# Crystal and Molecular Structure of Tricarbonyl(decafluorocyclohepta-1,3-dienyl)iron

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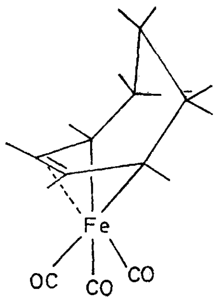
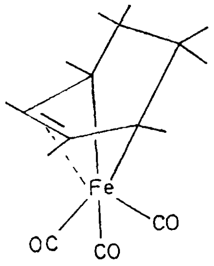
The crystal and molecular structure of the title compound (I) has been determined by a single-crystal *X*-ray analysis from three-dimensional counter data. Crystals are orthorhombic, space group *Pnma*, with $Z = 4$ in a cell of dimensions $a = 11{\cdot}305$, $b = 12{\cdot}110$, $c = 9{\cdot}075$ (all $\pm 0{\cdot}010$) Å. The structure was established by direct methods and refined by least squares to $R$ 3·7% for 1066 structure amplitudes. The complex has exact $C_s$ symmetry. The boat-like conformation of the ring and molecular dimensions (bond length $\sigma$ 0·003—0·007 Å) are consistent with a significant contribution to the metal–cycloheptadiene bonding of a $\sigma$–$\pi$ type of interaction.

MANY diene–iron tricarbonyl complexes have been prepared and there have been numerous *X*-ray crystallographic examinations of these.[1,2] We now report an *X*-ray structural investigation of (I) tricarbonyl(decafluorocyclohepta-1,3-dienyl)iron, prepared[3] by the reaction of decafluorocyclohepta-1,3-diene with $Fe_3(CO)_{12}$. Previous *X*-ray structural determinations on iron carbonyl complexes containing highly fluorinated ligands have included those of tricarbonyl[tetrakis(trifluoromethyl)cyclopentadienone]iron[4] and tetracarbonyl(hexafluorobutadiene)iron.[5] Of particular relevance to the present investigation, however, are the results of the *X*-ray crystallographic analysis[6] of the closely related compound (II) tricarbonyl(octafluorocyclohexa-1,3-dienyl)iron.



(I)



(II)

## EXPERIMENTAL

*Crystallographic Measurements*.—Crystals were obtained by slow sublimation (50°/760 mmHg) of the compound on to a water-cooled probe. For the *X*-ray investigation a crystal of dimensions *ca*. 0·6 × 0·5 × 0·5 mm was mounted about the direction of elongation, which coincided with the crystallographic *a* axis, and sealed in a Pantak glass capillary tube (0·75 mm diam.). Approximate cell dimensions were obtained from oscillation and Weissenberg photographs. Final cell dimensions and intensity data were measured with a Stoe two-circle computer-controlled diffractometer with graphite-monochromated Mo-$K_\alpha$ radiation and a scintillation counter. Of 1530 reflections scanned within the range $0{\cdot}1 < \sin\theta/\lambda \leqslant 0{\cdot}65$, 1066, having $I > 3\sigma(I)$, were considered observed and were used in the structure analysis. The $\omega$ scan mode was employed, and for each reflection 140 counts of 2 s at intervals of 0·01° were taken. For reflections on the 4th and higher-layer lines a variable scan-range technique was employed, $\Delta\omega$ being calculated by the expression † $A + B \sin\mu/\tan\theta'$, with $A = 1{\cdot}1$ and $B = 0{\cdot}5$. Backgrounds were measured for 60 s at each end of the scan. In the conversion of intensities to structure amplitudes, the polarisation factor appropriate to monochromated radiation was used. Absorption corrections were not applied.

