

De fait, il est possible que la disposition *cis* soit liée à la structure du composé que nous avons utilisé pour la préparation des thioacétates pyridiné ou picolinés.

Le composé, $\text{Ni}(\text{CH}_3\text{COS})_2 \cdot 0,5\text{C}_2\text{H}_5\text{OH}$, a vraisemblablement, eu égard à ses propriétés magnétiques, la même structure que le thiobenzoate de nickel $\text{Ni}_2(\text{C}_6\text{H}_5\text{COS})_4 \cdot \text{C}_2\text{H}_5\text{OH}$ (Melson, Greene & Bryan, 1970), soit un assemblage binucléaire où les deux atomes de nickel sont pontés par les ligands thioacétate bicoordinants. Chaque 'molécule' $\text{Ni}_2(\text{C}_6\text{H}_5\text{COS})_4 \cdot \text{C}_2\text{H}_5\text{OH}$ peut donc fixer une molécule d'amine sur le site libre d'un nickel ou par substitution de $\text{C}_2\text{H}_5\text{OH}$. Mais la deuxième molécule ne peut, en raison de la structure cage, que se fixer en *cis* de la première par rupture d'une liaison Ni—O ou Ni—S, et non de façon symétrique en *trans*. Il est intéressant de noter que ce résultat, que l'on peut rapprocher des classiques effets de guidage (template effect), a été observé également par Bonamico, Dessy, Fares, Flamini & Scaramuzza (1976) pour $\text{Ni}(\text{C}_6\text{H}_5\text{CH}_2\text{CS}_2)_2 \cdot 2\text{C}_5\text{H}_5\text{N}$ préparé à partir du composé de structure binucléaire $(\text{C}_6\text{H}_5\text{CH}_2\text{CS}_2)_4\text{Ni}_2$ (Bonamico, Dessy & Fares, 1969).

En ce qui concerne enfin la disposition des atomes de soufre sur l'octaèdre de coordination, on peut remarquer que les deux formes *a* et *b* du thioacétate de nickel 2(β picoline), ne diffèrent que par la disposition *cis* ou *trans* des deux atomes de soufre, ce qui laisse penser que le changement structural correspondant correspond à une faible variation d'énergie. Toutefois, quatre thioacétates de nickel solvatés sur cinq ayant les deux atomes de S en *trans*, il est possible que cette disposi-

tion soit légèrement favorisée par un recouvrement compétitif d'une orbitale *d* du nickel par deux orbitales *d* des atomes de soufre. On peut noter dans ce sens que les distances Ni—S dans le thioacétate de nickel 2(β picoline) forme *b*, où les deux S sont en position *cis*, sont légèrement différentes l'une de l'autre (2,476 et 2,435 Å) contrairement aux autres composés. Il n'est toutefois pas exclu, bien entendu, qu'en plus des facteurs suggérés ci-dessus, des facteurs d'empilement cristallin ne jouent également un rôle pour l'obtention de ces différentes formes.

Références

- BONAMICO, M., DESSY, G. & FARES, V. (1969). *Chem. Commun.* pp. 1106–1107.
 BONAMICO, M., DESSY, G., FARES, Y., FLAMINI, A. & SCARAMUZZA, L. (1976). *J. Chem. Soc. Dalton Trans.* pp. 1743–1746.
 BOREL, M. M., GEFFROUAI, A. & LEDÉSERT, M. (1976). *Acta Cryst.* B32, 2385–2387.
 BOREL, M. M., GEFFROUAI, A. & LEDÉSERT, M. (1977*a*). *Acta Cryst.* B33, 571–574.
 BOREL, M. M., GEFFROUAI, A. & LEDÉSERT, M. (1977*b*). *Acta Cryst.* B33, 568–571.
 BOREL, M. M. & LEDÉSERT, M. (1977). *Acta Cryst.* B33, 2993–2996.
 CROMER, D. T. (1965). *Acta Cryst.* 18, 17–23.
 CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* 18, 104–109.
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A27, 368–376.
 MELSON, G. A., GREENE, P. T. & BRYAN, R. F. (1970). *Inorg. Chem.* 9, 1116–1123.

