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Duarte *et al.* · $[Fe(C_5H_5)(C_7H_4N_2O_2)(C_{18}H_{15}O_3P)(C_3H_9P)]PF_6$

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A supramolecular zigzag chain of organometallic dipoles mediated by PF_6^- anions

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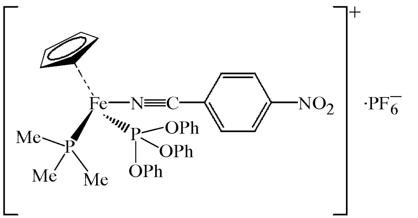
The title compound, (η^5-cyclopentadienyl)(4-nitrobenzonitrile-κN)(trimethylphosphine-κP)(triphenylphosphite-κP)iron(II) hexafluorophosphate, $[Fe(C_5H_5)(C_7H_4N_2O_2)(C_{18}H_{15}O_3P)(C_3H_9P)]PF_6$, has been characterized by spectroscopic and X-ray diffraction in order to evaluate the tuning of the electron density at the metal centre and the extension of the π delocalization on the molecule due to the presence of phosphite and phosphine co-ligands. The compound crystallizes in the centrosymmetric space group $P2_1/c$, which destroys the possibility of exhibiting any quadratic non-linear optical properties. The packing shows a supramolecular zigzag chain of antiparallel cations connected *via* the PF_6^- anions through $C-H\cdots F^{\delta-}$ interactions, with $H\cdots F$ distances ranging from 2.39 to 2.67 Å. Each zigzag chain is composed of isomeric organometallic fragments containing either *R* or *S* molecules. These chains are connected through weak intermolecular $C-H\cdots C$ interactions, forming a two-dimensional plane parallel to (101).

Comment

The design of new monocyclopentadienyl metal derivatives for application in materials science has engrossed scientists in recent years. Our interest in these compounds stems from their use as building blocks in a three-dimensional network, which would allow us to explore new variables for the engineering and development of new solids with potential non-linear optical (NLO) applications. It is well known that molecular polarization is responsible for high values of molecular hyperpolarizability, so tailoring the building blocks by modifying either the metal centre or the ligand environment will change and possibly enhance the molecular polarization (Whittall *et al.*, 1998; Nalwa, 1991; Goovaerts *et al.*, 2001). With the aim of modifying the electronic richness of the metallic centre, we have recently studied a family of iron(II) complexes $[Fe(Cp)(L)(L')(p\text{-}NCR)]BF_4$ [Cp is η^5-cyclopentadienyl; L and L' are CO, $P(OPh)_3$ or PPh_3; R is $C_6H_4NMe_2$, $C_6H_4NO_2$, (E)-$CH{=}CHC_6H_4NMe_2$ or (E)-$CH{=}CHC_6H_4NO_2$], where the organometallic fragment has been systematically enriched or depleted by changing the ligands L and L' or substituting the η^5-cyclopentadienyl ligand with η^5-indenyl (Garcia, Robalo, Teixeira *et al.*, 2001). Within these studies, we have synthesized and characterized the title novel complex, $[Fe(Cp)(PMe_3)\{P(OPh)_3\}(4\text{-}NCC_6H_4NO_2)]PF_6$, (I), and present its structural analysis here.



(I)

Complex (I) was synthesized by treatment of the precursor $[Fe(Cp)\{P(OPh)_3\}(PMe_3)I]$ with $TlPF_6$ and a slight excess of 4-nitrobenzonitrile in dichloromethane at room temperature. After work-up and recrystallization with dichloromethane–diethyl ether, complex (I) was obtained as dark-red crystals, which were fairly stable towards oxidation in air and moisture in both the solid state and solution. The formulation is supported by analytical data and IR and ^1H, ^{13}C and ^{31}P NMR spectra. In the IR spectrum, the typical ν(CN) band at 2220 cm^{-1} showed a negative shift of 12 cm^{-1} compared with the uncoordinated nitrile, as observed previously for other analogous Fe^{II} compounds (Garcia, Robalo, Dias *et al.*, 2001). In the NMR spectrum, we observed a doublet signal at 8.05 p.p.m. for the aromatic *ortho* H atoms of the nitrile ligand, which is slightly shielded when compared with the corresponding signal for the uncoordinated nitrile (8.13 p.p.m. in the same solvent). These spectroscopic data are consistent with a metal–nitrile interaction, described by a nitrile σ-type

