Crystal Structure of Tetracarbonyl-[1-(dimethylarsino)-2-(diphenylphosphino)tetrafluorocyclobutene-*P*]-iron

By **F. W. B. Einstein** * and **R. D. G. Jones**, Department of Chemistry, Simon Fraser University, Burnaby 2, British Columbia, Canada

The title compound crystallizes in the monoclinic space group $P2_1/c$ and has a unit cell of dimensions: a = 11·284(4), b = 10·800(6), c = 19·755(8) Å, β = 96·85(2)°, Z = 4. By use of three-dimensional data collected by counter methods, the structure was solved by the symbolic addition procedure and refined by full-matrix least squares to R 0·040 for 1149 observed reflections. The co-ordination geometry about the iron atom is a slightly distorted trigonal bipyramid. The ligand co-ordinates through the phosphorus atom in an axial position, Fe–P being 2·224(3) Å. There are no significant differences between axial and equatorial iron–carbon bond lengths, mean 1·74(2) Å. The arsenic atom lies above a phenyl ring with As–C contacts ranging from 3·44—4·19 Å.

WHETHER there is a difference between the lengths of the equatorial and axial iron–carbon bonds in iron pentacarbonyl has been a matter of discussion for some time. A. W. Hanson [1] was unable to detect any significant difference in bond lengths with a somewhat limited set of observations. Using the same data but another space group, Donohue and Caron came to a similar conclusion.[2] Later, an electron-diffraction study of the gas phase by Davis and H. P. Hanson indicated that the axial bond lengths were shorter than the equatorial.[3] Donohue and Caron [4] suggested the electron-diffraction study was subject to error and further communications by them [5] and Davis and Hanson [6] did not appear to resolve the matter. Recently, an exhaustive electron-diffraction study by Beagley *et al.*[7] has shown that there is a very high probability that the axial bond lengths are very slightly shorter in the gas phase.

An *X*-ray crystallographic study of $Fe(CO)_4$(asp) [asp = 1-(dimethylarsino)-2-(diphenylphosphino)tetrafluorocyclobutene] has been carried out to find if any differences in iron–carbon distances can be detected between the axial and equatorial bonds and to confirm that co-ordination had occurred through the phosphorus atom.

EXPERIMENTAL

The crystals were yellow plates, elongated in the b direction. The space group was determined from Weissenberg photographs of zones $h0l$—$h2l$ and precession photographs of zones $hk0$, 0—$1kl$, taken with Cu-K_α radiation (λ = 1·5418 Å).

Crystal Data.—$C_{22}H_{16}AsF_4FeO_4P$, M = 581·9, Monoclinic, a = 11·284(4), b = 10·800(6), c = 19·755(8) Å, β = 96·85(2)°, U = 2390·3 Å^3, D_m = 1·55 (Berman density balance), Z = 4, D_c = 1·62 g cm^{-3}, $F(000)$ = 1160. Mo-K_{α_1} radiation, λ = 0·70926 Å, μ(Mo-K_α) = 22·3 cm^{-1}. Space group $P2_1/c$ from systematic absences: $h0l$ when l is odd, $0k0$ when k is odd.

A crystal, cleaved to dimensions 0·02 × 0·016 × 0·03 cm was used for the measurement of cell dimensions and for data collection. In order to minimize intrinsic multiple reflections, the b^* axis was offset *ca.* 6° from the ϕ axis of the diffractometer. Cell dimensions, along with the estimated standard deviations, and the orientation matrix were determined from the least-squares analysis of the 2θ, ω, and χ angles for 30 high-angle reflections; the accurate centring of reflections and the measurement of the angles was done on a computer-controlled, Picker four-circle diffractometer by use of Mo-K_{α_1} radiation,[8] with a take-off angle of 1° at 23·5 °C.