*Crystal Data*.—$C_{10}F_{10}FeO_3$, $M = 413{\cdot}9$, Orthorhombic, $a = 11{\cdot}305 \pm 0{\cdot}010$, $b = 12{\cdot}110 \pm 0{\cdot}010$, $c = 9{\cdot}075 \pm 0{\cdot}010$ Å, $U = 1242{\cdot}4$ Å$^3$, $Z = 4$, $D_c = 2{\cdot}212$, $F(000) = 800$. Systematic absences: $0kl$ when $(k + l)$ is odd, $hk0$ when $h$ is odd, space group $Pna2_1$ ($C_{2v}^9$) or $Pnma$ ($D_{2h}^{16}$); *Pnma* was established as a result of the analysis. Molecular symmetry $m$ ($C_s$). Mo-$K_\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å; $\mu$(Mo-$K_\alpha$) = 14·1 cm$^{-1}$.

*Structure Analysis*.—The co-ordinates of the iron atom were obtained from a three-dimensional Patterson synthesis, but the electron density evaluated on the basis of the phase angles calculated from these co-ordinates could not be interpreted. The structure was finally solved by direct methods[7] using a multi-solution technique[8] and tangent refinement of phases.[9] The lower symmetry space group $Pna2_1$ was assumed but the resultant $E$ map indicated mirror symmetry. All the atoms of the molecule could be located from the $E$ map and at this stage $R$ was 38·8%. The centrosymmetric space group *Pnma* was then adopted and refinement of atomic parameters carried out by a full-matrix least-squares procedure. Initially, positional and isotropic thermal parameters were adjusted and after 6 cycles $R$ was reduced to 12·4%. Finally the atoms were allowed to vibrate anisotropically and the refinement was terminated when all calculated shifts were $< 0{\cdot}01\sigma$, giving a final value for $R$ of 3·7% for the 1066 observed structure amplitudes.‡

The weighting scheme used in the final cycles of refinement was $W^{\frac{1}{2}} = 1{\cdot}0$ if $|F_o| < 32$ and $W^{\frac{1}{2}} = 32/|F_o|$ if $|F_o| > 32$. Atomic scattering factors were taken from ref. 10.

Computations were performed on the Birmingham

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† θ′ Is equivalent to the azimuth angle ν defined by M. J. Buerger, 'Crystal Structure Analysis,' Wiley, New York, 1960, p. 122.

‡ Observed and calculated structure factors are published in Supplementary Publication No. SUP 20912 (8 pp., 1 microfiche). See Notice to Authors No. 7 in *J.C.S. Dalton*, 1973, Index issue.

[1] R. Pettit and G. F. Emerson, *Adv. Organometallic Chem.*, 1964, **1**, 1.

[2] M. R. Churchill and R. Mason, *Adv. Organometallic Chem.*, 1967, **5**, 93.

[3] P. Dodman and J. C. Tatlow, *J. Organometallic Chem.*, in the press.

[4] N. A. Bailey and R. Mason, *Acta Cryst.*, 1966, **21**, 652.

[5] P. B. Hitchcock and R. Mason, *Chem. Comm.*, 1967, 242.

[6] (*a*) M. R. Churchill and R. Mason, *Proc. Chem. Soc.*, 1964, 226; (*b*) *Proc. Roy. Soc.*, 1967, *A*, **301**, 433.

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

[8] G. Germain and M. M. Woolfson, *Acta Cryst.*, 1968, **B24**, 91.

[9] O. Kennard, N. W. Isaacs, W. D. S. Motherwell, J. C. Coppola, D. L. Wampler, A. C. Larson, and D. G. Watson, *Proc. Roy. Soc.*, 1971, *A*, **325**, 401.

[10] H. P. Hanson, F. Herman, J. D. Lea, and S. Skillman, *Acta Cryst.*, 1964, **17**, 1040.

University KDF9 computer by use of programs which are acknowledged in ref. 11.

RESULTS AND DISCUSSION

The stereochemistry of the molecule is illustrated in Figure 1, which also shows the atomic numbering scheme used. The molecule possesses exact $C_s$ symmetry with atoms F(4a), C(4), Fe, F(4b), C(6), and O(2) lying in the mirror plane. Atomic co-ordinates and thermal parameters are listed in Tables 1 and 2 respectively. Molecular dimensions are in Table 3, with mean standard deviations for bond lengths of *ca.* 0·005 Å and for bond angles and torsion angles *ca.* 0·3 and 0·5° respectively.