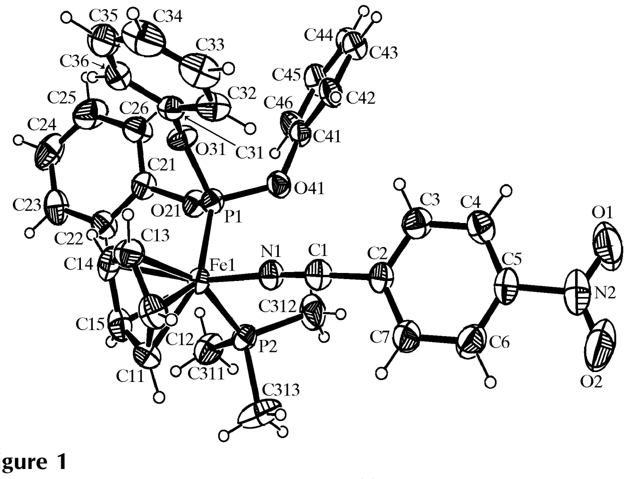


Figure 1
A view of the complex cation in (I), showing 30% probability displacement ellipsoids and the atomic labelling scheme.

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coordination with a small π back-donation contribution, owing to π-bonding between the metal *d* orbitals and the π* orbital of the CN group. The complex shows an intense broad visible absorption band at λ_{max} = 435 nm in chloroform. This absorption could be attributed to a *d*–π* metal-to-ligand charge-transfer (MLCT) transition from the Fe centre to the nitrile ligand. Such low-energy MLCT bands are typically associated with large molecular quadratic NLO responses (Garcia, Robalo, Dias *et al.*, 2001; Garcia *et al.*, 2002).

In the solid state, complex (I) crystallizes in the monoclinic centrosymmetric space group $P2_1/c$, thus destroying our hopes of obtaining dipole supramolecular alignment. The molecular structure of the cation is presented in Fig. 1. The coordination geometry can be described as a pseudo-octahedral three-legged piano stool on the assumption that the cyclopentadienyl group takes up three coordination sites. This geometry, similar to that of other compounds of the same family (Garcia, Robalo, Teixeira *et al.*, 2001), is confirmed by the angles around the metal centre, which are all close to 90° (Table 1), as well as by the remaining *X*—Fe—Cp(centroid) angles [P1—Fe1—Cp(centroid) = 124.62 (6)°, P2—Fe1—Cp(centroid) = 122.67 (6)° and N1—Fe1—Cp(centroid) = 123.4 (1)°]. As expected, all angles involving the Cp centroid are considerably larger than those involving the phosphite, phosphine and nitrile ligands.

In the molecule of (I), we observe the well known contraction of the Fe—P bond when using a phosphite ligand instead of phosphine. Thus, the observed value of 2.1206 (14) Å, which is shorter than that of the trimethylphosphine ligand [2.2332 (15) Å], can be attributed to the presence of the oxygen as the α atom of the pendent groups on the triphenylphosphite. This value agrees with the values observed for Fe(phosphine) and Fe(phosphite) derivatives in the Cambridge Structural Database (CSD, Version 5.25; Allen, 2002), presented in Table 3, where Fe—N and N≡C distances are also included for comparison.