Intensity data were collected by the θ—2θ scan technique by use of Mo-K_α radiation (niobium filtered) and a scintillation counter equipped with a pulse-height analyser. The take-off angle was 3°. Each reflection was scanned for 1·4° (extended for the $\alpha_1 - \alpha_2$ splitting) at a scan rate of 2° min^{-1}. For reflections with $2\theta \leq 25°$, a stationary background count was taken for 10 s at each end of the scan range and for, those with $25° < 2\theta \leq 40°$, it was taken for 20 s. Every 50 reflections, two standard reflections were scanned. The maximum deviation of any individual standard from the mean was 2·5%. In this manner, intensities for 2244 reflections with $2\theta \leq 40°$ were measured.

Intensities were corrected for background (normalized to the scan time) and Lorentz-polarization factors. An absorption correction was made by considering the crystal to be approximately a cylinder of radius 0·009 cm, μR 0·20. The maximum error in intensity introduced by this approximation is 4%. If the net count for a reflection was $< 1{\cdot}8\sigma$ [where $\sigma = (\text{total count} + \text{background})^{\frac{1}{2}}$] the reflection was considered unobserved. The number of observed reflections was 1149.

Structure Determination.—The symbolic addition procedure [9] was used to initiate the solution of the structure. *E*-Values were determined and five reflections were assigned signs; three reflections were used to fix the origin ($\bar{3}$, 8, 6, E = +3·35; 0, 3, 3, E = +2·99; and 1, 5, 2, E = 2·59) while the signs of the other two (0, 6, 6, E = +2·49; $\bar{6}$, 0, 12, E = +2·41) were deduced from Sayre relationships.[10] Initially the signs of reflections with $E \geq 1{\cdot}8$ were determined and later the process was extended to give signs for 305 reflections with $E \geq 1{\cdot}5$. From an F_o Fourier map with these 305 reflections, the positions of the arsenic, iron, and phosphorus atoms were located and, when the

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[1] A. W. Hanson, *Acta Cryst.*, 1962, **15**, 930.

[2] J. Donohue and A. Caron, *Acta Cryst.*, 1964, **17**, 663.

[3] M. I. Davis and H. P. Hanson, *J. Phys. Chem.*, 1965, **69**, 3405.

[4] J. Donohue and A. Caron, *J. Phys. Chem.*, 1966, **70**, 603.

[5] J. Donohue and A. Caron, *J. Phys. Chem.*, 1967, **71**, 777.

[6] M. I. Davis and H. P. Hanson, *J. Phys. Chem.*, 1967, **71**, 775.

[7] B. Beagley, D. W. J. Cruickshank, P. M. Pinder, A. G. Robiette, and G. M. Sheldrick, *Acta Cryst.*, 1969, *B*, **25**, 737.

[8] Data collection programs were those of P. G. Lenhert; Symbolic Addition Programs were those of F. Ahmed; all other programs are listed in F. W. B. Einstein and R. D. G. Jones, *J. Chem. Soc.* (*A*), 1971, 3359.

[9] J. Karle and I. L. Karle, *Acta Cryst.*, 1966, **21**, 849.

[10] D. Sayre, *Acta Cryst.*, 1952, **5**, 60.

co-ordinates and isotropic temperature factors for these atoms were refined by full-matrix least-squares techniques; R was 0·35. Reflections were assigned unit weight and the function minimized was $\Sigma w(|F_o| - |F_c|)^2$.

Electron-density difference syntheses allowed the location of all non-hydrogen atoms and indicated anisotropic motion for the arsenic atom. Further refinement resulted in R 0·068 and R' 0·079 {where $R' = [\Sigma w(||F_o| - |F_c||)^2/\Sigma wF_o^2]^{\frac{1}{2}}$}. An electron-density difference map showed that the iron and fluorine atoms, C(4), C(5), and O(33) had anisotropic motion and permitted assignment of the phenyl hydrogen positions, which were included in subsequent least-squares cycles but not refined. With changes to allow anisotropic thermal motion for the above-mentioned atoms, further refinement caused convergence at R 0·040 and R' 0·048. The maximum shift in any parameter in the final refinement cycle was 0·04σ. The weighting scheme used after all non-hydrogen atoms were located was $\sigma = A$ for $B \leq F_o \leq C$, $\sigma = (A^2B/F_o)^{\frac{1}{2}}$ for $B > F_o$, and $\sigma = (A^2F_o/C)^{\frac{1}{2}}$ for $C < F_o$, where A is a constant which reduces the error-of-fit to unity. Final values for the weighting-scheme parameters were: $A = 2{\cdot}36$, $B = 40{\cdot}0$, and $C = 50{\cdot}0$; error-of-fit was 0·97. At the conclusion of the refinement, the major features in an electron-density difference synthesis were peaks near C(4) and C(5) of height 0·3 eA^{-3}.