Acta Cryst. (1978). B34, 1486–1490

The Crystal Structure of [1,2-Bis(dicyclohexylphosphino)-3,3,4,4-tetrafluorocyclobut-1-ene-*P,P'*]tricarboxyliron

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(Received 19 September 1977; accepted 10 December 1977)

$\{(\text{C}_6\text{H}_{11})_2\text{PC}=\text{C}[\text{P}(\text{C}_6\text{H}_{11})_2]\text{CF}_2\text{CF}_2\}\text{Fe}(\text{CO})_3$, $M_r = 658.48$, space group *Pc*, $a = 9.473$ (4), $b = 11.026$ (4), $c = 15.561$ (9) Å, $\beta = 97.50$ (3)°, $V = 1611.4$ Å³, $Z = 2$, $D_c = 1.357$, $D_o = 1.34$ g cm⁻³, $R = 0.043$ for 1240 counter reflections. The Fe atom is five-coordinate with approximately trigonal-bipyramidal geometry. The Fe—P lengths are 2.281 (3) and 2.300 (3) Å (corrected for thermal motion). The molecule has approximate non-crystallographic mirror symmetry.

Experimental

The chunky crystals were ruby red. Weissenberg (*0kl*)

and precession (*h0l*, *hk0*) photographs taken with Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å) showed the crystal to be monoclinic, and systematically absent reflections *h0l* for $l = 2n + 1$ indicated the space group to be *P2/c* or *Pc*.

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A cleaved fragment $0.20 \times 0.14 \times 0.23$ mm [$\mu(\text{Mo } K\alpha) = 6.37 \text{ cm}^{-1}$] was mounted in a general orientation to minimize multiple reflection and was used to measure the entire diffraction data.

Preliminary photographic cell dimensions were refined by least squares from the angular setting of 12 reflections in the range $26 < 2\theta < 36^\circ$ measured with a Picker automated four-circle diffractometer and graphite-monochromatized Mo $K\alpha_1$ ($\lambda = 0.70926 \text{ \AA}$) radiation.

Integrated intensities from one quadrant were collected at 23°C with the θ - 2θ scan technique at a scan rate of 2° min^{-1} and a base scan width of 1.3° . (The scan

was modified to include dispersion.) Background was counted for 20 s at both scan limits. Two standard reflections were monitored every 60 reflections and subsequently used for scaling. All 1508 independent reflections within the asymmetric quadrant of reciprocal space up to $(\sin \theta)/\lambda = 0.4823 \text{ \AA}^{-1}$ were measured, but only the 1240 reflections with $I > 2.3\sigma(I)$ were used for the structure analysis; $\sigma(I)^2 = C + (t_c/2t_b)^2(B_1 + B_2) + 0.009[c - (B_1 + B_2)(t_c/2t_b)]$, where C is the total count in scan time t_c , and B_1 and B_2 are the two background counts each taken for time t_b . The intensities were corrected for Lorentz-polarization effects but not for absorption.

Table 1. Atomic coordinates ($\times 10^4$, for H $\times 10^3$)

Positions are designated in fractions of a cell edge; e.s.d.'s are in parentheses. H atoms were given isotropic temperature factors of 6.0 \AA^2 .