The Fe—N distance [1.871 (4) Å] in (I) is somewhat shorter than that found in $[Fe(Cp)(CO)\{P(OPh)_3\}(4\text{-}NCC_6H_4NO_2)]BF_4$ [1.878 (10) Å; Garcia, Robalo, Teixeira *et al.*, 2001], while it is very similar to that observed in $[Fe(Cp)(dppe)(4\text{-}NCC_6H_4NO_2)]PF_6$ [1.874 (11) Å; dppe is (diphenylphosphino)ethane; Garcia, Robalo, Dias *et al.*, 2001]. The N≡C bond [1.147 (6) Å], being somewhat longer than the corresponding bonds found in the complexes listed in Table 3, approaches the structural features of the bond in the free imine [1.155 (15) Å; Higashi & Osaki, 1997]. These values, altogether with the bond angles Fe1—N1≡C1 and N1≡C1—C2 [175.4 (4) and 173.7 (5)°, respectively], show that, in the solid state, the nitrile group departs somewhat from the expected linear geometry, and there is no evidence of any appreciable π back-donation contribution. These results confirm the spectroscopic data found for (I), since only a small π back-donation effect was noticed. According to this behaviour, which can be correlated with the donor ability of the metal centre, we can identify the $[Fe(Cp)(PMe_3)\{P(OPh)_3\}]^+$ fragment as a weak π-donor towards the nitrile ligand when compared with other fragments (Table 3).

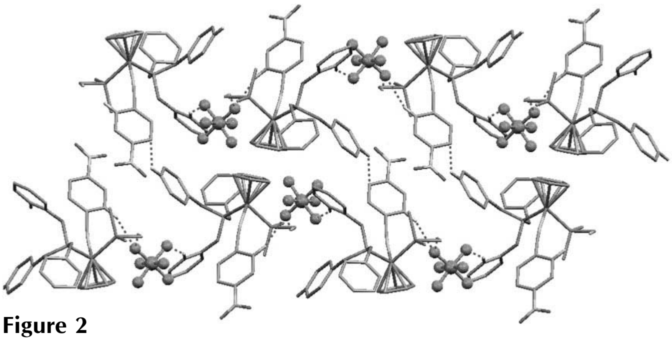

Figure 2
A view of the two-dimensional layer of zigzag chains formed by different optical isomers of the complex cation. H atoms not involved in hydrogen bonding have been omitted. Broken lines indicate C—H···F and C—H···C(π) interactions.

In recent publications, the importance of intermolecular interactions involving halogens, in particular F atoms, as a possible tool in crystal engineering has been studied in great detail (Chopra *et al.*, 2005). The three-dimensional packing of (I) shows a supramolecular organometallic zigzag chain of aligned cations (of the same conformational isomer) in an up–down configuration, obtained *via* a network of C—H···$F^{\delta-}$ interactions (Table 2) involving the F atoms of the anions and H atoms of the nitrile [H7···F6^{i}; symmetry code: (i) $1 - x, -y, 1 - z$], phosphine (H33*A*···F6^{i}) and phosphite [H42···F5^{ii}; symmetry code: (ii) $x, \frac{1}{2} - y, z - \frac{1}{2}$] (Fig. 2), generating in this way a one-dimensional chain along the *b* axis. We have taken into account the criteria used by Reichenbacher *et al.* (2005), where H···F distances up to 2.9 Å can be considered as weak intermolecular interactions. These interactions organize the complex cations into pairs through the spherical anion, in such a way that their dipole moments and second-order polarizabilities cancel. A weaker interaction of the type C—H···C(π), between a C atom of the benzene ring attached to the nitrile and a phenyl H atom of the phosphite ligand [C34—H34···C4^{iii}; symmetry code: (iii) $x, \frac{1}{2} - y, z + \frac{1}{2}$], gives rise to the formation of a two-dimensional aggregation of chains of different optical isomers of the complex cation parallel to the (101) plane.