Scattering factors for the non-hydrogen atoms were

TABLE 1

Positional (fractional co-ordinates) and thermal parameters (Å^2) with estimated standard deviations in parentheses

Atom	x	y	z	B_{iso}
As(1)	0·2897(2)	0·0121(1)	0·3242(1)	*
Fe(2)	0·1235(1)	−0·2361(1)	0·0688(1)	*
P(3)	0·2672(3)	−0·1418(3)	0·1367(1)	2·93(6)
C(4)	0·151(2)	−0·007(2)	0·3706(11)	*
C(5)	0·405(2)	−0·027(2)	0·4049(9)	*
C(6)	0·287(1)	−0·156(1)	0·2860(5)	3·8(2)
C(7)	0·281(1)	−0·202(1)	0·2228(5)	2·9(2)
C(8)	0·287(1)	−0·333(1)	0·2482(6)	4·5(3)
C(9)	0·293(1)	−0·281(1)	0·3200(6)	4·2(3)
F(10)	0·3806(8)	−0·4015(6)	0·2354(4)	*
F(11)	0·1896(7)	−0·4063(7)	0·2302(4)	*
F(12)	0·3916(7)	−0·3072(7)	0·3611(4)	*
F(13)	0·2006(7)	−0·3094(7)	0·3542(4)	*
C(14)	0·245(1)	0·023(1)	0·1488(5)	3·1(2)
C(15)	0·135(1)	0·066(1)	0·1584(6)	5·0(3)
C(16)	0·115(1)	0·192(1)	0·1710(7)	6·1(3)
C(17)	0·206(1)	0·274(1)	0·1695(6)	5·7(3)
C(18)	0·317(1)	0·235(1)	0·1599(6)	6·2(3)
C(19)	0·339(1)	0·108(1)	0·1487(6)	4·7(3)
C(20)	0·417(1)	−0·155(1)	0·1143(5)	3·2(2)
C(21)	0·516(1)	−0·150(1)	0·1636(6)	4·6(3)
C(22)	0·631(1)	−0·151(1)	0·1427(7)	5·4(3)
C(23)	0·647(1)	−0·162(1)	0·0764(7)	5·6(3)
C(24)	0·551(1)	−0·169(1)	0·0286(6)	5·7(3)
C(25)	0·435(1)	−0·167(1)	0·0465(6)	4·8(3)
C(26)	0·128(1)	−0·115(1)	0·0109(6)	4·5(3)
O(27)	0·130(1)	−0·033(1)	−0·0281(4)	6·1(2)
C(28)	0·215(1)	−0·369(1)	0·0705(6)	5·4(3)
O(29)	0·275(1)	−0·455(1)	0·0722(4)	7·2(2)
C(30)	0·028(1)	−0·223(1)	0·1293(7)	5·7(3)
O(31)	−0·041(1)	−0·209(1)	0·1702(5)	7·9(3)
C(32)	0·013(1)	−0·311(1)	0·0145(7)	5·6(3)
O(33)	−0·060(1)	−0·361(1)	−0·0215(5)	*
H(15)	0·070	0·004	0·158	8·0
H(16)	0·033	0·223	0·179	8·0
H(17)	0·194	0·361	0·176	8·0
H(18)	0·379	0·296	0·159	8·0
H(19)	0·417	0·079	0·141	8·0
H(21)	0·504	−0·143	0·211	8·0
H(22)	0·702	−0·146	0·177	8·0
H(23)	0·730	−0·166	0·064	8·0
H(24)	0·567	−0·176	−0·174	8·0
H(25)	0·365	−0·173	0·012	8·0

