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SYNTHESIS AND CRYSTAL STRUCTURE OF A π-ALLYL IRON CARBONYL COMPLEX DERIVED FROM A FLUOROCARBON CONTAINING LIGAND BY LOSS OF FLUORINE

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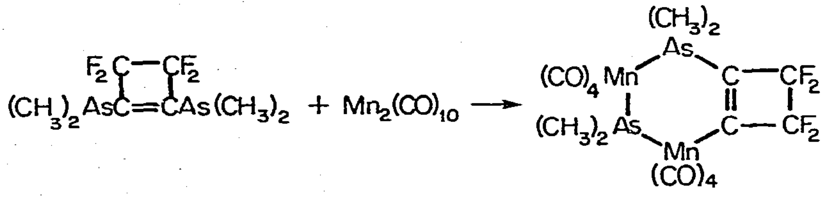
SUMMARY

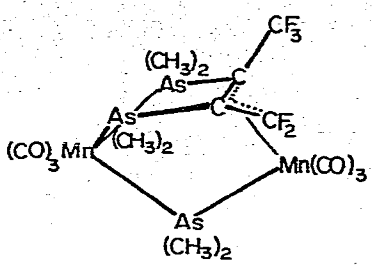
The β-chlorovinylphosphines $R_2P\overline{C = CCl(C}F_2)_3$ (R = C_6H_5, C_6H_{11}) react with $Fe(CO)_5$ yielding compounds of stoichiometry $R_2P\overline{C = C =\!= CF(C}F_2)_2Fe_2(CO)_6$. The crystal structure of one of these (R = C_6H_{11}) has been determined from X-ray diffraction data and refined by least-squares to $\underline{R}$ = 0.037 (2313 reflections with $\underline{I} > 2.3\sigma_I$). Crystals are triclinic, space group $P\bar{1}$, $\underline{a}$ = 10.253(5), $\underline{b}$ = 15.590(7), $\underline{c}$ = 9.390(4)Å, α = 99.88(3), β = 103.21(2), γ = 92.02(2)°, $\underline{Z}$ = 2. The fluorinated π-allyl group is σ-bonded to one Fe atom and π-bonded to the other.

Fluorocarbon bridged di(tertiary arsines and phosphines) have been extensively studied in these laboratories as ligands in metal carbonyl complexes (1-4). In a number of cases, products with unexpected structures are obtained as an $-AsR_2$ moiety is lost from a vinylic position. Usually the displaced group is bound elsewhere in the molecule as in I (1,5,6).



I

An even more dramatic reorganization occurs when cis-$(CH_3)_2AsC(CF_3) = C(CF_3)As(CH_3)_2$ is heated with $Mn_2(CO)_{10}$ and amongst the product is a molecule with structure II (7). Here a novel fluorine containing π-allyl fragment has been created by loss of a fluorine atom from a vinylic CF_3 group.



II

The β-chlorovinyl derivatives $R_2EC = CCl(CF_2)_n$ (R = alkyl, aryl; E = As,P) are easily obtained by treating R_2EH with $Cl\overline{C = CCl(C}F_2)_n$ (1,8) and these react normally with group VI hexacarbonyls to give compounds such as $(C_6H_{11})_2P\overline{C = CCl(C}F_2)_2$-$Mo(CO)_5$. The crystal structure of this derivative shows no interaction between the vinylic chlorine atom and the metal(9). The same ligands (n=2) also react with $Fe(CO)_5$ to give only the usual carbonyl substituted products. However, when $Fe(CO)_5$ is heated with $R_2P\overline{C = CCl(C}F_2)_3$ (R = C_6H_{11} or C_6H_5), complexes of unexpected stoichiometry are obtained. The mass spectra show a parent peak corresponding to the formula $R_2P\overline{C = CCF(C}F_2)_2Fe_2(CO)_6$ and indicate the presence of six carbonyl groups. The formula is confirmed by microanalysis. The infrared spectra show the absence of ν(C=C) and ν(C–Cl) and the Mössbauer spectrum shows the presence of two inequivalent iron atoms.

CRYSTALLOGRAPHY

Crystals of the complex with $R=C_6H_{11}$,III, were obtained by allowing a saturated n-hexane solution of the compound to cool slowly. Subsequent analysis showed that n-hexane solvate molecules are incorporated in the crystal lattice.

