Acta Cryst. (1992). **C48**, 159–160

Structure of [Bis(diphenylphosphino)methane-*P*]tetracarbonyliron(0)

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(*Received* 15 *January* 1991; *accepted* 29 *July* 1991)

Abstract. $[Fe(CO)_4(C_{25}H_{22}P_2)]$, $M_r = 552.29$, monoclinic, $P2_1/c$, $a = 8.698$ (3), $b = 27.478$ (3), $c = 11.161$ (2) Å, $\beta = 95.77$ (2)°, $V = 2654$ (1) Å^3, $Z = 4$, $D_x = 1.382$, $D_m = 1.38$ (1) g cm^{-3} (flotation), graphite-monochromated Mo $K\alpha$ ($\lambda = 0.71073$ Å), $\mu = 7.17$ cm^{-1}, $F(000) = 1136$, $T = 298$ (1) K, final $R = 0.055$ and $wR = 0.051$ for 1550 observed reflections. Coordination around Fe is roughly trigonal bipyramidal involving the four CO groups and one of the phosphines occupying an axial site with an Fe—P bond length of 2.267 (2) Å. The distances of the other phosphine to Fe preclude any other Fe—P bond.

Experimental. Crystalline samples of the title compound were prepared and provided by E. J. S. Vichi (Institute of Chemistry – UNICAMP, SP, Brazil). A crystal of dimensions 0.40 × 0.20 × 0.15 mm was used for the data collection on a CAD-4 automatic diffractometer. Cell dimensions were determined by a least-squares fit of settings for 25 reflections with $9 < \theta < 17°$. Intensity measurements were carried out up to $\theta = 25°$ using an $\omega/2\theta$-scan mode and graphite-monochromated Mo $K\alpha$ radiation, index range $-10 \leq h \leq 10$, $0 \leq k \leq 32$, $0 \leq l \leq 13$. Lorentz and polarization corrections were applied. Correction for absorption (Coppens, Leiserowitz & Rabinovich, 1965) was applied (maximum and minimum transmission coefficients were 0.8853 and 0.8756 respectively) and correction for intensity decay (linear-with-time) was also applied since the intensity of two standard reflections ($2,\bar{1}\bar{4},0$, 400) decreased 8.7% over 32.1 h of data collection. Of the 5042 measured reflections 4556 were unique and only 1550 were considered observed [$F > 6\sigma(F)$] and retained for use in the structure analysis. $R_{\rm int} = 0.038$.

The structure was solved by the analysis of the Patterson map (*SHELXS*86; Sheldrick, 1986) followed by difference Fourier syntheses and refined by minimization of $\sum w_i(k|F_o| - |F_c|)^2$ through iterative blocked full-matrix least-squares (two blocks containing both Fe and from C1*A* to C12 and from C25 to C24 with 199 and 136 parameters respectively) calculations using *SHELX*76 (Sheldrick, 1976).

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (*Hamilton*, 1959) *with e.s.d.'s in parentheses*

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
Fe	0.0152 (2)	0.4417 (1)	0.2883 (1)	4.94 (6)
C1*A*	0.033 (1)	0.4299 (4)	0.447 (1)	4.6 (4)
O1	0.0423 (8)	0.4246 (3)	0.5488 (7)	6.4 (3)
C2*A*	−0.151 (1)	0.4787 (4)	0.305 (1)	6.3 (5)
O2	−0.2616 (9)	0.5003 (3)	0.3106 (8)	8.8 (4)
C3*A*	−0.108 (1)	0.4005 (5)	0.201 (1)	6.9 (5)
O3	−0.191 (1)	0.3724 (4)	0.1517 (9)	12.2 (5)
C4*A*	0.119 (1)	0.4916 (4)	0.230 (1)	6.0 (5)
O4	0.183 (1)	0.5242 (3)	0.194 (1)	11.4 (5)
P1	0.2177 (3)	0.3926 (1)	0.2594 (2)	3.90 (9)
C1	0.392 (1)	0.3972 (3)	0.3665 (8)	3.9 (4)
C2	0.403 (1)	0.4329 (3)	0.4536 (9)	4.2 (4)
C3	0.535 (1)	0.4352 (4)	0.5358 (9)	4.7 (4)
C4	0.655 (1)	0.4025 (5)	0.529 (1)	5.6 (5)
C5	0.644 (1)	0.3674 (4)	0.440 (1)	5.8 (5)
C6	0.512 (1)	0.3641 (3)	0.3606 (8)	4.4 (4)
C7	0.295 (1)	0.4017 (3)	0.1138 (8)	3.6 (4)
C8	0.205 (1)	0.3886 (4)	0.009 (1)	5.6 (5)
C9	0.254 (2)	0.3956 (5)	−0.102 (1)	8.0 (6)
C10	0.395 (2)	0.4157 (5)	−0.110 (1)	7.1 (6)
C11	0.484 (1)	0.4312 (5)	−0.012 (1)	7.5 (6)
C12	0.436 (1)	0.4234 (4)	0.104 (1)	5.4 (4)
C25	0.1695 (9)	0.3274 (3)	0.2597 (7)	4.1 (3)
P2	0.0895 (3)	0.3082 (1)	0.3999 (2)	4.05 (9)
C13	−0.0070 (9)	0.2506 (3)	0.3529 (8)	3.3 (3)
C14	0.018 (1)	0.2238 (3)	0.2517 (8)	4.2 (4)
C15	−0.057 (1)	0.1810 (3)	0.2288 (9)	5.0 (4)
C16	−0.156 (1)	0.1629 (3)	0.305 (1)	5.5 (4)
C17	−0.182 (1)	0.1877 (4)	0.407 (1)	5.4 (4)
C18	−0.109 (1)	0.2324 (3)	0.4299 (8)	4.5 (4)
C19	0.2625 (9)	0.2865 (4)	0.4915 (8)	3.9 (4)
C20	0.322 (1)	0.3151 (3)	0.5871 (9)	4.4 (4)
C21	0.450 (1)	0.3011 (5)	0.6610 (9)	5.7 (5)
C22	0.520 (1)	0.2566 (6)	0.642 (1)	6.6 (5)
C23	0.461 (1)	0.2270 (4)	0.550 (1)	6.1 (5)
C24	0.334 (1)	0.2420 (4)	0.4740 (8)	4.8 (4)