* Anisotropic temperature parameters in the form: $\exp -[2\pi(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + 2U_{12}a^*b^*hk + 2U_{13}a^*c^*hl + 2U_{23}b^*c^*kl)]$ where the parameters are as follows:

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
As(1)	0·167(2)	0·0507(9)	0·0527(8)	0·001(1)	0·0202(9)	−0·0067(9)
Fe(2)	0·046(1)	0·048(1)	0·0490(9)	−0·012(1)	−0·0016(8)	0·0013(9)
F(10)	0·132(7)	0·069(5)	0·083(5)	0·053(5)	0·023(5)	0·011(4)
F(11)	0·131(7)	0·067(5)	0·086(5)	−0·045(5)	−0·024(5)	0·019(4)
F(12)	0·088(5)	0·095(6)	0·072(5)	0·024(5)	−0·026(4)	0·010(4)
F(13)	0·089(5)	0·095(6)	0·084(5)	−0·005(5)	0·040(4)	0·025(4)
O(33)	0·130(9)	0·117(9)	0·106(8)	−0·058(8)	−0·052(7)	0·011(7)
C(4)	0·19(2)	0·15(2)	0·21(2)	0·03(2)	0·15(2)	−0·01(2)
C(5)	0·21(2)	0·09(1)	0·11(1)	0·01(1)	−0·07(1)	−0·04(1)

taken from ref. 11, for hydrogen from ref. 12, and anomalous dispersion corrections for the arsenic and iron atoms from ref. 13.

RESULTS

Final parameters for all the atoms are listed in Table 1. Table 2 contains interatomic distances and angles. Errors in these distances and angles include contributions from the variance-covariance matrix and errors in cell dimensions. The equations of selected planar atom-groups are given in Table 3.

TABLE 2

Molecular geometry with estimated standard deviations in parentheses

(*a*) Distances (Å)

As(1)–C(4)	1·98(2) [2·00(2) *]
As(1)–C(5)	1·98(2) [2·03(2) *]
As(1)–C(6)	1·96(1)
Fe(2)–P(3)	2·224(3)
Fe(2)–C(26)	1·74(1)
Fe(2)–C(28)	1·76(1)
Fe(2)–C(30)	1·71(1)
Fe(2)–C(32)	1·74(2)
C(26)–O(27)	1·18(1)
C(28)–O(29)	1·14(1)
C(30)–O(31)	1·19(1)
C(32)–O(33)	1·16(1)
P(3)–C(7)	1·81(1)
P(3)–C(14)	1·82(1)
P(3)–C(20)	1·80(1)
C(14)–C(15)	1·37(1)
C(15)–C(16)	1·41(2)
C(16)–C(17)	1·36(2)
C(17)–C(18)	1·35(2)
C(18)–C(19)	1·42(2)
C(19)–C(14)	1·39(1)
C(20)–C(21)	1·39(1)
C(21)–C(22)	1·42(2)
C(22)–C(23)	1·35(2)
C(23)–C(24)	1·35(2)
C(24)–C(25)	1·40(2)
C(25)–C(20)	1·38(1)
C(6)–C(7)	1·34(1)
C(7)–C(8)	1·51(2)
C(8)–C(9)	1·50(2)
C(9)–C(6)	1·52(2)
C(8)–F(10)	1·34(1)
C(8)–F(11)	1·37(1)
C(9)–F(12)	1·33(1)
C(9)–F(13)	1·34(1)
As(1) ··· C(14)	3·44
As(1) ··· C(15)	3·57
As(1) ··· C(16)	3·92
As(1) ··· C(17)	4·19
As(1) ··· C(18)	4·08
As(1) ··· C(19)	3·72

* Corrected for riding motion.

(*b*) Interatomic angles (deg.)