Cell dimensions were obtained by a least-squares fit of the angular parameters of 12 reflections ($2\theta > 30°$) centered on a Picker FACS-I four-circle diffractometer using Mo-Kα radiation (graphite monochromator, $\lambda = 0.70926$ Å). As the compound decomposes slowly in an X-ray beam data was collected on two crystals: one for reflections $2\theta < 30°$ and the other for $30 < 2\theta < 40°$. In each case two standard reflections were measured after every 75 data points and the data scaled accordingly. The scale factors of the two data sets were refined independently. 2659 independent reflections were measured by the θ-2θ scan technique and of these 2313 with $\underline{I} > 2.3\sigma_{\underline{I}}$ were used in refinement.

<u>Crystal data</u>. - $C_{26}H_{29}F_5Fe_2O_6P$, f.w. 675.17, triclinic, space group $P\bar{1}$, $\underline{a}$ = 10.253(5), $\underline{b}$ = 15.590(7), $\underline{c}$ = 9.390(4)Å, α = 99.88(3), β = 103.21(2), γ = 92.02(2)°, $\underline{U}$ = 1435Å^3, $\underline{D}_m$ = 1.54, $\underline{D}_c$(Z=2) = 1.56 g cm^{-3}, μ = 11.68 cm^{-1}.

The structure was solved by conventional Patterson and Fourier methods and refined by full-matrix least-squares with anisotropic temperature factors for all non-hydrogen atoms except the cyclohexyl and n-hexane carbon atoms which were refined isotopically. The function being minimized was $\Sigma\underline{w}(|\underline{F}_o|-|\underline{F}_c|)^2$ where the weights $\underline{w}$ were taken to be $1/\sigma_{\underline{F}}^2$. The central carbon atom of the n-hexane molecule was found to be disordered over two sites. Each site was therefore assigned an estimated relative occupancy proportional to

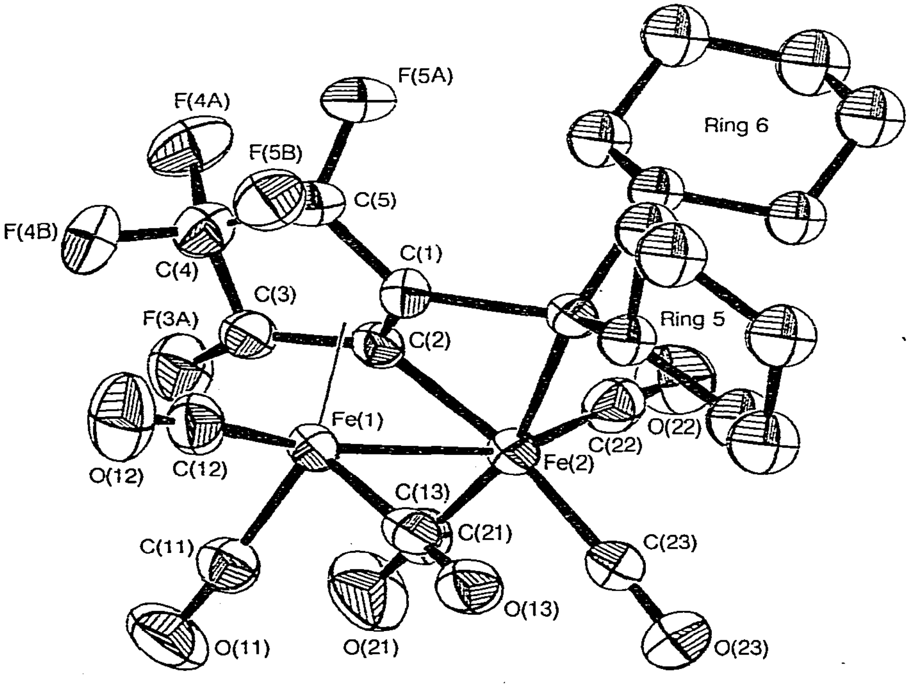


Figure 1

Projected view of the molecule showing 50% probability thermal ellipsoids and atomic labelling scheme. Hydrogen atoms have been deleted for clarity.

the electron density in a Fourier synthesis and the positional and temperature factors allowed to refine in further least-squares cycles. The hydrogen atoms were held invarient at their geometrically calculated positions ($\underline{r}_{C-H}$=0.95Å) and were assigned the temperature factors of the carbon atoms to which they were attached. The methyl hydrogens of the n-hexane molecule were located in a difference map and the positions modified to give an idealized geometry about the carbon atom.