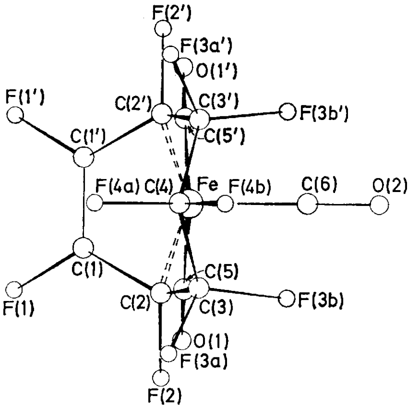

FIGURE 1 The molecule viewed along the *c* axis (the positive direction of the *c* axis is towards the viewer, *a* and *b* axes as in Figure 2)

TABLE 1

Fractional atomic co-ordinates (× $10^4$), with estimated standard deviations in parentheses

| | *x* | *y* | *z* |
|---|---|---|---|
| Fe | −1143(1) | 7500 | −1239(1) |
| C(1) | −2584(3) | 6923(3) | −86(4) |
| C(2) | −1523(3) | 6362(3) | 324(4) |
| C(3) | −1011(3) | 6429(3) | 1856(4) |
| C(4) | −1281(5) | 7500 | 2685(5) |
| C(5) | −1199(3) | 6414(3) | −2678(4) |
| C(6) | 465(5) | 7500 | −1048(5) |
| F(1) | −3532(2) | 6383(2) | −587(3) |
| F(2) | −1510(2) | 5273(2) | −100(3) |
| F(3a) | −1408(3) | 5593(2) | 2693(3) |
| F(3b) | 186(2) | 6310(2) | 1802(3) |
| F(4a) | −2436(3) | 7500 | 3043(4) |
| F(4b) | −660(4) | 7500 | 3962(3) |
| O(1) | −1226(3) | 5761(3) | −3545(3) |
| O(2) | 1442(3) | 7500 | −939(5) |

TABLE 2

Anisotropic thermal parameters (× $10^4$),* with estimated standard deviations in parentheses

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| Fe | 231(3) | 281(3) | 239(3) | 0 | 14(3) | 0 |
| C(1) | 248(15) | 378(18) | 332(16) | −58(13) | 45(12) | −13(11) |
| C(2) | 351(16) | 233(14) | 357(16) | −4(12) | 40(13) | −1(11) |
| C(3) | 410(19) | 373(18) | 341(16) | 34(15) | 33(15) | 92(12) |
| C(4) | 364(25) | 618(32) | 231(21) | 0 | 11(19) | 0 |
| C(5) | 364(17) | 418(18) | 338(16) | 13(15) | −5(14) | −20(13) |
| C(6) | 386(28) | 609(34) | 262(24) | 0 | 11(20) | 0 |
| F(1) | 342(11) | 520(13) | 603(14) | −159(8) | −26(9) | −49(9) |
| F(2) | 642(14) | 252(9) | 569(13) | 8(9) | 45(11) | −42(8) |
| F(3a) | 908(19) | 534(14) | 527(14) | −100(14) | 2(13) | 267(10) |
| F(3b) | 411(12) | 603(14) | 574(13) | 172(10) | −80(10) | 47(9) |
| F(4a) | 433(18) | 883(27) | 414(17) | 0 | 204(14) | 0 |
| F(4b) | 719(23) | 861(27) | 280(15) | 0 | −104(15) | 0 |
| O(1) | 787(20) | 563(18) | 457(15) | 14(17) | −7(16) | −209(12) |
| O(2) | 263(20) | 1120(40) | 514(24) | 0 | 8(16) | 0 |

* In the form: $T = \exp[-2\pi^2(U_{11}h^2a^{*2} + \cdots + 2U_{12}hka^*b^* + \cdots)]$.

