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Hexakis(pyridine *N*-oxide-*κO*)iron(II) tetraphenylborate

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The Fe^{II} cation in $[Fe(C_5H_5NO)_6](C_{24}H_{20}B)_2$ has a distorted octahedral coordination with the Fe—O distances in the range 2.085 (2)–2.148 (2) Å. In the crystal structure the $[Fe(C_5H_5NO)_6]^{2+}$ cations and the BPh_4^- anions are involved in a π–π and six C—H$\cdots\pi$ intermolecular interactions between pyridine and the phenyl rings of the counter-ions.

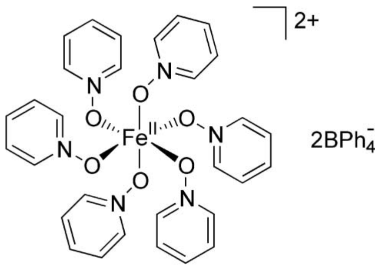
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.065
wR factor = 0.122
Data-to-parameter ratio = 16.7

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

Comment

Recent work with ruthenium dihydrogen complexes has shown that dihydrogen hydrogen bonding (DHHB) occurs in solution (Szymczak *et al.*, 2007). Further evidence for DHHB was sought by cocrystallizing a neutral hydrogen-bond acceptor, pyridine *N*-oxide, with an iron dihydrogen complex. However, our attempts at obtaining such cocrystals failed. Instead, addition of an excess of pyridine *N*-oxide resulted in the complete substitution of all ligands, forming a homoleptic pyridine *N*-oxide iron(II) complex, $[Fe(C_5H_5NO)_6](BPh_4)_2$, (I), with tetraphenylborate as a counter-ion.



(I)

The crystal structure of this compound is reported here. The perchlorate salt of this complex has been previously synthesized and structurally characterized (Taylor, 1978). The structures of numerous other homoleptic transition metal complexes containing pyridine *N*-oxide also have been determined including nickel(II), copper(II), cobalt(I), cobalt(II), cadmium(II), zinc(II), lanthanum(III) and mercury(II) (Wood, 1995; Jin *et al.*, 1992; Nieuwenhuyzen *et al.*, 1991, and references therein). The Fe^{II} cation in $[Fe(C_5H_5NO)_6](BPh_4)_2$ lies on an inversion center and has a distorted octahedral coordination with the Fe—O distances in the range 2.085 (2)–2.148 (2) Å; the average Fe—O distance is 2.12 (3) Å. In the crystal structure the $[Fe(C_5H_5NO)_6]^+$ cations and the BPh_4^- anions are involved in π–π interactions between the N2/C6–C10(x, y, z) and N2/C6—C10($-x$, $1-y$, $-z$) pyridine rings [with interplanar spacing of 3.519 (2) Å and the centroid offset (slippage) of 1.353 Å] and six C—H$\cdots\pi$ intermolecular interactions between pyridine and the phenyl rings of the counter-ions (Table 2).

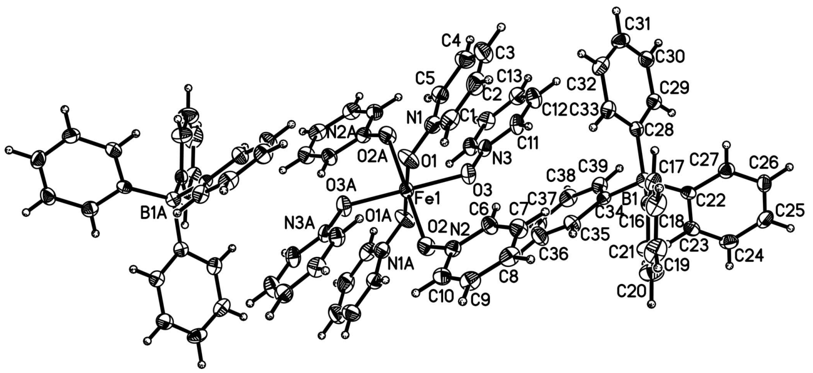


Figure 1

The molecular structure of $[Fe(C_5H_5NO)_6](BPh_4)_2$ with 50% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: (A) $1 - x, 1 - y, -z$.]

Experimental

The precursor complex, $[Fe(DMeOPrPE)_2H(H_2)](BPh_4)$ {DMeOPrPE = 1,2-bis[bis(methoxypropyl)phosphino]ethane}, was prepared by reported procedures, using $NaBPh_4$ instead of $TlPF_6$ (Gilbertson *et al.*, 2007). The title compound was prepared by adding an excess of pyridine *N*-oxide to a solution of $[Fe(DMeOPrPE)_2H(H_2)](BPh_4)$ in THF. The resulting solution was layered with hexanes and allowed to stand at room temperature for one week. $[Fe(C_5H_5NO)_6](BPh_4)_2$ formed as pale-yellow plates.

Crystal data

$[Fe(C_5H_5NO)_6](C_{24}H_{20}B)_2$
$M_r = 1264.86$
Monoclinic, $P2_1/n$
$a = 9.8939$ (16) Å
$b = 14.909$ (2) Å
$c = 21.963$ (4) Å
$\beta = 93.846$ (4)°
$V = 3232.5$ (9) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.29$ mm^{-1}
$T = 173$ (2) K
0.28 × 0.18 × 0.02 mm

Data collection

Bruker SMART CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1995)
$T_{min} = 0.922$, $T_{max} = 0.924$ (expected range = 0.992–0.994)
35624 measured reflections
7049 independent reflections
4094 reflections with $I > 2\sigma(I)$
$R_{int} = 0.122$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.122$
$S = 1.05$
7049 reflections
421 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Fe1—O3	2.085 (2)	O1—N1	1.333 (3)
Fe1—O1	2.131 (2)	O2—N2	1.341 (3)
Fe1—O2	2.148 (2)	O3—N3	1.334 (3)
O3—Fe1—O1	89.81 (8)	$O1^i$—Fe1—O2	93.57 (8)
$O3^i$—Fe1—O1	90.19 (8)	N1—O1—Fe1	125.38 (17)
O3—Fe1—O2	89.96 (8)	N2—O2—Fe1	125.55 (17)
$O3^i$—Fe1—O2	90.04 (8)	N3—O3—Fe1	131.26 (16)
O1—Fe1—O2	86.43 (8)		

Symmetry code: (i) $-x + 1, -y + 1, -z$.

Table 2

Geometry of intermolecular C—H···π interactions (Å, °).

C—H···π	H···π	C···π	C—H···π
C1—H1*A*···$Cg2^i$	2.89	3.612 (3)	133
C2—H2*A*···$Cg3^i$	2.78	3.604 (4)	146
C8—H8*A*···$Cg4^i$	2.65	3.454 (3)	143
C12—H12*A*···*Cg*4	2.82	3.583 (4)	138
C14—H14*A*···$Cg3^{ii}$	2.65	3.481 (3)	147
C24—H24*A*···$Cg1^{iii}$	2.94	3.562 (3)	124

Symmetry codes: (i) $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (ii) $1 - x, 2 - y, -z$; (iii) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$. *Cg* is the centroid of a six-membered ring (*Cg*1: N3/C11–C15; *Cg*2: C22–C27; *Cg*3: C28–C33; *Cg*4: C34–C39).

Crystals of $[Fe(C_5H_5NO)_6](BPh_4)_2$ are very thin plates and as a result reflections at high angles were very weak and R_{int} is relatively high. H atoms were positioned geometrically and refined in the riding-model approximation; C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2005); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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