	x	y	z		x	y	z
Fe	0	1972 (1)	2500	H(41)	88	565	172
P(1)	1228 (4)	3627 (3)	2190 (2)	H(51)	-59	459	68
P(2)	576 (4)	1128 (3)	1263 (2)	H(52)	-164	432	135
F(1)	4148 (9)	3970 (7)	720 (5)	H(61)	-101	668	78
F(2)	2221 (10)	4786 (6)	91 (4)	H(62)	-247	602	53
F(3)	3493 (7)	1904 (6)	-134 (4)	H(71)	-265	745	163
F(4)	1548 (8)	2710 (6)	-747 (4)	H(72)	-297	615	195
O(1)	1001 (15)	-223 (9)	3442 (6)	H(81)	-35	734	233
O(2)	-189 (12)	3065 (9)	4176 (6)	H(82)	139	707	301
O(3)	-3047 (11)	1938 (11)	2102 (6)	H(91)	47	568	315
C(1)	671 (17)	639 (12)	3055 (9)	H(92)	-98	501	289
C(2)	-112 (15)	2610 (13)	3499 (9)	H(101)	243	432	348
C(3)	-1811 (15)	1952 (13)	2223 (8)	H(111)	285	592	265
C(4)	228 (12)	5074 (10)	1908 (7)	H(112)	382	513	213
C(5)	-962 (13)	4892 (10)	1180 (7)	H(121)	527	629	311
C(6)	-1691 (14)	6125 (11)	977 (8)	H(122)	449	582	387
C(7)	-2252 (13)	6667 (10)	1775 (9)	H(131)	659	483	392
C(8)	-1034 (13)	6778 (11)	2498 (8)	H(132)	622	444	295
C(9)	-310 (14)	5571 (11)	2703 (7)	H(141)	486	358	432
C(10)	2778 (12)	4134 (10)	2947 (6)	H(142)	586	281	381
C(11)	3515 (12)	5264 (9)	2685 (6)	H(151)	420	287	257
C(12)	4808 (13)	5604 (11)	3330 (7)	H(152)	342	240	334
C(13)	5834 (13)	4597 (12)	3479 (7)	H(161)	124	-81	169
C(14)	5165 (13)	3447 (11)	3766 (8)	H(171)	371	54	151
C(15)	3873 (12)	3084 (10)	3114 (7)	H(172)	309	44	239
C(16)	1705 (11)	-243 (8)	1359 (6)	H(181)	355	-163	244
C(17)	3198 (12)	31 (10)	1858 (7)	H(182)	497	-93	235
C(18)	4038 (13)	-1133 (10)	2063 (8)	H(191)	463	-260	140
C(19)	4181 (15)	-1837 (11)	1244 (9)	H(192)	476	-138	90
C(20)	2744 (13)	-2064 (11)	718 (8)	H(201)	288	-244	18
C(21)	1928 (13)	-886 (11)	535 (7)	H(202)	220	-260	104
C(22)	-792 (12)	726 (9)	353 (6)	H(211)	102	-106	21
C(23)	-1797 (11)	1766 (10)	10 (7)	H(212)	246	-37	20
C(24)	-2856 (14)	1319 (10)	-731 (8)	H(221)	-30	47	-11
C(25)	-3774 (13)	274 (11)	-486 (8)	H(231)	-230	205	47
C(26)	-2764 (12)	-758 (11)	-143 (7)	H(232)	-125	242	-19
C(27)	-1711 (12)	-337 (8)	614 (7)	H(241)	-347	198	-93
C(28)	1973 (12)	3292 (9)	1176 (7)	H(242)	-234	105	-119
C(29)	2748 (15)	2754 (12)	521 (8)	H(251)	-440	0	-99
C(30)	2398 (14)	2577 (9)	19 (8)	H(252)	-433	53	-4
C(31)	1634 (10)	2253 (10)	771 (6)	H(261)	-225	-103	-60
				H(262)	-331	-142	4
				H(271)	-110	-100	81
				H(272)	-222	-8	107

Determination and refinement of the structure

The noncentrosymmetric space group Pc was favoured, since D_c required $Z = 2$ and the Patterson synthesis suggested that no special position was occupied. This was confirmed by the successful refinement.

The positions of the Fe and two P atoms were determined from a Patterson synthesis. These positions and the scale factor were refined by full-matrix least squares to $R = \sum |F_o| - |F_c| / \sum |F_o| = 0.329$. A subsequent Fourier synthesis revealed the majority of the non-hydrogen atoms. The remaining non-hydrogen atoms were located from difference maps. With isotropic thermal parameters, the structure was refined to $R = 0.090$. At this stage, weights $w = 1/(\sigma F)^2$, where $\sigma F = \sigma(I)/(Lp)2F_o$, were used instead of the unit weight which was previously assigned. All H atoms were then included in the structure factor calculations at fixed positions. It was impossible to refine all parameters

simultaneously, and refinement proceeded on a block basis. Further cycles, eventually incorporating anisotropic thermal parameters for all non-hydrogen atoms, converged to $R = 0.043$.*

Scattering factors for the non-hydrogen atoms and anomalous-dispersion corrections, f' and f'' , for Fe and P atoms were from *International Tables for X-ray Crystallography* (1974). The scattering factor of H was from Stewart, Davidson & Simpson (1965).