In the final cycle no parameter shift exceeded 0.1σ and a difference map revealed no significant features. Final $\underline{R}$ was 0.037 and $\underline{R}_w$0.045.* Scattering factors were for the neutral atoms (10,11) with anomalous dispersion corrections being applied for Fe and P (12); no correction was applied for absorption. Results are presented in Tables I - III.**

RESULTS AND DISCUSSION

The loss of the β vinylic chlorine atom enables the ring to σ-bond through C(2) to Fe(2) to complete a four membered chelate ring. The Fe(2)-C(2) bonding distance of 1.938(5)Å is comparable with Fe-C σ-bonds in related compounds (13-15).

In addition a γ fluorine atom is displaced, creating a π-allyl system through C(1), C(2) and C(3) which enables the group to π-bond to Fe(1). The bonds in the C(3)-C(4)-C(5)-C(1) portion of the ring are considerably longer than the C(1)-C(2)-C(3) region showing that the π-allyl electron delocalization is largely confined to the latter. Moreover the C(2)-C(3) bond (1.393(7)Å) is significantly shorter than C(1)-C(2) (1.447(7)Å) suggesting that delocalization is not complete and that C(2)-C(3) possesses greater double bond character than C(1)-C(2). As the 3-electron π-bonding between the π-allyl segment of the fluorocarbon ring and Fe(1) can be regarded as a combination of bonding modes A and B, A would be expected to be the major contributor. The greater σ nature of

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* $\underline{R} = \Sigma||\underline{F}_o|-|\underline{F}_c||/\Sigma|\underline{F}_o|$

$\underline{R}_w = (\Sigma \underline{w}(|\underline{F}_o|-|\underline{F}_c|)^2/\Sigma|\underline{F}_o|^2)^{\frac{1}{2}}$

** The table of structure factors has been deposited as NAPS Document No. 03277. Order from ASIS,NAPS, c/o Microfiche Publications, P.O. Box 3513, Grand Central Station, New York, N.Y. 10017. Price on request. Advance payment is required

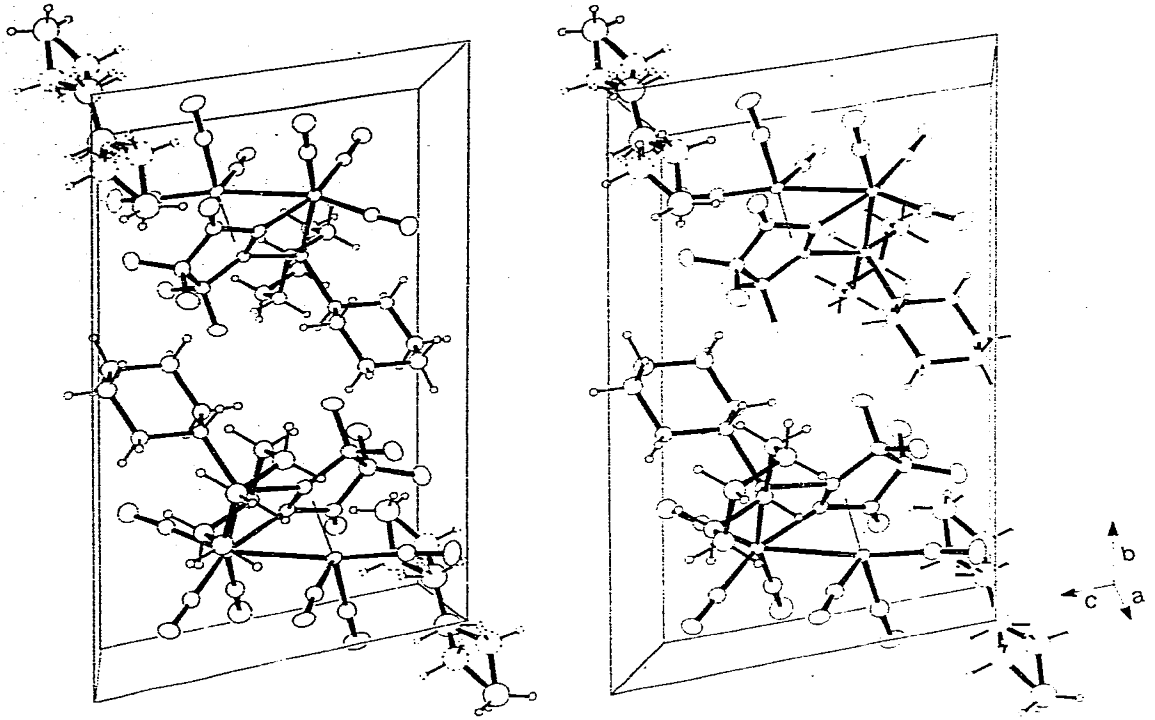
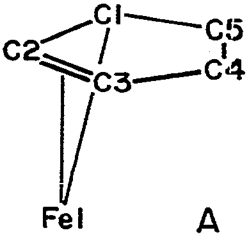
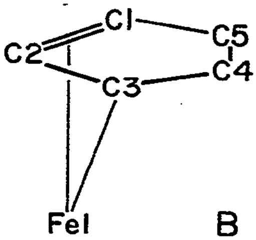


