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(η^5-Cyclopentadienyl)(η^6-9*H*-fluorene)iron(II) hexafluorophosphate

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Key indicators

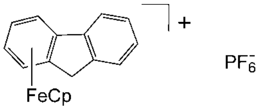
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.035
wR factor = 0.092
Data-to-parameter ratio = 10.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $[Fe(C_5H_5)(C_{13}H_{10})]PF_6$, the fluorene skeleton is disordered over two positions and parallel with the cyclopentadienyl ring.

Comment

The title compound, (I), is an important starting material, employed in the synthesis of the contentious complex (η^5-cyclopentadienyl)(η^5-fluorenyl)iron (Johnson & Treichel, 1977; Ustynyuk *et al.*, 1986).



(I)

The cyclopentadienyl and aryl rings are parallel [dihedral angle = 1.9 (3)°] and the metal atom is coordinated centrally to both rings. This is in contrast to (η^5-cyclopentadienyl)(η^6-fluorenyl)iron (Johnson & Treichel, 1977) where the Fe atom is displaced away from the ring junction between the five- and six-membered rings, leading to Fe—C distances ranging from 2.032 (5) to 2.316 (5) Å. In (I), the Fe—C distances are in the range 2.078 (14)–2.132 (6) Å, similar to the bonding pattern observed in the analogous 4*H*-cyclopenta[*def*]phenanthrene (Decken *et al.*, 1997) and *endo*-9-(2-methylphenylamino)-fluorene (Pierre *et al.*, 1998) complexes. The fluorene skeleton is disordered over two positions as previously observed in the corresponding methyl-substituted complex (η^5-cyclopentadienyl)-*exo*-(η^6-9-methyl-9*H*-fluorene)iron hexafluorophosphate (Johnson & Treichel, 1977), where publishable data could not be obtained.

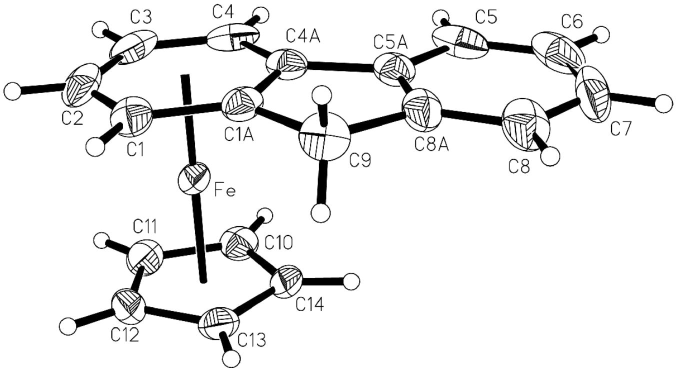


Figure 1
A view of the cation, with displacement ellipsoids drawn at the 30% probability level. Only the major component of the disordered fluorene group is shown.

Experimental

The title compound was prepared according to a published procedure (Nesmeyanov *et al*., 1966).

Crystal data

$[\mathrm{Fe(C_5H_5)(C_{13}H_{10})}]\mathrm{PF_6}$
$M_r = 432.12$
Monoclinic, $P2_1/n$
$a = 7.3373\ (4)$ Å
$b = 9.5498\ (5)$ Å
$c = 23.9163\ (12)$ Å
$\beta = 97.498\ (1)^\circ$
$V = 1661.48\ (15)$ Å^3
$Z = 4$

$D_x = 1.728$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6469 reflections
$\theta = 2.3\text{–}32.2^\circ$
$\mu = 1.07$ mm^{-1}
$T = 293\ (2)$ K
Irregular fragment, orange
$0.18 \times 0.13 \times 0.13$ mm

Data collection

Bruker AXS SMART1000/P4 diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997*a*)
$T_{\min} = 0.780$, $T_{\max} = 0.875$
13243 measured reflections

3826 independent reflections
2711 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.022$
$\theta_{\max} = 27.5^\circ$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 12$
$l = -31 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.092$
$S = 0.97$
3826 reflections
352 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}

H atoms were placed in calculated positions, with C—H distances fixed at 0.93 (CH groups) or 0.96 Å ($\mathrm{CH_2}$ groups), and U_{iso} values set at $1.2U_{\mathrm{eq}}$ of the carrier C atom. The fluorene ligand was disordered over two positions. The atomic distances in the minor component were restrained to the corresponding distances in the major component. The site occupancies were determined using an isotropic model and fixed at 0.74 (C1*A*–C9) and 0.26 (C1*A*′–C9′) in subsequent refinement cycles.

Data collection: *SMART* (Bruker, 1997–1999); cell refinement: *SAINT* (Bruker, 1997–2003); data reduction: *SAINT*); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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