Experimental

$TlPF_6$ (0.40 mmol) was added to a solution of $[Fe(Cp)\{P(OPh)_3\}(PMe_3)I]$ (0.40 mmol) and 4-nitrobenzonitrile (0.72 mmol) in dichloromethane (40 ml) at room temperature. The mixture was stirred at room temperature for 22 h. A colour change was observed from dark brown to red, with simultaneous precipitation of thallium iodide. The red solution was filtered, evaporated under vacuum to dryness, and washed several times with diethyl ether and *n*-hexane to remove the excess of nitrile. The dark-red residue was further purified by vapour diffusion of diethyl ether into a concentrated dichloromethane solution, affording dark-red crystals of (I) (yield 47%; m.p. 435–436 K). Analysis calculated for $C_{33}H_{33}F_6FeN_2O_5P_3$: C 49.49, H 4.16, N 3.50%; found: C, 49.44, H 4.12, N 3.31%. IR (KBr, cm^{-1}): ν(N≡C) 2221, ν(NO_2) 1526 and 1345; ^{1}H NMR (300 MHz, CD_3COCD_3): δ 1.85 (*d*, 9H, *J* = 9.0 Hz, PMe_3), 4.64 (*s*, 5H, η^5-C_5H_5), 7.23 [*m*, 3H, $P(OPh)_3$:H-*para*], 7.34–7.42 [*m*, 12H, $P(OPh)_3$:H-*ortho* and H-*meta*], 8.05 (*d*, 2H, *J* = 9.0 Hz, H2, H6), 8.41 (*d*, 2H, *J* = 9.0 Hz, H3, H5); ^{13}C{^{1}H} NMR (75 MHz, CD_3COCD_3): δ 18.85 (*d*, J_{CP} =

−29.8 Hz, PMe$_3$), 81.69 (η^5-C$_5$H$_5$), 118.29 (C1), 121.70 [*d*, $J_{\rm CP}$ = 6.8 Hz, P(OPh)$_3$:C-*ortho*], 125.28 (C3, C5), 126.17 [P(OPh)$_3$:C-*para*], 131.00 [P(OPh)$_3$:C-*meta*], 132.81 (NC), 135.00 (C2, C6), 152.44 [P(OPh)$_3$:C-*ipso*], 152.59 (C4); ^{31}P{^{1}H} NMR (75 MHz, CD$_3$COCD$_3$): δ −144.05 (*h*, $J_{\rm PF}$ = 704.4 Hz, PF$_6$), 27.93 (*d*, $J_{\rm PP}$ = 100.3 Hz, PMe$_3$), 167.93 [*d*, $J_{\rm PP}$ = 100.3 Hz, P(OPh)$_3$]. During the NMR experiment, no sign of nitrile ligand dissociation was found in deutero-acetone.

Crystal data

[Fe(C$_5$H$_5$)(C$_7$H$_4$N$_2$O$_2$)-
(C$_{18}$H$_{15}$O$_3$P)(C$_3$H$_9$P)]PF$_6$
M_r = 800.37
Monoclinic, $P2_1/c$
a = 10.4938 (6) Å
b = 18.9715 (7) Å
c = 17.8707 (11) Å
β = 98.221 (5)°
V = 3521.2 (3) Å^3
Z = 4

D_x = 1.510 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 16–21°
μ = 5.39 mm^{-1}
T = 293 (2) K
Plate, red
0.3 × 0.14 × 0.1 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: part of the refinement model (ΔF) (Parkin *et al.*, 1995)
$T_{\rm min}$ = 0.276, $T_{\rm max}$ = 0.583
6598 measured reflections
6598 independent reflections

3926 reflections with $I > 2\sigma(I)$
$\theta_{\rm max}$ = 69.8°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 21$
3 standard reflections every 400 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.150
S = 1.08
6598 reflections
451 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 1.6778P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.013
$\Delta\rho_{\rm max}$ = 0.41 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N1	1.871 (4)	P1—O21	1.615 (3)
Fe1—C15	2.055 (5)	P2—C313	1.800 (6)
Fe1—C14	2.057 (5)	P2—C311	1.809 (5)
Fe1—C13	2.102 (5)	P2—C312	1.819 (6)
Fe1—C11	2.104 (5)	N1—C1	1.147 (6)
Fe1—C12	2.112 (5)	C1—C2	1.429 (7)
Fe1—P1	2.1206 (14)	C5—N2	1.483 (7)
Fe1—P2	2.2332 (15)	N2—O1	1.208 (8)
P1—O41	1.598 (3)	N2—O2	1.213 (8)
P1—O31	1.604 (3)		
N1—Fe1—P1	95.12 (13)	C1—N1—Fe1	175.4 (4)
N1—Fe1—P2	88.55 (13)	N1—C1—C2	173.7 (5)
P1—Fe1—P2	93.08 (5)		
C4—C5—N2—O1	−10.7 (9)	C4—C5—N2—O2	172.4 (7)
C6—C5—N2—O1	169.2 (6)	C6—C5—N2—O2	−7.6 (10)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···F6$^{\rm i}$	0.93	2.39	3.278 (8)	159
C313—H33*A*···F6$^{\rm i}$	0.96	2.64	3.510 (8)	152
C42—H42···F5$^{\rm ii}$	0.93	2.67	3.546 (7)	157
C34—H34···C4$^{\rm iii}$	0.93	2.76	3.55 (1)	143

Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (iii) $x, \frac{1}{2} - y, z + \frac{1}{2}$.

