Crystal structure of ((triphenylphosphoranylidene)methyl)triphenylphosphonium octacarbonyl-diiron, $[(C_6H_5)_3PCHP(C_6H_5)_3]_2[Fe_2(CO)_8]$

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Source of material: The compound was obtained as a byproduct in the reaction of $Fe(CO)_5$ with $Ph_3P=C=PPh_3$ in toluene; main product is $(CO)_4Fe=C=C=PPh_3$ (see ref. 1).

The bond angles at C(5) are: $H(5)-C(5)-P(1) = 112.69^\circ$, $H(5)-C(5)-P(2) = 115.69^\circ$, $P(1)-C(5)-P(2) = 131.50^\circ$ (sum amounting to 359.88°); they correspond to a sp^2 hybridized carbon atom. The angle $P(1)-C(5)-P(2)$ is 3.3° larger than in $[Ph_3PCHPPh_3][P_2(CN)_{10}]$, but the other bonding parameters agree well (see ref. 2). In the anion the angle $C(ax)-Fe-C(eq)$ vary between 95° and 98° (average 96.0°) so the equatorial CO groups are tilted toward the center of symmetry; the other bonding parameters correspond to those in $[Et_4N]_2[Fe_2(CO)_8]$ (see ref. 3).

$C_{82}H_{62}Fe_2O_8P_4$, triclinic, $P\bar{1}$ (No. 2), $a = 10.352(1) \text{ \AA}$, $b = 13.542(2) \text{ \AA}$, $c = 14.043(2) \text{ \AA}$, $\alpha = 63.451(9)^\circ$, $\beta = 85.57(1)^\circ$, $\gamma = 83.61(1)^\circ$, $V = 1749.2 \text{ \AA}^3$, $Z = 1$, $R(F) = 0.046$, $R_w(F^2) = 0.117$.

Table 1. Parameters used for the X-ray data collection

Crystal:	dark red, rugged ellipsoid, size $0.15 \times 0.31 \times 0.44 \text{ mm}$
Wavelength:	$Cu K\alpha$ radiation ($1.54178 \text{ \AA}$)
μ :	46.51 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	ω
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	110°
$N(hkl)_{\text{unique}}$:	4378
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	371
Program:	SHELXL-96

Table 2. Final atomic coordinates and displacement parameters (in $\text{\AA}^2$)

Atom	Site	x	y	z	U_{iso}
H(5)	2i	0.489(4)	0.675(3)	0.400(3)	0.05(1)
H(112)	2i	0.3372	0.8847	0.1081	0.087(7)
H(113)	2i	0.2607	0.8006	0.0109	0.087(7)
H(114)	2i	0.3565	0.6266	0.0308	0.087(7)
H(115)	2i	0.5288	0.5368	0.1478	0.087(7)
H(116)	2i	0.6053	0.6209	0.2450	0.087(7)
H(122)	2i	0.6734	1.0200	0.2136	0.090(7)
H(123)	2i	0.8851	1.0722	0.1531	0.090(7)
H(124)	2i	1.0363	0.9551	0.1083	0.090(7)
H(125)	2i	0.9757	0.7859	0.1239	0.090(7)
H(126)	2i	0.7639	0.7336	0.1844	0.090(7)
H(132)	2i	0.3429	0.8899	0.3788	0.076(6)
H(133)	2i	0.1897	1.0443	0.3381	0.076(6)
H(134)	2i	0.1910	1.1943	1.1668	0.076(6)
H(135)	2i	0.3455	1.1900	0.0363	0.076(6)
H(136)	2i	0.4987	1.0356	0.0769	0.076(6)
H(212)	2i	0.8966	0.7427	0.4582	0.084(6)
H(213)	2i	1.0200	0.8940	0.4143	0.084(6)
H(214)	2i	0.9158	1.066	0.3931	0.084(6)
H(215)	2i	0.6882	1.0866	0.4158	0.084(6)
H(216)	2i	0.5648	0.9352	0.4597	0.084(6)
H(222)	2i	0.3618	0.6596	0.5537	0.076(6)
H(223)	2i	0.2192	0.6457	0.6974	0.076(6)
H(224)	2i	0.2877	0.6855	0.8316	0.076(6)
H(225)	2i	0.4988	0.7392	0.8222	0.076(6)
H(226)	2i	0.6414	0.7531	0.6786	0.076(6)
H(232)	2i	0.7433	0.5683	0.4084	0.093(7)
H(233)	2i	0.8748	0.3995	0.4753	0.093(7)
H(234)	2i	0.9358	0.3146	0.6544	0.093(7)
H(235)	2i	0.8652	0.3985	0.7666	0.093(7)
H(236)	2i	0.7337	0.5673	0.6997	0.093(7)