Figure 2
Stereoscopic drawing (20% ellipsoids) showing molecular packing within the unit cell. Disordered atoms are shown with broken outlines and hydrogen atoms have an arbitrary radius of 0.1Å.





Fe-C(1) would cause it to be shorter than Fe-C(3), which is in agreement with the crystallographic results (2.083(5) c.f. 2.121(5)Å).

The eclipsed configuration of the carbonyls on the iron atoms is probably a consequence of the bridging nature of the fluorocarbon. This feature has been observed in several related compounds with bridging ligands (13,16). The Fe-Fe distance of 2.668(2)Å is intermediate in the range 2.51-2.87Å

(Continued on p. 268)

TABLE I

Final atomic fractional cell and thermal ($\times 10^3$) parameters with least-squares estimated standard deviations in parentheses. The thermal parameters are in the form $\exp[-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{23}klb^*c^*)]$

Atom	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	0.11776(7)	0.14805(4)	0.35651(7)	46.4(6)	31.3(6)	39.3(6)	0.7(3)	17.4(4)	1.0(3)
Fe(2)	0.23581(7)	0.19039(4)	0.64615(7)	40.9(6)	31.9(6)	38.8(6)	4.9(3)	15.0(3)	6.0(3)
P	0.08738(12)	0.29883(7)	0.61641(13)	39.4(8)	28.7(8)	37.8(8)	2.4(5)	14.0(6)	2.9(5)
C(1)	0.1252(5)	0.2817(3)	0.4366(5)	46(3)	35(3)	35(3)	1(2)	17(2)	4(2)
C(2)	0.2503(5)	0.2414(3)	0.4756(5)	43(3)	29(3)	41(3)	-5(2)	18(2)	-1(2)
C(3)	0.2887(5)	0.2233(3)	0.3411(6)	47(3)	35(3)	54(4)	-1(3)	27(3)	4(2)
C(4)	0.2427(6)	0.2903(4)	0.2469(7)	75(4)	51(4)	51(4)	-9(3)	26(3)	9(3)
C(5)	0.1250(6)	0.3288(4)	0.3105(6)	68(4)	37(4)	51(3)	6(3)	15(3)	13(3)
F(3A)	0.4038(3)	0.1873(2)	0.3238(4)	59(2)	69(2)	73(2)	6(2)	39(2)	7(2)
F(4A)	0.3431(4)	0.3533(3)	0.2626(5)	96(3)	72(3)	96(3)	-21(2)	44(2)	30(2)
F(4B)	0.2042(4)	0.2584(2)	0.0988(4)	102(3)	82(3)	46(2)	4(2)	35(2)	19(2)
F(5A)	0.1501(4)	0.4172(2)	0.3561(4)	119(3)	34(2)	70(2)	5(2)	31(2)	16(2)
F(5B)	0.0090(4)	0.3198(2)	0.2047(4)	81(3)	68(2)	53(2)	18(2)	10(2)	22(2)
C(11)	0.1895(6)	0.0460(4)	0.3143(6)	60(4)	44(4)	56(4)	-4(3)	23(3)	1(3)
C (12)	0.0188(6)	0.1386(4)	0.1671(7)	57(3)	54(4)	51(4)	-2(3)	25(3)	0(3)
C(13)	-0.0160(6)	0.1033(3)	0.4224(6)	55(3)	30(3)	51(3)	4(3)	20(3)	1(2)
C(21)	0.3554(6)	0.1122(4)	0.6154(6)	71(4)	50(4)	48(3)	11(3)	26(3)	8(3)
C(22)	0.3508(6)	0.2486(4)	0.8088(7)	45(3)	47(3)	52(4)	5(3)	17(3)	14(3)
C(23)	0.1497(5)	0.1252(3)	0.7442(6)	53(3)	36(3)	46(3)	9(3)	16(3)	7(3)
O(11)	0.2367(5)	-0.0172(3)	0.2808(5)	93(3)	48(3)	102(4)	19(3)	39(3)	-6(2)