TABLE 3

Molecular dimensions

(*a*) Bonded distances (Å) with standard deviations (× $10^3$) in parentheses

| | | | |
|---|---|---|---|
| Fe–C(1) | 2·059(3) | C(1)–F(1) | 1·336(4) |
| Fe–C(2) | 2·024(4) | C(2)–F(2) | 1·374(4) |
| Fe–C(5) | 1·854(4) | C(3)–F(3a) | 1·343(4) |
| Fe–C(6) | 1·826(6) | C(3)–F(3b) | 1·361(4) |
| C(1)–C(1′) | 1·398(7) | C(4)–F(4a) | 1·345(6) |
| C(1)–C(2) | 1·428(5) | C(4)–F(4b) | 1·355(6) |
| C(2)–C(3) | 1·509(5) | C(5)–O(1) | 1·116(4) |
| C(3)–C(4) | 1·530(5) | C(6)–O(2) | 1·108(7) |

(*b*) Selected intramolecular non-bonded distances

| | | | |
|---|---|---|---|
| Fe ··· C(3) | 3·098 | C(3) ··· C(3′) | 2·594 |
| Fe ··· C(4) | 3·564 | F(4a) ··· C(1) | 2·929 |
| Fe ··· F(4a) | 4·152 | C(5) ··· C(5′) | 2·631 |
| Fe ··· F(4b) | 4·752 | C(5) ··· C(6) | 2·731 |
| C(4) ··· C(1) | 2·996 | O(1) ··· O(1′) | 4·212 |
| C(2) ··· C(2′) | 2·756 | O(1) ··· O(2) | 4·373 |

(*c*) Bond angles (°); mean standard deviation *ca.* 0·3°

| | | | |
|---|---|---|---|
| C(1)–Fe–C(1′) | 39·7 | C(1)–C(2)–Fe | 70·9 |
| C(1)–Fe–C(2) | 40·9 | F(2)–C(2)–Fe | 117·1 |
| C(1)–Fe–C(5) | 95·2 | C(3)–C(2)–Fe | 121·9 |
| C(1)–Fe–C(6) | 137·7 | C(2)–C(3)–C(4) | 115·0 |
| C(2)–Fe–C(2′) | 85·9 | C(2)–C(3)–F(3a) | 110·6 |
| C(2)–Fe–C(5) | 90·2 | C(2)–C(3)–F(3b) | 110·0 |
| C(2)–Fe–C(6) | 98·3 | C(4)–C(3)–F(3a) | 107·1 |
| C(5)–Fe–C(5′) | 90·4 | C(4)–C(3)–F(3b) | 107·8 |
| C(5)–Fe–C(6) | 95·8 | F(3a)–C(3)–F(3b) | 105·8 |
| C(2)–C(1)–C(1′) | 118·4 | C(3)–C(4)–C(3′) | 115·9 |
| C(2)–C(1)–F(1) | 122·0 | C(3)–C(4)–F(4a) | 108·2 |
| C(1′)–C(1)–F(1) | 119·3 | C(3)–C(4)–F(4b) | 108·5 |
| C(1)–C(2)–C(3) | 122·4 | F(4a)–C(4)–F(4b) | 107·2 |
| C(1)–C(2)–F(2) | 113·1 | Fe–C(5)–O(1) | 179·6 |
| C(3)–C(2)–F(2) | 107·8 | Fe–C(6)–O(2) | 179·7 |

(*d*) Torsion angles (°); mean standard deviation *ca.* 0·5°. Sign convention as defined in ref. 17

| | |
|---|---|
| C(1′)–C(1)–C(2)–C(3) | 65·4 |
| C(1′)–C(1)–C(2)–F(2) | −162·9 |
| F(1)–C(1)–C(2)–C(3) | −120·6 |
| F(1)–C(1)–C(2)–F(2) | 11·1 |
| C(1)–C(2)–C(3)–C(4) | −28·9 |
| C(1)–C(2)–C(3)–F(3a) | 92·6 |
| C(1)–C(2)–C(3)–F(3b) | −150·8 |
| F(2)–C(2)–C(3)–C(4) | −162·8 |
| F(2)–C(2)–C(3)–F(3a) | −41·3 |
| F(2)–C(2)–C(3)–F(3b) | 75·3 |
| C(2)–C(3)–C(4)–C(3′) | −50·8 |
| C(2)–C(3)–C(4)–F(4a) | 71·0 |
| C(2)–C(3)–C(4)–F(4b) | −173·0 |
| F(3a)–C(3)–C(4)–C(3′) | −174·2 |
| F(3a)–C(3)–C(4)–F(4a) | −52·4 |
| F(3a)–C(3)–C(4)–F(4b) | 63·6 |
| F(3b)–C(3)–C(4)–C(3′) | 72·3 |
| F(3b)–C(3)–C(4)–F(4a) | −166·0 |
| F(3b)–C(3)–C(4)–F(4b) | −50·0 |
| C(2)–C(1)–C(1′)–F(1′) | 174·2 |
| C(2)–C(1)–C(1′)–C(2′) | 0·0 * |

