factors for the non-H atoms are given in Table 1,* and interatomic distances, angles and torsion angles in Table 2.

Related literature. For the preparation of the title compound see Gmelin (1971). Spectral studies and the kinetics of thermal decomposition were described by Dennenberg & Paronbourg (1972). Electrochemical studies and solvent effects were described by Chadwick, Díaz, González, Santa Ana & Yútronic (1986).

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Structure of (η^5-Cyclopentadienyl)(η^6-p-isopropylnitrobenzene)iron(II) Trifluoroacetate at 198 K

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Abstract. [Fe(C$_5$H$_5$)(C$_9$H$_{11}$NO$_2$)].C$_2$F$_3$O$_2$, (I), M_r = 399·1, monoclinic, $P2_1/c$, a = 8·995 (2), b = 10·641 (2), c = 17·538 (5) Å, β = 95·22 (2)°, V = 1671·7 (8) Å^3, Z = 4, D_x = 1·586 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 9·50 cm^{-1}, $F(000)$ = 816, T = 163 K, R = 0·0648 and wR = 0·0713 for 2502 reflections [$I \geq 3\sigma(I)$]. The phenyl ring exhibits a significant asymmetry in the C—C and Fe—C bond lengths. C(3)—C(4) and C(4)—C(5) [1·427 (6) and 1·424 (6) Å] are longer than the rest of the phenyl bonds; the longest bond with the Fe atom is that of C(4) [2·131 (4) Å]. The lengthening of these bonds may be attributed to the bulky isopropyl group and the presence of H(7) on the same side of the phenyl ring as the FeCp moiety. The phenyl ring and the cyclopentadienyl (Cp) are nearly parallel to each other [dihedral angle 1·3 (2)°], and the plane of the nitro group is twisted by an angle of 5·7 (2)° with respect to the phenyl ring. The Fe atom is closer to the phenyl ring [1·5451 (8) Å] than the Cp ring [1·6711 (9) Å]; this phenomenon was also seen in all similar compounds studied in this laboratory. Each acetyl O atom is involved in a close contact with an aromatic H atom. For C(3)—H(3)···O(3) ($1 - x$, $-0.5 + y$, $0.5 - z$): C—H = 0·86 (5), H···O = 2·59 (6), C···O = 3·207 (6) Å, C—H···O = 130 (3)°. For C(5)—H(5)···O(4) ($1 - x$, $0.5 + y$, $0.5 - z$): C—H = 0·80 (5), H···O = 2·46 (6), C···O = 3·063 (6) Å, C—H···O = 132 (3)°.

Experimental. Compound (I) was synthesized by oxidation of the (η^5-cyclopentadienyl)(η^6-p-aminoisopropylbenzene)iron(II) hexafluorophosphate complex with H$_2$O$_2$ in trifluoracetic acid according to a method similar to that reported by Sheats & Rausch (1970). Crystals were grown by slow evaporation from a mixture of acetone–iethyl ether–methylene chloride. A suitable crystal of dimensions 0·14 × 0·27 × 0·40 mm was chosen for X-ray investigation. Data were collected on a Siemens *R3m/V* diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ radiation. 50 reflections with 20·82 ≤ 2θ ≤ 26·08° were used to refine the cell parameters. 7564 reflections were collected using the ω-scan method (h, 0→11; k, −13→13; l, −22→22), 3835 unique reflections, R_{int} = 0·0259; 2θ range 3→55°, 1·2° ω

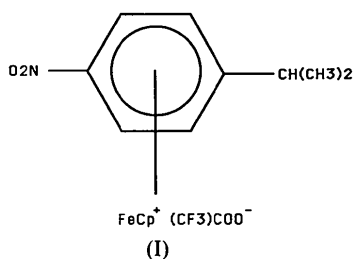Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic displacement coefficients* $(\text{Å}^2 \times 10^3)$