C(4)–As(1)–C(5)	95·1(10)
C(4)–As(1)–C(6)	96·2(7)
C(5)–As(1)–C(6)	95·4(6)
P(3)–Fe(2)–C(26)	88·6(4)
P(3)–Fe(2)–C(28)	88·7(4)
P(3)–Fe(2)–C(30)	91·0(4)
P(3)–Fe(2)–C(32)	178·8(9)
C(26)–Fe(2)–C(28)	123·9(6)
C(26)–Fe(2)–C(30)	117·8(6)
C(26)–Fe(2)–C(32)	91·0(6)
C(28)–Fe(2)–C(30)	118·3(6)
C(28)–Fe(2)–C(32)	90·6(6)
C(30)–Fe(2)–C(32)	90·2(6)
Fe(2)–C(26)–O(27)	179(1)
Fe(2)–C(28)–O(29)	179(2)
Fe(2)–C(30)–O(31)	177(1)
Fe(2)–C(32)–O(33)	180(4)
Fe(2)–P(3)–C(7)	112·3(3)
Fe(2)–P(3)–C(14)	115·3(3)
Fe(2)–P(3)–C(20)	116·9(3)
C(7)–P(3)–C(14)	103·0(5)
C(7)–P(3)–C(20)	102·8(5)
C(14)–P(3)–C(20)	104·9(5)
C(19)–C(14)–C(15)	119(1)
C(14)–C(15)–C(16)	121(1)
C(15)–C(16)–C(17)	120(1)
C(16)–C(17)–C(18)	121(1)
C(17)–C(18)–C(19)	120(1)
C(18)–C(19)–C(14)	119(1)
C(25)–C(20)–C(21)	119(1)
C(20)–C(21)–C(22)	119(1)
C(21)–C(22)–C(23)	121(1)
C(22)–C(23)–C(24)	120(1)
C(23)–C(24)–C(25)	121(1)
C(24)–C(25)–C(20)	120(1)
C(9)–C(6)–C(7)	94·4(9)
C(6)–C(7)–C(8)	92·5(9)
C(7)–C(8)–C(9)	87·5(9)
C(8)–C(9)–C(6)	85·6(9)
C(7)–C(8)–F(10)	117(1)
C(7)–C(8)–F(11)	117(1)
C(9)–C(8)–F(10)	116(1)
C(9)–C(8)–F(11)	114(1)
F(10)–C(8)–F(11)	105(1)
C(6)–C(9)–F(12)	117(1)
C(6)–C(9)–F(13)	116(1)
C(8)–C(9)–F(12)	116(1)
C(8)–C(9)–F(13)	116(1)
F(12)–C(9)–F(13)	107(1)

TABLE 3

(*a*) Least-squares planes, referred to orthogonal axes along *a*, *b*, and *c**, of the form $AX + BY + CZ + D = 0$ where X, Y, Z, and D are in Å.

	A	B	C	D	χ^2
Plane (I): C(14)—(19)	−0·1236	0·1325	−0·9834	3·100	4·4
Plane (II): C(20)—(25)	0·0016	0·9967	−0·0817	1·855	2·2
Plane (III): As(1), P(3), C(6)—(9)	−0·9986	−0·0173	−0·0495	2·529	6·0
Plane (IV): C(26), O(27), C(28), O(29), C(30), O(31)	−0·6880	−0·4420	−0·5986	0·4465	2·3

Fe(2) lies 0·025 Å out of Plane (IV) towards C(32).