The final positional parameters are listed in Table 1. Table 2 contains bond lengths and angles, and Table 3 shows the deviations of the atoms from various planes. A perspective view of the molecule is shown in Fig. 1 with the atom labelling.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33291 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. Bond lengths (Å) and angles (°) with *e.s.d.*'s in parentheses

Fe—P(1)	2.250 (4)	2.281 (3)*	C(2)—O(2)	1.177 (17)	C(13)—C(14)	1.511 (18)	C(24)—C(25)	1.522 (18)	
Fe—P(2)	2.268 (4)	2.300 (3)*	C(3)—O(3)	1.161 (18)	C(14)—C(15)	1.538 (16)	C(25)—C(26)	1.536 (17)	
Fe—C(1)	1.780 (14)	1.789 (13)†	C(4)—C(5)	1.504 (15)	C(15)—C(16)	1.553 (16)	C(26)—C(27)	1.515 (15)	
Fe—C(2)	1.722 (14)	1.725 (14)†	C(5)—C(6)	1.538 (17)	C(16)—C(17)	1.552 (15)	C(27)—C(22)	1.547 (15)	
Fe—C(3)	1.714 (4)	1.732 (14)†	C(6)—C(7)	1.534 (19)	C(17)—C(18)	1.523 (16)	C(28)—C(29)	1.425 (18)	
P(1)—C(4)	1.878 (12)		C(7)—C(8)	1.507 (17)	C(18)—C(19)	1.514 (19)	C(29)—C(30)	1.529 (17)	
P(1)—C(10)	1.845 (11)		C(8)—C(9)	1.512 (17)	C(19)—C(20)	1.515 (18)	C(29)—F(1)	1.343 (16)	1.377 (16)†
P(1)—C(28)	1.849 (12)		C(9)—C(4)	1.501 (17)	C(20)—C(21)	1.520 (17)	C(29)—F(2)	1.380 (15)	1.408 (15)†
P(2)—C(16)	1.846 (10)		C(10)—C(11)	1.510 (15)	C(21)—C(16)	1.505 (15)	C(30)—F(3)	1.322 (14)	1.351 (14)†
P(2)—C(22)	1.845 (10)		C(11)—C(12)	1.525 (15)	C(22)—C(23)	1.541 (15)	C(30)—F(4)	1.356 (13)	1.392 (13)†
P(2)—C(31)	1.824 (11)		C(12)—C(13)	1.474 (17)	C(23)—C(24)	1.510 (16)	C(30)—C(31)	1.497 (16)	
C(1)—O(1)	1.147 (17)						C(31)—C(28)	1.327 (15)	
P(1)—Fe—P(2)	87.8 (1)		C(5)—C(4)—P(1)	111.6 (8)	C(23)—C(22)—C(27)	108.2 (9)			
C(1)—Fe—P(1)	127.7 (5)		C(5)—C(4)—C(9)	111.6 (10)	C(27)—C(22)—P(2)	110.1 (7)			
C(1)—Fe—P(2)	87.9 (5)		C(9)—C(4)—P(1)	109.4 (8)	C(22)—C(23)—C(24)	109.9 (9)			
C(1)—Fe—C(2)	87.7 (6)		C(4)—C(5)—C(6)	108.0 (9)	C(23)—C(24)—C(25)	113.6 (10)			
C(1)—Fe—C(3)	113.2 (7)		C(5)—C(6)—C(7)	111.8 (10)	C(24)—C(25)—C(26)	107.3 (10)			
C(2)—Fe—P(1)	87.5 (5)		C(6)—C(7)—C(8)	108.8 (10)	C(25)—C(26)—C(27)	111.1 (9)			
C(2)—Fe—P(2)	169.7 (5)		C(7)—C(8)—C(9)	111.6 (10)	C(26)—C(27)—C(22)	111.3 (9)			
C(2)—Fe—C(3)	93.1 (6)		C(8)—C(9)—C(4)	109.9 (10)	C(29)—C(28)—P(1)	145.9 (9)			
C(3)—Fe—P(1)	119.0 (5)		C(11)—C(10)—P(1)	115.8 (7)	C(29)—C(28)—C(31)	94.9 (10)			
C(3)—Fe—P(2)	97.2 (5)		C(11)—C(10)—C(15)	109.7 (9)	C(31)—C(28)—P(1)	118.8 (9)			
C(4)—P(1)—C(10)	103.1 (5)		C(15)—C(10)—P(1)	109.8 (7)	C(28)—C(29)—C(30)	88.0 (10)			
C(4)—P(1)—C(28)	102.0 (5)		C(10)—C(11)—C(12)	112.6 (8)	C(28)—C(29)—F(1)	119.0 (10)			
C(4)—P(1)—Fe	118.7 (4)		C(11)—C(12)—C(13)	112.0 (9)	C(28)—C(29)—F(2)	117.0 (11)			
C(10)—P(1)—C(28)	104.2 (5)		C(12)—C(13)—C(14)	112.6 (11)	C(30)—C(29)—F(1)	114.2 (11)			
C(10)—P(1)—Fe	120.0 (4)		C(13)—C(14)—C(15)	110.6 (9)	C(30)—C(29)—F(2)	114.3 (9)			
C(28)—P(1)—Fe	106.6 (4)		C(14)—C(15)—C(10)	111.9 (9)	F(1)—C(29)—F(2)	104.4 (10)			
C(16)—P(2)—C(22)	102.0 (5)		C(17)—C(16)—P(2)	111.2 (7)	C(29)—C(30)—C(31)	84.1 (9)			
C(16)—P(2)—C(31)	104.3 (5)		C(17)—C(16)—C(21)	107.3 (9)	C(29)—C(30)—F(3)	116.5 (10)			
C(16)—P(2)—Fe	117.9 (3)		C(21)—C(16)—P(2)	117.5 (7)	C(29)—C(30)—F(4)	114.9 (9)			
C(22)—P(2)—C(31)	102.2 (5)		C(16)—C(17)—C(18)	111.0 (9)	C(31)—C(30)—F(3)	118.9 (9)			
C(22)—P(2)—Fe	121.8 (4)		C(17)—C(18)—C(19)	110.9 (10)	C(31)—C(30)—F(4)	114.9 (10)			
C(31)—P(2)—Fe	106.5 (3)		C(18)—C(19)—C(20)	111.5 (11)	F(3)—C(30)—F(4)	107.0 (9)			
Fe—C(1)—O(1)	174.4 (15)		C(19)—C(20)—C(21)	111.0 (10)	C(28)—C(31)—C(30)	93.0 (9)			
Fe—C(2)—O(2)	178.9 (16)		C(20)—C(21)—C(16)	111.6 (9)	C(28)—C(31)—P(2)	120.0 (8)			
Fe—C(3)—O(3)	174.7 (12)		C(23)—C(22)—P(2)	115.3 (7)	C(30)—C(31)—P(2)	146.9 (8)			