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe	2346 (1)	528 (1)	1631 (1)	46 (1)
F(1)	5939 (5)	1812 (4)	3865 (2)	108 (2)
F(2)	6278 (6)	−147 (4)	4050 (2)	131 (2)
F(3)	8073 (5)	1168 (4)	4326 (2)	107 (2)
O(1)	5845 (6)	1891 (6)	764 (3)	117 (2)
O(2)	6059 (6)	−145 (6)	820 (3)	111 (2)
O(3)	7713 (5)	1780 (3)	2697 (2)	87 (2)
O(4)	7596 (5)	−309 (3)	2761 (3)	82 (2)
N	5572 (6)	868 (6)	1047 (3)	84 (2)
C(1)	4618 (6)	826 (5)	1676 (3)	56 (2)
C(2)	4346 (6)	−325 (5)	2023 (3)	56 (2)
C(3)	3376 (6)	−377 (4)	2599 (3)	49 (2)
C(4)	2650 (5)	723 (4)	2844 (2)	45 (1)
C(5)	2952 (6)	1869 (4)	2468 (3)	55 (2)
C(6)	3931 (6)	1935 (5)	1899 (3)	59 (2)
C(7)	1566 (6)	705 (4)	3453 (3)	56 (2)
C(8)	2198 (10)	1446 (8)	4147 (4)	85 (3)
C(9)	1094 (9)	−613 (6)	3692 (4)	75 (2)
C(10)	752 (8)	−745 (6)	1246 (4)	89 (3)
C(11)	81 (7)	432 (10)	1424 (4)	109 (4)
C(12)	651 (7)	1302 (6)	931 (4)	82 (2)
C(13)	1610 (7)	702 (6)	499 (3)	69 (2)
C(14)	1679 (7)	−548 (5)	689 (3)	75 (2)
C(15)	7470 (6)	764 (4)	3003 (3)	61 (2)
C(16)	6947 (7)	872 (5)	3809 (3)	71 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

Fe—C(1)	2·063 (5)	Fe—C(2)	2·075 (5)
Fe—C(3)	2·093 (5)	Fe—C(4)	2·131 (4)
Fe—C(5)	2·083 (5)	Fe—C(6)	2·092 (5)
Fe—C(10)	2·042 (7)	Fe—C(11)	2·039 (6)
Fe—C(12)	2·043 (6)	Fe—C(13)	2·043 (5)
Fe—C(14)	2·054 (6)	F(1)—C(16)	1·360 (7)
F(2)—C(16)	1·328 (7)	F(3)—C(16)	1·335 (7)
O(1)—N	1·231 (9)	O(2)—N	1·243 (9)
O(3)—C(15)	1·235 (6)	O(4)—C(15)	1·227 (6)
N—C(1)	1·458 (7)	C(1)—C(2)	1·400 (7)
C(1)—C(6)	1·404 (7)	C(2)—C(3)	1·394 (7)
C(3)—C(4)	1·427 (6)	C(4)—C(5)	1·424 (6)
C(4)—C(7)	1·510 (7)	C(5)—C(6)	1·392 (8)
C(7)—C(8)	1·517 (9)	C(7)—C(9)	1·535 (8)
C(10)—C(11)	1·437 (12)	C(10)—C(14)	1·357 (10)
C(11)—C(12)	1·397 (11)	C(12)—C(13)	1·357 (9)
C(13)—C(14)	1·371 (8)	C(15)—C(16)	1·534 (8)
O(1)—N—O(2)	123·3 (6)	O(1)—N—C(1)	119·0 (6)
O(2)—N—C(1)	117·7 (6)	N—C(1)—C(2)	119·6 (5)
N—C(1)—C(6)	119·3 (5)	C(2)—C(1)—C(6)	121·0 (5)
C(1)—C(2)—C(3)	119·7 (5)	C(2)—C(3)—C(4)	121·3 (4)
C(3)—C(4)—C(5)	116·8 (4)	C(3)—C(4)—C(7)	123·1 (4)
C(5)—C(4)—C(7)	120·1 (4)	C(4)—C(5)—C(6)	122·4 (4)
C(1)—C(6)—C(5)	118·7 (4)	C(4)—C(7)—C(8)	110·0 (5)
C(4)—C(7)—C(9)	114·6 (4)	C(8)—C(7)—C(9)	110·4 (5)
C(11)—C(10)—C(14)	108·6 (6)	C(10)—C(11)—C(12)	104·9 (6)
C(11)—C(12)—C(13)	108·7 (6)	C(12)—C(13)—C(14)	109·8 (5)
C(10)—C(14)—C(13)	107·9 (6)	O(3)—C(15)—O(4)	129·7 (6)
O(3)—C(15)—C(16)	114·6 (4)	O(4)—C(15)—C(16)	115·7 (5)
F(1)—C(16)—F(2)	104·7 (5)	F(1)—C(16)—F(3)	104·3 (4)
F(2)—C(16)—F(3)	108·3 (5)	F(1)—C(16)—C(15)	112·6 (4)
F(2)—C(16)—C(15)	114·7 (4)	F(3)—C(16)—C(15)	111·5 (5)

scan at 3–6° min^{-1}, depending upon intensity. Four reflections ($\overline{23}1$, $\overline{2}1\overline{3}$, 313, 032) were measured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was < 1·08%). Absorption corrections were applied based on measured crystal faces using *SHELXTL-Plus* (Sheldrick, 1987); min. and max. transmission 0·9138 and 0·9679.