(*b*) Dihedral angles (deg.) between planes

(I)–(II)	77·7	(II)–(III)	90·8
(I)–(III)	80·2	(II)–(IV)	66·9
(I)–(IV)	52·2	(III)–(IV)	45·2

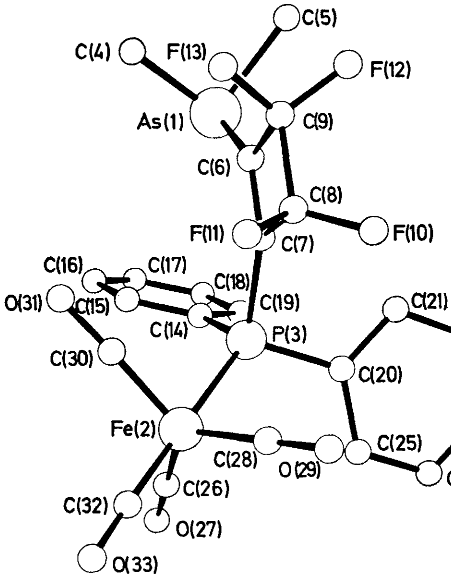

FIGURE 1 A diagram of the molecule showing the numbering used

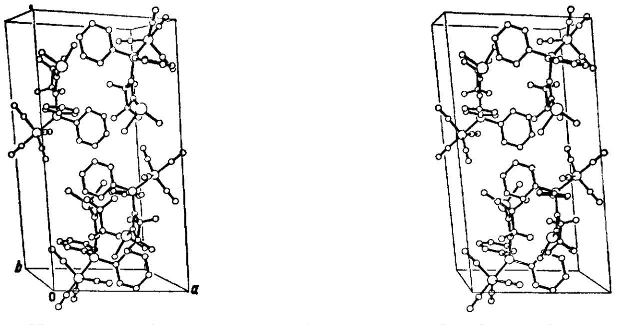

FIGURE 2 A stereoscopic view of the molecular packing

Figure 1 is a diagram of the molecule and indicates the numbering used. Figure 2 illustrates the packing in the unit cell.

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[11] D. T. Cromer and J. T. Waber, *Acta Cryst.*, 1965, **18**, 104.

[12] R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

[13] 'International Tables for *X*-ray Crystallography,' vol. III, 1962, Kynoch Press, Birmingham, p. 213.

Observed and calculated structure factors listed in Supplementary Publication No. SUP 20266 (4 pp., 1 microfiche).*

DISCUSSION

As indicated by other physical methods,[14] the co-ordination geometry about iron is a slightly, but significantly, distorted trigonal bipyramid with the non-carbonyl ligand in an axial position. The angles C(26)–Fe(2)–C(28), C(26)–Fe(2)–C(30), and C(28)–Fe(2)–C(30) are 123·9(6), 117·8(6), and 118·3(6)°. This distortion may result in the opening of the angle C(26)–Fe(2)–C(28) from 120° to minimize the contact of C(20) and H(25) with C(26) and C(28); the final orientation adopted allows nearly equal contacts between each carbon attached to P(3) and adjacent carbonyl groups (Figure 3). In addition, C(7), C(14), and C(20) adopt a staggered conformation with respect to C(26), C(28), and C(30) (Figure 3) when these atoms are viewed along the Fe(2)–P(3) bond. Fe(2) is displaced 0·025 Å from the plane of the equatorial carbonyl groups towards C(32).

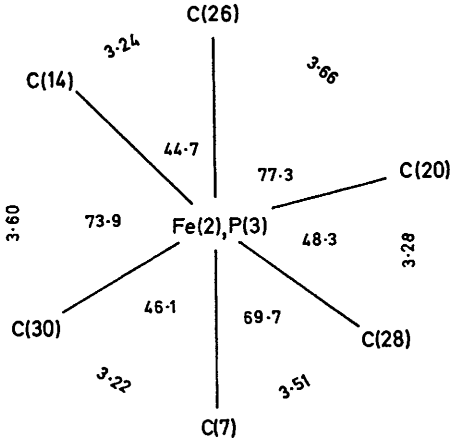

FIGURE 3 Torsion angles looking down the Fe(2)–P(3) bond. Non-bonded contacts between carbon atoms are also shown

