10 (10) and 91.23 (10)°.

Experimental. Dark yellowish-green crystals of bis(triphenylphosphine)iminium hexachlororhenate, $[PPN]_2[ReCl_6]$, were obtained by combining 0.5 g rhenium pentachloride, 0.1 g rhenium powder, 0.2 g selenium powder, and 1.0 g $[PPN]Cl$ with 1.0 g bis(dimethyloctylsilyl) selenide (Chau, Wardle & Ibers,