O2N—⟨benzene ring with Fe⟩—CH(CH3)2

FeCp⁺ (CF3)COO⁻

(I)

The structure was solved by the heavy-atom method (Patterson) in *SHELXTL-Plus* from which the locations of all non-H atoms were obtained. The structure was refined (*SHELX76*; Sheldrick, 1976) using full-matrix least squares. The positions of all H atoms were determined from a difference Fourier map except those of the cyclopentadienyl ligand which were calculated and fixed in idealized positions; their thermal parameters were also fixed. The non-H atoms were treated anisotropically, whereas the H atoms were refined with isotropic thermal parameters. 271 parameters were refined and $\sum w(|F_o| - |F_c|)^2$ was minimized; $w = 1/(\sigma|F_o|)^2$,

$\sigma(F_o) = 0·5kI^{-1/2}\{[\sigma(I)]^2 + (0·02I)^2\}^{1/2}$, I(intensity) $= (I_{peak} - I_{background})$(scan rate), and $\sigma(I) = (I_{peak} + I_{background})^{1/2}$(scan rate), k is the correction due to decay and Lp effects, 0·02 is a factor used to down weight intense reflections and to account for instrument instability. An extinction correction $\chi = 0·0008$ (2) {where $F^* = F[1 + 0·002\chi F^2/\sin(2\theta)]^{-1/4}$} was also applied (Sheldrick, 1987). Final $R = 0·0648$, $wR = 0·0713$ ($R_{all} = 0·0976$, $wR_{all} = 0·0784$) for 2502 reflections having $I \geq 3\sigma(I)$, and goodness-of-fit = 2·32. Maximum $\Delta/\sigma = 0·004$ in the final refinement cycle and the min. and max. peaks in the ΔF map were −0·91 and 0·73 e Å^{-3}, respectively. The linear absorption coefficient was calculated using values from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55). Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with anomalous-dispersion corrections from Cromer & Liberman (1970), while those of H atoms were from Stewart, Davidson & Simpson (1965). The positional parameters and the equivalent isotropic thermal parameters of the non-H atoms are listed in Table 1;* bond lengths and angles are in Table 2. The
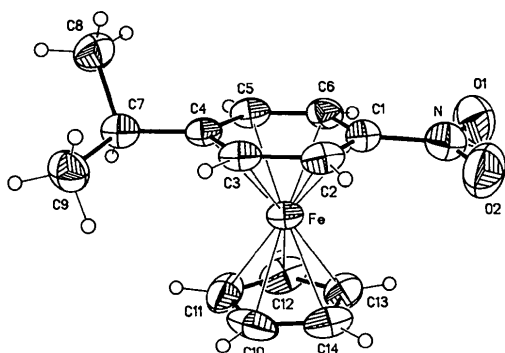
Fig. 1. 50% probability ellipsoids of the cation showing the atom-numbering scheme.

thermal ellipsoids drawing (*SHELXTL-Plus*, Sheldrick, 1987) of the cation with the atom-labelling scheme is given in Fig. 1.

Related literature. For synthesis of related compounds refer to Lee, Gill, Iqbal, Azogu & Sutherland (1982) and Sutherland, Piórko, Chowdbury & Lee (1987). For structurally similar compounds refer to Abboud, Simonsen, Piórko & Sutherland (1990, 1991), Sutherland, Piórko, Lee, Simonsen & Lynch (1988), Lynch, Thomas, Simonsen, Pióko & Sutherland (1986) and Simonsen, Lynch, Sutherland & Piórko (1985).

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Structure of Dicarbonyl(dimethylphosphonate)iodo(η⁵-pentamethylcyclopentadienyl)rhenium(III)

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Abstract. [Re(C₂H₆O₃P)(C₁₀H₁₅)(CO)₂I], [Cp*-Re(CO)₂{PO(OMe)₂}I] (Cp* = pentamethylcyclopentadienyl), $M_r = 613.4$, trigonal axes, $R\overline{3}$, $a = 36.833$ (6) $c = 7.535$ (2) Å, $V = 8853$ Å³, $Z = 18$, $D_x = 2.071$ g cm⁻³, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 79.1$ cm⁻¹, $F(000) = 5183$, room temperature, final $R = 0.044$, $wR = 0.060$ for 1608 observed reflections.

The structure may be described as a five coordinate square pyramid with the Cp ring centroid occupying the unique apical site and the CO groups mutually *trans* in the square base. The ring is closely planar with the C atoms of the methyl groups displaced on average 0.18 Å away from the mean plane on the opposite side to the Re atom. There are only weak