Table 3
Comparative geometrical parameters (Å) for selected complexes.

Ddpe is diphenylphosphphinoethane, dppm is bis(diphenylphosphino)-methane and geometrical parameters taken from the Cambridge Structural Database are indicated without their s.u. values.

Compound	Fe—P$_{\rm PPh}$	Fe—P$_{\rm POPh}$	Fe—N	N≡C
[Fe(Cp)(dppe)(NCCH$_3$)]BPh$_4$^{a}	2.205 2.194		1.881	1.137
[Fe(Cp)(dppm)(NCCH$_3$)]PF$_6$^{b}	2.196 2.207		1.892	1.135
[Fe(acetyl-Cp)(dppe)(NCCH$_3$)]PF$_6$^{c}	2.207 2.232		1.895	1.126
[Fe(Cp*)(dppe)(NCCH$_3$)]PF$_6$^{d}	2.218 2.237		1.905	1.133
[Fe(Cp){P(OPh)$_3$}$_2$(NCCH$_3$)]PF$_6$^{e}		2.143 2.165	1.918	1.132
[Fe(Cp){P(OMe)$_3$}$_2$(NCCH$_3$)]PF$_6$^{f}		2.175 2.182	1.924	1.094
[Fe(Cp)(dppe)(NCPhNO$_2$)]-PF$_6$, (II)	2.209 (3) 2.210 (4)		1.874 (11)	1.129 (14)
[Fe(Cp)(CO){P(OPh)$_3$}(NCPh-NO$_2$)]BF$_4$, (III)		2.159 (3)	1.878 (10)	1.139 (14)
[Fe(Ind)(CO){P(OPh)$_3$}(NCPh-NO$_2$)]BF$_4$ ^{g}		2.139 (3)	1.900 (8)	1.155 (12)
[Fe(Cp)(dppp)(NCPhNO$_2$)]-PF$_6$, (IV)	2.211 (3) 2.219 (3)		1.902 (9)	1.141 (15)
[Fe(Cp)(PMe$_3$){P(OPh)$_3$}-(NCPhNO$_2$)]PF$_6$, (I)	2.233 (2)	2.121 (1)	1.871 (4)	1.147 (6)
p-NCPhNO$_2$, (V)				1.155 (15)

References: (I) this work; (II) Garcia, Robalo, Dias *et al.* (2001); (III) Garcia, Robalo, Teixeira *et al.* (2001); (IV) Wenseleers *et al.* (2003); (V) Higashi & Osaki (1977). CSD refcodes: (*a*) PEACFE (Riley *et al.*, 1978); (*b*) VEBSIE (Ruiz *et al.*, 1989); (*c*) KEJPOE (Ruiz, Gonzalez *et al.*, 1990); (*d*) KICNIT (Ruiz, Astruc *et al.*, 1990); (*e*) XATDUR (Katayama *et al.*, 2000); (*f*) JIPYAI (Schumann *et al.*, 1991); (*g*) QUSPOJ (Garcia, Robalo, Teixeira *et al.*, 2001).

Methyl H atoms were positioned geometrically, with C—H = 0.96 Å, and constrained. The H atoms of the Ph and Cp rings were also positioned geometrically, with C—H = 0.93 Å. For all H atoms, $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *XCAD4* (Harms & Wocadlo, 1995); data reduction: *XCAD4*; program(s) used to solve structure: *SIR99* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *WinGX* (Farrugia, 1999), *PLATON* (Spek, 2003) and *enCIFer* (Allen *et al.*, 2004).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1231). Services for accessing these data are described at the back of the journal.

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