* After thermal-motion correction, the atoms are assumed to move independently.

† After thermal-motion correction, the second atom is assumed to ride on the first.

Discussion

The structural parameters of the molecule are close to those in related complexes (Tables 4 and 5). The Fe atom coordination departs from ideal trigonal-bipyramidal symmetry, possibly caused by the close approach of the cyclohexane groups on the two P atoms. Fe, P(2), C(2), C(3), O(2) and O(3) form a plane.

The two phosphine ligands are *cis* to each other with $P(1)-Fe-P(2) = 87.8(1)^\circ$. The Fe-P lengths, 2.250 (4) and 2.268 (4) Å, are significantly different (0.018 Å) but are in the range of those shown in Table 4, e.g. 2.260 (5) Å in $Fe(CO)_4(PMe_2)_2Fe(CO)_4$ (Jarvis, Mais, Owston & Thompson, 1968). Both these compounds contain pentacoordinated Fe. The effect of the bulky cyclohexyl groups on the Fe-P length appears to be insignificant. $Fe-C(1) = 1.780(14)$ Å is longer

[0.06 (2) Å] than $Fe-C(2)$ and $Fe-C(3)$, and $C(1)-Fe-C(3) = 113.2(7)^\circ$ is significantly larger than $C(1)-Fe-C(2) = 87.7(6)^\circ$ and $C(2)-Fe-C(3) = 93.1(6)^\circ$.

The cyclohexyl rings are in the chair form. The C-C lengths range from 1.47 (2) to 1.55 (2) Å with an average of 1.52 Å. The angles range from 107.3 (9) to 113.6 (10)° with a mean of 110.6°. These values are in excellent accord with those expected.