* Required to be zero by the $C_s$ symmetry.

As in (II),[6] the bonding of the iron to the unsaturated ring system is through atoms C(1), C(2), C(1′), and C(2′), and stabilises a state intermediate between the ground and first excited states, the molecule being regarded as a substituted butadiene complex.[6b] Hückel MO calculations [4,6b] predict bond lengths of 1·36, 1·45—1·46, and

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[11] J. J. Guy and T. A. Hamor, *J.C.S. Perkin II*, 1973, 942.

1·36 Å for the ground state of butadiene and lengths of 1·45, 1·39, and 1·45 Å for the first excited state. Our values for these lengths are 1·428, 1·398, and 1·428 Å, which may be compared with values of 1·397, 1·374, and 1·397 Å in (II). The distances Fe–C(1) and Fe–C(2), (2·059 and 2·024 Å), are similar to the corresponding distances in (II) (2·060 and 1·993 Å). Transformation towards the excited state involves rehybridisation of atoms C(2) [and C(2′)] from their formal $sp^2$ state towards a tetrahedral $sp^3$ state. Thus fluorine atom F(2) lies 0·371 Å below the butadiene plane [plane (*a*) in Table 4] on the same side of this plane as the iron atom and the angles F(2)–C(2)–C(1) and F(2)–C(2)–C(3) are 113·1 and 107·8° respectively.

The lengths of the carbon–fluorine bonds C(1)–F(1) (1·336 Å) and C(2)–F(2) (1·374 Å) further reflect the change of C(2) towards $sp^3$ hybridisation. The former is in good agreement with the accepted value[12] of a C–F bond length for a vinylic fluorine (1·333 Å), while the latter is more comparable with that of a paraffinic C–F bond in monofluoro-substituted carbon[12] (1·379 Å).

TABLE 4

Plane calculations

(i) Deviations (Å) of atoms from certain planes.

In the equations of the planes, $x$, $y$, and $z$ are fractional coordinates relative to the cell axes

Plane (*a*): C(1), C(2), C(1′), C(2′)

$$-3{\cdot}342x + 0{\cdot}000y + 8{\cdot}669z = 0{\cdot}789$$

F(1) 0·118, F(2) 0·371, Fe 1·482

Plane (*b*): C(2), C(3), C(2′), C(3′)

$$-10{\cdot}439x + 0{\cdot}000y + 3{\cdot}484z = 1{\cdot}702$$

C(4) –0·571, F(2) 0·161

Plane (*c*): C(3), C(4), C(3′)

$$-10{\cdot}472x + 0{\cdot}000y - 3{\cdot}419z = 0{\cdot}424$$

F(4a) –1·086, F(4b) 1·087

Plane (*d*): C(5), C(6), C(5′)

$$6{\cdot}987x + 0{\cdot}000y - 7{\cdot}134z = 1{\cdot}073$$

C(1) 2·817, C(2) 2·367, Fe 0·987, O(1) –0·600, O(2) –0·604

(ii) Inter-planar angles (°)

| | | | |
|---|---|---|---|
| (*a*)–(*b*) | 129·8 | (*a*)–(*d*) | 21·0 |
| (*a*)–(*c*) | 94·9 | (*b*)–(*c*) | 135·3 |