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Fe	2i	0.02498(5)	0.42259(4)	0.10442(4)	0.0456(4)	0.0316(3)	0.0496(4)	-0.0001(2)	-0.0050(3)	-0.0151(3)
C(1)	2i	0.1921(4)	0.4394(3)	0.0693(3)	0.054(3)	0.037(2)	0.075(3)	-0.002(2)	-0.015(2)	-0.023(2)
O(1)	2i	0.3024(3)	0.4445(3)	0.0479(3)	0.048(2)	0.082(2)	0.131(3)	-0.009(2)	-0.006(2)	-0.046(2)
C(2)	2i	-0.0735(4)	0.5172(3)	0.1373(4)	0.066(3)	0.040(2)	0.079(3)	-0.001(2)	0.000(2)	-0.026(2)
O(2)	2i	-0.1408(4)	0.5724(3)	0.1690(3)	0.106(3)	0.073(2)	0.135(3)	0.005(2)	0.025(2)	-0.064(2)
C(3)	2i	-0.0414(4)	0.3357(3)	0.0613(3)	0.048(2)	0.041(2)	0.065(3)	-0.001(2)	-0.008(2)	-0.014(2)
O(3)	2i	-0.0818(3)	0.2723(3)	0.0388(3)	0.091(2)	0.060(2)	0.109(3)	-0.027(2)	-0.015(2)	-0.041(2)
C(4)	2i	0.0438(4)	0.3238(3)	0.2346(4)	0.071(3)	0.052(2)	0.060(3)	0.001(2)	-0.007(2)	-0.019(2)
O(4)	2i	0.0528(4)	0.2560(3)	0.3219(3)	0.142(4)	0.092(3)	0.054(2)	0.006(2)	-0.019(2)	-0.004(2)
C(5)	2i	0.5431(4)	0.7252(3)	0.3876(3)	0.053(2)	0.034(2)	0.043(2)	-0.012(2)	-0.009(2)	-0.013(2)
P(1)	2i	0.54093(8)	0.82452(7)	0.25922(7)	0.0462(5)	0.0296(5)	0.0374(5)	0.0004(4)	-0.0065(4)	-0.0147(4)
P(2)	2i	0.62331(8)	0.71168(7)	0.49465(7)	0.0424(5)	0.0316(5)	0.0351(5)	-0.0051(4)	-0.0027(4)	-0.0118(4)
C(112)	2i	0.3764(2)	0.8134(2)	0.1163(2)	0.071(3)	0.043(2)	0.058(2)	-0.011(2)	-0.021(2)	-0.014(2)
C(113)	2i	0.3313(3)	0.7638(2)	0.0589(2)	0.107(4)	0.063(3)	0.065(3)	-0.032(3)	-0.040(3)	-0.009(2)
C(114)	2i	0.3878(3)	0.6611(2)	0.0706(2)	0.139(5)	0.069(3)	0.054(3)	-0.046(3)	-0.018(3)	-0.025(2)
C(115)	2i	0.4895(3)	0.6081(2)	0.1397(2)	0.109(4)	0.050(2)	0.069(3)	-0.014(2)	-0.005(3)	-0.036(2)
C(116)	2i	0.5347(2)	0.6578(2)	0.1971(2)	0.075(3)	0.047(2)	0.060(2)	0.000(2)	-0.014(2)	-0.029(2)
C(111)	2i	0.4781(2)	0.7604(2)	0.1853(2)	0.054(2)	0.040(2)	0.041(2)	-0.004(2)	-0.008(2)	-0.016(2)
C(122)	2i	0.7353(2)	0.9720(2)	0.1952(2)	0.065(3)	0.044(2)	0.043(2)	-0.010(2)	-0.002(2)	-0.013(2)
C(123)	2i	0.8603(3)	1.0028(2)	0.1595(2)	0.077(3)	0.055(3)	0.054(3)	-0.026(2)	-0.003(2)	-0.006(2)
C(124)	2i	0.9495(2)	0.9337(3)	0.1331(2)	0.060(3)	0.093(4)	0.055(3)	-0.020(3)	0.005(2)	-0.009(3)
C(125)	2i	0.9137(2)	0.8338(2)	0.1423(2)	0.059(3)	0.091(4)	0.072(3)	0.002(3)	0.010(2)	-0.034(3)
C(126)	2i	0.7888(2)	0.8030(2)	0.1780(2)	0.058(3)	0.051(2)	0.066(3)	0.001(2)	0.003(2)	-0.028(2)
C(121)	2i	0.6996(2)	0.8721(2)	0.2044(2)	0.052(2)	0.036(2)	0.035(2)	-0.001(2)	-0.006(2)	-0.014(2)