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(12)	-0.0441(5)	0.1267(3)	0.0496(5)	81(3)	114(4)	45(3)	-8(3)	9(2)	-3(2)
O(13)	-0.1032(4)	0.0717(2)	0.4575(4)	62(2)	40(2)	67(3)	-7(2)	29(2)	4(3)
O(21)	0.4320(5)	0.0630(3)	0.5988(5)	86(3)	80(3)	90(3)	47(3)	31(3)	10(3)
O(22)	0.4284(4)	0.2829(3)	0.9128(5)	63(3)	72(3)	62(3)	-6(2)	-3(2)	7(2)
O(23)	0.0991(4)	0.0826(3)	0.8054(5)	87(3)	53(3)	67(3)	1(2)	37(2)	19(2)

Atom	x	y	z	U	Atom	x	y	z	U
C(51)	-0.0976(5)	0.2876(3)	0.5957(5)	40(1)	C(61)	0.1471(5)	0.4107(3)	0.7179(5)	38(1)
C(52)	-0.1645(5)	0.3699(4)	0.5654(6)	48(1)	C(62)	0.1376(5)	0.4191(3)	0.8805(5)	44(1)
C(53)	-0.3169(6)	0.3524(4)	0.5245(6)	60(2)	C(63)	0.1774(6)	0.5117(4)	0.9660(6)	56(1)
C(54)	-0.3645(6)	0.3149(4)	0.6449(6)	59(2)	C(64)	0.3163(6)	0.5433(4)	0.9561(7)	63(2)
C(55)	-0.2958(6)	0.2343(4)	0.6777(7)	62(2)	C(65)	0.3256(5)	0.5350(4)	0.7962(6)	54(1)
C(56)	-0.1436(5)	0.2514(4)	0.7193(6)	53(1)	C(66)	0.2882(5)	0.4424(4)	0.7102(6)	48(1)
C(91)	0.6648(9)	0.1808(6)	0.1411(10)	109(3)	C(92)P[b]	0.6257(43)	0.1032(28)	0.0270(50)	107(12)
C(92)[a]	0.6335(12)	0.0863(8)	0.1236(15)	111(4)	C(93)	0.5159(11)	0.0474(6)	0.0184(12)	120(3)
H(51A)	-0.1190	0.2448	0.5073		H(61A)	0.0834	0.4453	0.6684	
H(52A)	-0.1371	0.3891	0.4857		H(62A)	0.1952	0.3809	0.9263	
H(52B)	-0.1387	0.4141	0.6524		H(62B)	0.0484	0.4037	0.8818	
H(53A)	-0.3433	0.3121	0.4333		H(63A)	0.1761	0.5131	1.0671	
H(53B)	-0.3567	0.4057	0.5138		H(63B)	0.1143	0.5496	0.9252	
H(54A)	-0.4574	0.2998	0.6121		H(64A)	0.3802	0.5092	1.0061	
H(54B)	-0.3466	0.3578	0.7331		H(64B)	0.3350	0.6029	1.0042	

Atom	x	y	z	Atom	x	y	z
H(55A)	-0.3204	0.1898	0.5926	H(65A)	0.4152	0.5514	0.7955
H(55B)	-0.3228	0.2165	0.7589	H(65B)	0.2682	0.5734	0.7503
H(56A)	-0.1046	0.1980	0.7318	H(66A)	0.3501	0.4046	0.7513
H(56B)	-0.1171	0.2923	0.8102	H(66B)	0.2896	0.4412	0.6090
H(91A)	0.7166	0.1992	0.0802	H(92A)P[b]	0.6987	0.0683	0.0352
H(91B)	0.7126	0.1925	0.2400	H(92B)P[b]	0.6088	0.1222	-0.0663
H(91C)	0.5858	0.2112	0.1305	H(93A)[a]	0.5186	0.0658	-0.0719
H(92A)[a]	0.6270	0.0748	0.2183	H(93B)[a]	0.4439	0.0715	0.0546
H(92B)[a]	0.7063	0.0578	0.0959	H(93A)P[b]	0.4473	0.0684	-0.0493
				H(93B)P[b]	0.5018	0.0616	0.1163

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[a] Population parameter 0.78

[b] Population parameter 0.22

TABLE II

Interatomic distances (Å) and angles (deg.), with least-squares e.s.d's in parentheses. For C/F and C/O the values corrected for thermal motion follow the uncorrected values.