The C–F bond lengths of difluoro-substituted carbon atoms are 1·36 in $CH_2F_2$[13] and 1·345 Å in $CHF_2{\cdot}CH_3$.[14] In 1,4-dibenzoyldecafluoronorbornane[15] they range from 1·336 to 1·350, mean 1·343 Å (uncorrected for thermal libration), and from 1·354 to 1·368, mean 1·363 Å (corrected). The bond lengths C(3)–F(3a) and C(3)–F(3b) (1·343 and 1·361 Å) appear to differ significantly from each other, although neither differs significantly from the values observed in the fluoronorbornane. The corresponding C–F bonds (1·336 and 1·368 Å) in the crystal structure of (II) are in good agreement with our values, although their difference in length is more pronounced. When our C–F bond lengths are corrected for thermal effects by use of the 'riding' model[16] this difference virtually disappears, the corrected lengths for C(3)–F(3a) and C(3)–F(3b) being 1·374 and 1·379 Å. The lengths of the C(4)–F bonds (1·345 and 1·355 Å) are virtually equal and in good agreement with the values already quoted for this type of C–F bond.

The carbocyclic ring adopts a boat-like conformation. The 'boat' can be described in terms of the three planes (*a*)—(*c*) (Table 4). The angles between the central plane of the boat and the four-atom 'stern' and the three-atom 'bow' are 129·8 and 135·3° respectively, so that the boat is flatter than indicated by a model of the excited state of the cycloheptadiene ligand, constructed on the basis of standard bond lengths and angles. Compared with the standard model, the distances between F(4a) and C(1) and the centre of the C(1)–C(1′) bond are increased from 2·1 and 2·0 Å to 2·93 and 2·84 Å. This distortion from the model conformation manifests itself in large values for the ring angles at C(2), C(3), and C(4) (122·4, 115·0, and 115·9°) and a rotation of *ca.* 9° about the C(3)–C(4) bond from the fully staggered conformation [torsion angle[17] C(2)–C(3)–C(4)–C(3′) –50·8°]. The C(3)–C(4) bond length (1·530 Å) is normal for $C(sp^3)$–$C(sp^3)$, and can be compared with the analogous bond in (II) of length 1·558 Å, where, it was suggested,[6b] eclipsing of fluorine atoms lengthens the bond.

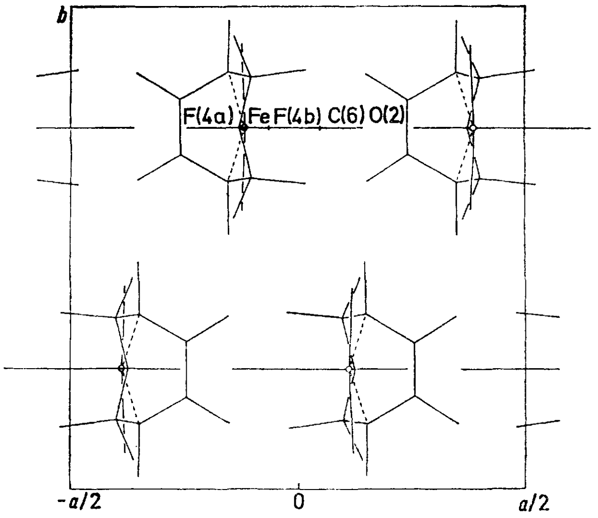

FIGURE 2 The crystal structure projected along the $c$ axis

As has been found in other iron tricarbonyl complexes, the three carbonyl groups are slightly distorted from strict $C_{3v}$ symmetry.[18] Thus the angle C(5)–Fe–C(5′) is 90·4°, while the angles C(5)–Fe–C(6) [and C(5′)–Fe–C(6)] are 95·8°, the smallest angle being ***trans*** to the two σ-bonded carbon atoms. The C···C and O···O distances between the carbonyl groups show a corresponding difference. The bond lengths Fe–C(5) and Fe–C(6) (1·854 and 1·826 Å) differ significantly, con-

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[12] *Chem. Soc. Special Publ.*, No. 18, 1965.

[13] S. P. S. Porto, *J. Mol. Spectroscopy*, 1959, **3**, 248.