C(132)	2i	0.3434(2)	0.9514(2)	0.3086(2)	0.052(2)	0.047(2)	0.060(2)	-0.002(2)	0.001(2)	-0.020(2)
C(133)	2i	0.2530(2)	1.0425(2)	0.2846(2)	0.061(3)	0.062(3)	0.085(3)	0.009(2)	0.007(2)	-0.035(3)
C(134)	2i	0.2538(2)	1.1311(2)	0.1835(2)	0.069(3)	0.050(3)	0.101(4)	0.021(2)	-0.013(3)	-0.034(3)
C(135)	2i	0.3450(3)	1.1285(2)	0.1064(2)	0.083(3)	0.041(2)	0.072(3)	0.008(2)	-0.021(3)	-0.017(2)
C(136)	2i	0.4354(2)	1.0374(2)	0.1305(2)	0.068(3)	0.039(2)	0.050(2)	0.006(2)	-0.007(2)	-0.016(2)
C(131)	2i	0.4346(2)	0.9488(2)	0.2315(2)	0.048(2)	0.033(2)	0.047(2)	0.003(2)	-0.007(2)	-0.018(2)
C(212)	2i	0.8539(2)	0.8131(2)	0.4495(2)	0.051(2)	0.053(2)	0.050(2)	-0.013(2)	-0.006(2)	-0.011(2)
C(213)	2i	0.9267(2)	0.9025(2)	0.4236(2)	0.059(3)	0.081(3)	0.068(3)	-0.035(3)	-0.005(2)	-0.012(3)
C(214)	2i	0.8652(3)	1.0040(2)	0.4111(2)	0.104(4)	0.069(3)	0.061(3)	-0.049(3)	-0.003(3)	-0.021(2)
C(215)	2i	0.7309(3)	1.0161(2)	0.4245(2)	0.111(4)	0.045(2)	0.061(3)	-0.025(2)	0.004(3)	-0.025(2)
C(216)	2i	0.6581(2)	0.9268(2)	0.4504(2)	0.072(3)	0.040(2)	0.056(2)	-0.011(2)	0.004(2)	-0.024(2)
C(211)	2i	0.7196(2)	0.8253(2)	0.4629(2)	0.052(2)	0.037(2)	0.032(2)	-0.012(2)	0.000(2)	-0.012(2)
C(222)	2i	0.3899(2)	0.6759(2)	0.6087(2)	0.057(2)	0.050(2)	0.056(2)	-0.015(2)	0.005(2)	-0.019(2)
C(223)	2i	0.3057(2)	0.6677(2)	0.6935(2)	0.055(3)	0.071(3)	0.072(3)	-0.017(2)	0.020(2)	-0.022(2)
C(224)	2i	0.3461(3)	0.6912(2)	0.7728(2)	0.078(3)	0.070(3)	0.055(3)	-0.003(2)	0.024(2)	-0.017(2)
C(225)	2i	0.4707(3)	0.7229(2)	0.7672(2)	0.084(3)	0.073(3)	0.046(2)	-0.007(2)	0.006(2)	-0.028(2)
C(226)	2i	0.5549(2)	0.7311(2)	0.6824(2)	0.059(2)	0.059(2)	0.045(2)	-0.006(2)	0.001(2)	-0.024(2)
C(221)	2i	0.5145(2)	0.7076(2)	0.6032(2)	0.046(2)	0.036(2)	0.037(2)	-0.004(2)	0.002(2)	-0.013(2)
C(232)	2i	0.7683(3)	0.5335(2)	0.4818(2)	0.071(3)	0.056(2)	0.071(3)	0.014(2)	-0.024(2)	-0.034(2)
C(233)	2i	0.8459(3)	0.4339(2)	0.5213(2)	0.083(3)	0.071(3)	0.107(4)	0.025(3)	-0.027(3)	-0.056(3)
C(234)	2i	0.8819(3)	0.3838(2)	0.6270(2)	0.078(3)	0.051(3)	0.096(4)	0.021(2)	-0.021(3)	-0.021(3)
C(235)	2i	0.8402(3)	0.4333(2)	0.6932(2)	0.088(3)	0.069(3)	0.059(3)	0.021(3)	-0.015(3)	-0.004(2)
C(236)	2i	0.7626(3)	0.5329(2)	0.6537(2)	0.075(3)	0.057(3)	0.047(2)	0.013(2)	-0.009(2)	-0.014(2)
C(231)	2i	0.7266(2)	0.5830(2)	0.5480(2)	0.044(2)	0.033(2)	0.049(2)	-0.004(2)	-0.007(2)	-0.012(2)

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