Table 2. *Selected interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Fe—C1*A*	1.79 (1)	C4*A*—O4	1.15 (2)
Fe—C2*A*	1.79 (1)	P1—C1	1.838 (9)
Fe—C3*A*	1.79 (1)	P1—C7	1.84 (2)
Fe—C4*A*	1.80 (1)	P1—C25	1.841 (9)
Fe—P1	2.267 (3)	C25—P2	1.853 (9)
C1*A*—O1	1.15 (1)	P2—C13	1.843 (9)
C2*A*—O2	1.14 (1)	P2—C19	1.832 (8)
C3*A*—O3	1.16 (1)		

C1*A*—Fe—C2*A*	89.4 (5)	Fe—P1—C1	117.9 (3)
C1*A*—Fe—C3*A*	114.4 (5)	Fe—P1—C7	113.8 (3)
C1*A*—Fe—C4*A*	119.9 (5)	Fe—P1—C25	113.5 (3)
C1*A*—Fe—P1	92.4 (3)	C1—P1—C7	102.4 (4)
C2*A*—Fe—C3*A*	88.3 (5)	C1—P1—C25	103.8 (5)
C2*A*—Fe—C4*A*	92.6 (5)	C7—P1—C25	103.8 (4)
C2*A*—Fe—P1	176.9 (4)	P1—C25—P2	112.6 (4)
C3*A*—Fe—C4*A*	125.7 (5)	C25—P2—C13	101.7 (4)
C3*A*—Fe—P1	88.7 (4)	C25—P2—C19	101.9 (4)
C4*A*—Fe—P1	88.7 (4)	C13—P2—C19	101.8 (5)

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Weights $w_i = [\sigma^2_{oi}(F) + 0.00026|F_o|^2]^{-1}$, where $\sigma^2_{oi}(F)$ is the variance based on counting statistics, were assigned to observed reflections. The refinement was conducted until convergence. Final unweighted and weighted R factors were 0.055 and 0.051, and goodness-of-fit = 2.21. Maximum and minimum heights in final difference Fourier synthesis were 0.45 and -0.29 e Å^{-3} respectively. Maximum ratio of least-squares shift to e.s.d. in final refinement cycle was < 0.01.

Anisotropic temperature factors were associated with all non-H atoms. The H-atom positions were calculated and an isotropic temperature factor of 6.00 Å^2 was assigned to them. Complex neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Final atomic parameters are listed in Table 1.* One discrete molecule and its numbering scheme are shown in Fig. 1. Relevant interatomic distances and angles are given in Table 2.

Related literature. A discussion about the chemistry of this kind of compound has been published (Santos, Stein & Vichi, 1985). When binding to Fe, the ligand is usually bidentate (Fontaine, Jacobsen, Shaw & Thornton-Pett, 1987; Jacobsen, Shaw & Thornton-Pett, 1987); however, there are cases where it is monodentate, for instance with Os (Cartwright, Clucas, Dawson, Foster, Harding & Smith, 1986). In the case of the title compound the closest contact between Fe and P2 is intramolecular with a distance of 3.907 (3) Å.

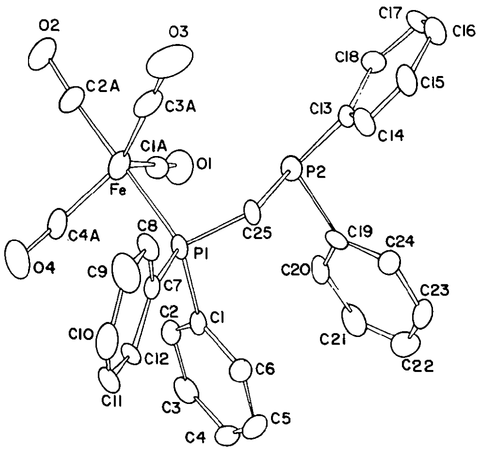

Fig. 1. *ORTEP* (Johnson, 1965) representation of the title compound.

This work has received the support of FAPESP, CNPQ and FINEP, which is hereby gratefully acknowledged.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and interatomic distances and angles in the phenyl rings have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54480 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1992). C**48**, 160–162

Structure of Dicarbonyl(η^5-cyclopentadienyl)[(α,1,2-η)-p-methylbenzyl]tungsten

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(*Received* 16 *November* 1990; *accepted* 17 *April* 1991)

Abstract. [W(CO)$_2$(C$_5$H$_5$)(C$_8$H$_9$)], $M_r = 410.12$, monoclinic, $P2_1/n$, $a = 6.339$ (1), $b = 17.141$ (2), $c = 12.526$ (1) Å, $\beta = 101.86$ (1)°, $V = 1332.0$ Å^3, $Z = 4$, $D_x = 2.05$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 82.92$ cm^{-1}, $F(000) = 776$, ambient temperature, $R = 0.022$ for 2446 unique observed reflections [$F_o >$

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