No significant differences are observed between the Fe(2)–C(32) bond length [1·74(2) Å] and the equatorial iron–carbon bond lengths [mean 1·74(2) Å]. Since, in general, carbonyl groups bonded to metal atoms show considerable thermal motion at room temperature, any differences in bond length which did exist would be masked by the relatively high estimated standard deviations in the bond lengths. Insignificantly different iron–carbon bond lengths have been obtained for other compounds of type $Fe(CO)_4L$ [where L = diphenylphosphine,[15] (—)-fumaric acid,[16] and cumulene [17]]. However, in addition to iron pentacarbonyl (L = CO), differences between equatorial and axial iron–carbon bond lengths have been observed for the compounds where L is acrylonitrile,[18] or racemic fumaric acid,[19] and for 1,5-cyclo-octatrienebis(tetracarbonyliron) [20] and μ-tetramethyldiphosphine-bis(tetracarbonyliron).[21] In the last-mentioned compound, where phosphorus is in an axial position, the equatorial iron–carbon bond lengths are longer whereas, in the other compounds where differences were found, co-ordination was by an ethylene-type group in the equatorial position and the axial iron–carbon bond lengths were longer. Considering the accuracy of many of those structures, the difference is only clear-cut for the acrylonitrile derivative [Fe–C(*ax*) 1·99(1), Fe–C(*eq*) 1·76(1) Å], the structure of which was determined at 120 K.

In the asp ligand, phosphorus and arsenic are both potential donor atoms. However, in the present compound, the ligand is unidentate, co-ordination taking place through the phosphorus. Steric considerations would favour co-ordination from arsenic and the electronegativities indicate similar donor ability. However, phosphorus, in this ligand, in addition to the fluorocyclobutene ring, has two attached electron-withdrawing phenyl groups which would enhance π bonding from filled metal *d* orbitals to empty phosphorus *d* orbitals; the arsenic atom has two donor methyl groups. Fe(2)–P(3) is 2·224(3) Å while in $Fe(CO)_4(PHPh_2)$,[15] $Fe_3(CO)_{11}(PPh_3)$,[22] and $[Fe(CO)_4(PMe)_2]_2$ [21] the iron–phosphorus distances are 2·237(2), 2·25, and 2·260(5) Å.

Table 3 and Figure 1 show that As(1) has close contacts (3·44—4·19 Å) with one phenyl ring (*cf.* sum of van der Waals radii,[23] 3·85 Å). We suggest that these short contacts are due to an electronic interaction between the lone pair of electrons on As(1) and the π^* orbitals of the phenyl group. C–As(1)–C bond angles are all *ca.* 95° which suggest that the arsenic sp^3 orbitals used in the As–C carbon bonds have a high *p* orbital character. As a result, the arsenic lone-pair is in an orbital of high *s* orbital character which has less directional nature. Calculation shows that the lone pair is directed in the region of C(14), C(15), and C(19). Although the contact between the arsenic atom and the phenyl ring is not symmetrical, the diffuse nature of the arsenic lone pair might lead one to expect that the interaction extends over the whole ring. Within the limits of experimental error, this interaction has not affected the carbon–carbon bond lengths in the phenyl ring.

P(3)–C bond lengths are 1·80, 1·81, and 1·82(1) Å (*cf.* 1·820(7) and 1·828(5) Å in $Fe(CO)_4(PHPh_2)$ [15] and PPh_3 [24]). The angles Fe(2)–P(3)–C are larger than

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* For details of Supplementary Publications see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), Issue No. 20, 1970 (items less than 10 pp. will be supplied as full size copies).

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C–P(3)–C bond angles. Since Fe(2)–P(3) has some multiple-bond character, the angle differences are probably due to double-bond–single-bond repulsions. There are no unusual trends in the bond lengths and bond angles in either phenyl group.

Bond lengths and bond angles in the substituted cyclobutene ring show no variation from those found in complexes of 1,2-(dimethylarsino)tetrafluorocyclobutene.

Calculations.—All calculations were performed on an IBM 360 50 computer at the Simon Fraser University Computing Centre. Computer programs used are listed in ref. 8.

We thank Dr. W. R. Cullen for a gift of crystals, and the National Research Council of Canada for financial support of this work.

[1/1673 *Received, September* 13*th*, 1971]