[14] N. Solimene and B. P. Dailey, *J. Chem. Phys.*, 1954, **22**, 2042.

[15] T. A. Hamor and D. J. Watkin, *J.C.S. Perkin II*, 1974, 140.

[16] W. R. Busing and H. A. Levy, *Acta Cryst.*, 1964, **17**, 142.

[17] W. Klyne and V. Prelog, *Experientia*, 1960, **16**, 521.

[18] See refs. 4 and 6(*b*), and examples cited therein.

sistent with the suggestion [6b] that the metal *d* electrons are preferentially associated with the C(1)–C(1′) bond and are therefore not so readily available for bonding in the Fe–C(6) bond *trans* to C(1)–C(1′). The angular distortion is also consistent with this, as is the comparatively short C(1)–C(1′) bond. However, unlike the corresponding angles in (*II*) the Fe–C–O bond angles do not differ significantly from 180°. The butadiene and carbonyl-carbon planes [(*a*) and (*d*)] are not parallel, the angle between them being 21·0°, exactly the same as in the structure of (II).

TABLE 5

Intermolecular contacts $\leqslant$3·7 Å

| | | | |
|---|---|---|---|
| F(1) · · · F(3a$^{\mathrm{I}}$) | 2·857 | F(4b) · · · C(5$^{\mathrm{IV}}$) | 3·376 |
| F(2) · · · F(3b$^{\mathrm{II}}$) | 2·881 | F(1) · · · C(5$^{\mathrm{V}}$) | 3·401 |
| F(4a) · · O(2$^{\mathrm{III}}$) | 2·919 | O(1) · · · O(2$^{\mathrm{V}}$) | 3·407 |
| F(4a) · · · F(3b$^{\mathrm{III}}$) | 3·055 | F(3a) · · · O(1$^{\mathrm{IV}}$) | 3·426 |
| F(1) · · · F(4b$^{\mathrm{III}}$) | 3·129 | F(1) · · · O(2$^{\mathrm{V}}$) | 3·431 |
| F(4b) · · · O(1$^{\mathrm{IV}}$) | 3·156 | F(2) · · · F(2$^{\mathrm{II}}$) | 3·481 |
| O(1) · · · F(2$^{\mathrm{I}}$) | 3·180 | F(3a) · · · O(1$^{\mathrm{II}}$) | 3·487 |
| F(3b) · · · O(1$^{\mathrm{II}}$) | 3·190 | O(2) · · · F(2$^{\mathrm{VI}}$) | 3·489 |
| O(1) · · · F(1$^{\mathrm{I}}$) | 3·201 | F(1) · · · C(6$^{\mathrm{V}}$) | 3·527 |
| C(5) · · · O(2$^{\mathrm{V}}$) | 3·228 | F(3b) · · · C(5$^{\mathrm{II}}$) | 3·581 |
| F(1) · · · O(1$^{\mathrm{V}}$) | 3·235 | F(4a) · · · C(6$^{\mathrm{III}}$) | 3·615 |
| F(2) · · · F(3a$^{\mathrm{I}}$) | 3·264 | C(5) · · · F(3a$^{\mathrm{I}}$) | 3·652 |
| O(1) · · · F(3a$^{\mathrm{I}}$) | 3·332 | C(1) · · · F(4b$^{\mathrm{III}}$) | 3·691 |

The superscripts refer to the following equivalent positions:

I $-\frac{1}{2} - x, 1 - y, -\frac{1}{2} + z$
II $-x, 1 - y, -z$
III $-\frac{1}{2} + x, y, \frac{1}{2} - z$
IV $x, y, 1 + z$
V $-\frac{1}{2} + x, y, -\frac{1}{2} - z$
VI $-x, \frac{1}{2} + y, -z$

The crystal structure is illustrated in Figure 2 and shorter intermolecular distances are listed in Table 5. The shortest intermolecular contact, F(1) · · · F(3a$^{\mathrm{I}}$), is made in (II) by the analogous fluorine atoms. None of the contact distances is shorter than the sum of the van der Waals radii of the atoms concerned.

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