The cyclobutene ring is planar with one P atom displaced from the plane. The geometry of the tetra-fluorocyclobutene ligand is comparable with those in Table 5. $C(28)-C(31)$, 1.327 (15) Å, corresponding to the C=C double bond of cyclobutene, retains its double-bond character in this complex.

Table 3. Deviations (Å) of atoms from least-squares planes

χ^2 is $\sum p^2/\sigma^2(p)$, where p is the distance in Å of the atom normal to the plane.

(1) Plane through the cyclobutene ring C atoms

$$0.7829x - 0.4150y + 0.4635z - 0.6103 = 0; \chi^2 = 0$$

C(28)	0.000	P(1)*	0.142
C(29)	0.000	P(2)*	-0.003
C(30)	0.000	Fe*	0.123
C(31)	0.000		

(2) Plane through Fe, P(2), C(2), C(3), O(2) and O(3)

$$0.0820x + 0.9098y - 0.4068z - 0.3659 = 0; \chi^2 = 6.07$$

Fe	0.002	C(3)	0.020
P(2)	-0.003	O(2)	0.004
C(2)	-0.010	O(3)	-0.012

* Atom not used to define plane.

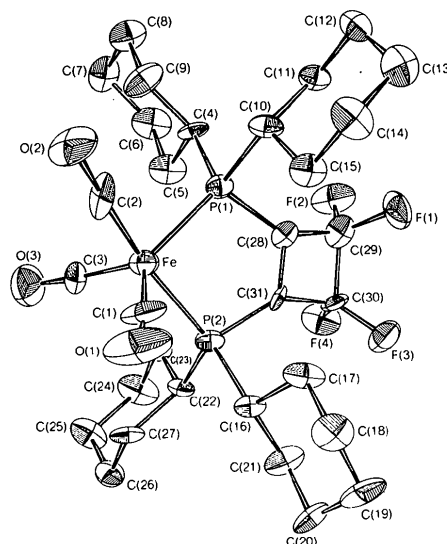


Fig. 1. A perspective view of the refined molecule with the atom labelling. 40% probability thermal ellipsoids are shown.

Table 4. Comparison of Fe-P(phosphine), Fe-C(carbonyl) and C-O bond lengths (Å), and Fe-C-O angles (°) in various Fe-carbonyl-phosphine complexes

Mean values are given where there are several chemically equivalent dimensions.

Fe-P	Fe-C	C-O	Fe-C-O	Complexes	References
2.263	1.792	1.140	174.8	$[Fe(CO)_2(N_2Ph)(PPh_3)] [BF_4]$	Haymore & Ibers (1975)
2.254	1.770	1.151	176.0	$[Ph_2PC=C(Me_2As)CF_2CF_2]_2Fe_2(CO)_4$	Einstein & Jones (1973)
2.248	1.74	1.17	174	$(C_4F_4)Fe_3(CO)_9(AsMe_2)(PPh_2)$	Einstein & Jones (1972c)
2.224 (3)	1.74	1.17	179	$(C_4F_4)Fe(CO)_4(AsMe_2)(PPh_2)$	Einstein & Jones (1972b)
2.190 (4)	1.80	1.15 (2)	175.8	<i>trans</i> - $P(OCH_2)_3PFe(CO)_3P(OCH_2)_3P$	Allison, Clardy & Verkade (1972)
2.234 (3)	1.75 (1)	1.17	-	$Fe(CO)_2[CH_2CHC_6H_4PPh_2]_2$	Bennett, Robertson, Tomkins & Whimp (1971)
2.237 (2)	1.793	1.15	178.4	$Fe(CO)_4(PHPh_2)$	Kilbourn, Raeburn & Thompson (1969)
2.260 (5)	1.79 (1)	1.16 (2)	176.6	$Fe(CO)_4(PMe_2)_2Fe(CO)_4$	Jarvis, Mais, Owston & Thompson (1968)
2.259	1.739	1.162	176.0		This work

Table 5. Comparison of $-C=C-$, $=C-C-$, $-C-C-$ and $C-F$ bond lengths (Å) in the tetrafluorocyclobutene ring

Mean values are given where there are several chemically equivalent dimensions.