Fe(1) - Fe(2)	2.668(2)	C(11) - O(11)	1.141,1.182(7)
Fe(1) - C(1)	2.083(5)	C(12) - O(12)	1.125,1.170(7)
Fe(1) - C(2)	1.955(5)	C(13) - O(13)	1.144,1.161(6)
Fe(1) - C(3)	2.121(5)	C(21) - O(21)	1.134,1.180(7)
Fe(1) - C(11)	1.797(6)	C(22) - O(22)	1.144,1.177(7)
Fe(1) - C(12)	1.813(7)	C(23) - O(23)	1.133,1.163(6)
Fe(1) - C(13)	1.794(6)	C(51) - C(52)	1.517(7)
Fe(2) - P	2.324(2)	C(52) - C(53)	1.524(8)
Fe(2) - C(2)	1.938(5)	C(53) - C(54)	1.521(8)
Fe(2) - C(21)	1.794(6)	C(54) - C(55)	1.504(9)
Fe(2) - C(22)	1.781(7)	C(55) - C(56)	1.522(8)
Fe(2) - C(23)	1.814(6)	C(56) - C(51)	1.531(7)
P - C(1)	1.796(5)	C(61) - C(62)	1.536(7)
P - C(51)	1.860(5)	C(62) - C(63)	1.519(8)
P - C(61)	1.847(5)	C(63) - C(64)	1.519(8)
C(1) - C(2)	1.447(7)	C(64) - C(65)	1.510(8)
C(2) - C(3)	1.393(7)	C(65) - C(66)	1.518(8)
C(3) - C(4)	1.502(8)	C(66) - C(61)	1.534(7)
C(4) - C(5)	1.559(8)	C(91) - C(92)	1.47(1)
C(5) - C(1)	1.496(7)	C(92) - C(93)	1.41(2)
C(3) - F(3A)	1.356,1.376(6)	C(93) - C(93)[a]	1.47(2)
C(4) - F(4A)	1.362,1.391(7)	C(91) - C(92)P	1.45(4)
C(4) - F(4B)	1.354,1.371(7)	C(92)P- C(93)	1.38(4)
C(5) - F(5A)	1.368,1.392(7)		
C(5) - F(5B)	1.350,1.367(7)	C(92)....C(92)P	0.98(4)

Fe(2) - Fe(1) - C(11)	99.0(2)	C(4) - C(3) -F(3A)	115.8(4)
Fe(2) - Fe(1) - C(12)	168.4(2)	C(3) - C(4) -F(4A)	111.1(5)
Fe(2) - Fe(1) - C(13)	83.3(2)	C(3) - C(4) -F(4B)	114.6(5)
C(11) - Fe(1) - C(12)	92.6(3)	C(5) - C(4) -F(4A)	110.9(5)
C(11) - Fe(1) - C(13)	95.5(2)	C(5) - C(4) - F(4B)	112.3(5)
C(12) - Fe(1) - C(13)	94.4(2)	F(4A)- C(4) - F(4B)	105.1(4)
Fe(1) - Fe(2) - P	78.31(5)	C(1) - C(5) - F(5A)	112.8(4)
Fe(1) - Fe(2) -C(2)	47.0(1)	C(1) - C(5) - F(5B)	115.9(4)
Fe(1) - Fe(2) -C(21)	88.0(2)	C(4) - C(5) - F(5A)	109.8(4)
Fe(1) - Fe(2) -C(22)	156.6(2)	C(4) - C(5) - F(5B)	111.8(4)
Fe(1) - Fe(2) -C(23)	107.9(2)	F(5A)- C(5) - F(5B)	103.9(4)