$-C=C-$	$=C-C-$	$-C-C-$	$C-F$	Complexes	References
1.347	1.508	1.526	1.344	$(C_4F_4)_2Mo(CO)_4[P(C_6H_{11})_2]_2$	Cullen, Williams, Einstein & Huang (1977)
1.361 (10)	1.502	1.516 (12)	1.347	$(C_4F_4)[Mn(CO)_4]_2(AsMe_2)_2$	Einstein & MacGregor (1974)
1.326 (8)	1.515	1.550 (9)	1.341	$(C_4F_4)_2Fe_2(CO)_4(AsMe_2)_2(PPh_2)_2$	Einstein & Jones (1973)
1.476 (8)	1.499	1.538 (9)	1.368		
1.36 (1)	1.51 (1)	1.54 (1)	1.347	$(C_4F_4)Co_3(CO)_7(AsMe_2)_2(CMe)$	Einstein & Jones (1972a)
1.34 (1)	1.51 (2)	1.50 (2)	1.34	$(C_4F_4)Fe(CO)_4(AsMe_2)(PPh_2)$	Einstein & Jones (1972b)
1.39	1.48	1.54	1.37	$(C_4F_4)Fe_3(CO)_9(AsMe_2)(PPh_2)$	Einstein & Jones (1972c)
1.41	1.52	1.53	1.36		
1.44 (3)	1.55	1.48 (3)	—	$(C_4F_4)Fe_3(CO)_9(AsMe_2)_2$	Einstein, Pilotti & Restivo (1971)
1.383 (9)	1.501 (9)	1.594 (9)	1.335 (5)	$(C_4F_4)_3$	Cobbedick & Einstein (1976)
1.342 (6)	1.508 (3)	(1.595)	1.336 (6)	$F_2C=CF=CF-CF_2$	Chang, Porter & Bauer (1971)
1.327 (15)	1.461	1.529 (17)	1.350	$(C_4F_4)[P(C_6H_{11})_2]_2Fe(CO)_3$	This work

We thank the National Research Council of Canada for financial support and Dr W. R. Cullen for his gift of the crystals and interest in the work.

References

- ALLISON, D. A., CLARDY, J. & VERKADE, J. G. (1972). *Inorg. Chem.* **11**, 2804–2809.
- BENNETT, M. A., ROBERTSON, G. B., TOMKINS, I. B. & WHIMP, P. O. (1971). *Chem. Commun.* pp. 341–342.
- CHANG, C. H., PORTER, R. F. & BAUER, S. H. (1971). *J. Mol. Struct.* **7**, 89–99.
- COBBLEDICK, R. E. & EINSTEIN, F. W. B. (1976). *Acta Cryst.* **B32**, 1908–1909.
- CULLEN, W. R., WILLIAMS, M., EINSTEIN, F. W. B. & HUANG, C.-H. (1977). *J. Fluorine Chem.* Submitted.
- EINSTEIN, F. W. B. & JONES, R. D. G. (1972a). *Inorg. Chem.* **11**, 395–400.
- EINSTEIN, F. W. B. & JONES, R. D. G. (1972b). *J. Chem. Soc. Dalton Trans.* pp. 442–446.
- EINSTEIN, F. W. B. & JONES, R. D. G. (1972c). *J. Chem. Soc. Dalton Trans.* pp. 2563–2568.
- EINSTEIN, F. W. B. & JONES, R. D. G. (1973). *Inorg. Chem.* **12**, 255–259.
- EINSTEIN, F. W. B. & MACGREGOR, A. C. (1974). *J. Chem. Soc. Dalton Trans.* pp. 783–786.
- EINSTEIN, F. W. B., PILOTTI, A.-M. & RESTIVO, R. (1971). *Inorg. Chem.* **10**, 1947–1951.
- HAYMORE, B. L. & IBERS, J. A. (1975). *Inorg. Chem.* **14**, 1369–1376.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.
- JARVIS, J. A. J., MAIS, R. H. B., OWSTON, P. G. & THOMPSON, D. T. (1968). *J. Chem. Soc. A*, pp. 622–626.
- KILBOURN, B. T., RAEBURN, J. A. & THOMPSON, D. T. (1969). *J. Chem. Soc. A*, pp. 1906–1909.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.