P - Fe(2) - C(2)	69.8(1)	Fe(1) -C(11) -O(11)	176.1(6)
P - Fe(2) - C(21)	162.7(2)	Fe(1) -C(12) -O(12)	175.2(6)
P - Fe(2) - C(22)	97.0(2)	Fe(1) -C(13) -O(13)	176.4(7)
P - Fe(2) - C(23)	99.4(2)	Fe(2) -C(21) -O(21)	178.7(6)
C(2)-Fe(2) - C(21)	93.1(2)	Fe(2) -C(22) -O(22)	176.9(5)
C(2)-Fe(2) - C(22)	109.8(2)	Fe(2) -C(23) -O(23)	177.8(6)
C(2)-Fe(2) - C(23)	153.4(2)	P-C(51) - C(52)	112.8(3)
C(21)-Fe(2) - C(22)	91.4(2)	P-C(51) - C(56)	115.5(3)
C(21)-Fe(2) - C(23)	94.9(2)	C(56) - C(51) - C(52)	111.6(4)
C(22)-Fe(2) - C(23)	95.4(2)	C(51) - C(52) - C(53)	110.3(4)
Fe(2) - P - C(1)	80.4(2)	C(52) - C(53) - C(54)	110.9(5)
Fe(2) - P - C(51)	127.1(2)	C(53) - C(54) - C(55)	112.0(5)
Fe(2) - P - C(61)	117.5(2)	C(54) - C(55) - C(56)	111.7(5)
C(1) - P - C(51)	110.1(2)	C(55) - C(56) - C(51)	109.7(4)
C(1) - P - C(61)	110.4(2)	P - C(61) - C(62)	109.7(3)
C(51) - P - C(61)	107.0(2)	P - C(61) - C(66)	115.4(3)
C(5) - C(1) - C(2)	110.5(4)	C(66) - C(61) - C(62)	110.5(4)
C(1) - C(2) - C(3)	103.2(4)	C(61) - C(62) - C(63)	111.5(4)
C(2) - C(3) - C(4)	110.9(4)	C(62) - C(63) - C(64)	111.4(5)
C(3) - C(4) - C(5)	103.0(4)	C(63) - C(64) - C(65)	111.3(5)
C(4) - C(5) - C(1)	102.7(4)	C(64) - C(65) - C(66)	112.1(5)
P - C(1) - C(2)	98.1(3)	C(65) - C(66) - C(61)	110.6(4)
P - C(1) - C(5)	139.9(4)	C(91) - C(92) - C(93)	118(1)
Fe(2) - C(2) - C(1)	142.0(4)	C(92) - C(93) - C(93)[a]	122(1)
Fe(2) - C(2) - C(3)	104.3(3)	C(91) - C(92)P- C(93)	122(3)
C(2) - C(3) - F(3A)	124.6(5)	C(92)P- C(93) - C(93)[a]	135(2)

[a] Atom at (1-x, -y, -z)

reported for Fe-Fe bonds in iron carbonyl complexes with ligands containing phosphorus or arsenic (17).

The structural feature that seems to be necessary for the formation of the π-allyl systems in II and III is the grouping $R_2E-C=C(Z)-CF_2$ but it is difficult to account for the normal reaction of $(C_6H_{11})_2P\overline{C = CCl(C}F_2)_2$. Reactions of other tertiary phosphines and arsines of this type are being investigated.

In related studies Carty and co-workers (15,18) have found that the alkrynyl derivatives $R_2EC{\equiv}CCF_3$ give unusual clusters on reaction with iron carbonyls but loss of a fluorine atom was not observed.

TABLE III

Deviations of atoms (Å) from least-squares planes. The equations of the planes are referred to the orthogonal axes, *a*,*b*' and *c**

a) Plane defined by C(1), C(2) and C(3)

$-0.4366x - 0.8748y - 0.2102z + 4.1037 = 0$

C(1)	0.0	Fe(2)	0.837	C(4)	-0.740
C(2)	0.0	P	0.034	C(5)	-0.696
C(3)	0.0	F(3A)	-0.042		

b) Plane defined by C(1), C(3), C(4) and C(5)

$-0.5038x - 0.5942y - 0.6270z + 4.7178 = 0$ $\chi^2=27.1$

C(1)	-0.009	P	-0.605	F(4A)	-1.160
C(3)	0.010	C(2)	-0.432	F(4B)	0.992
C(4)	-0.019	F(3A)	-0.200	F(5A)	-1.068
C(5)	0.017			F(5B)	1.072

Dihedral angle between planes a and b is 29.4°.

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