Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

μ-Oxido-bis{chlorido[tris(2-pyridylmethyl)amine]iron(III)} bis(hexafluorophosphate)

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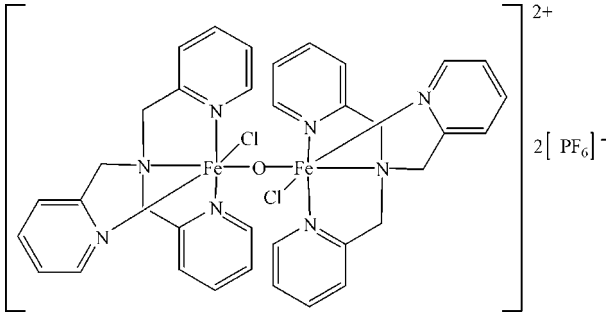
Received 3 October 2007; accepted 10 October 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.029; wR factor = 0.107; data-to-parameter ratio = 13.6.

The dinuclear Fe^{III} complex in the title compound, $[Fe_2Cl_2O(C_{18}H_{18}N_4)_2](PF_6)_2$, lies on a center of inversion. The Fe^{III} atom is chelated by a tetradentate tris(2-pyridylmethyl)amine ligand *via* four N atoms and further coordinated by one chloride ion and one bridging oxide O atom, giving a distorted octahedral coordination geometry.

Related literature

For related literature, see: Scapin *et al.* (1997); Okabe & Oya (2000); Serre *et al.* (2005).



Experimental

Crystal data

$[Fe_2Cl_2O(C_{18}H_{18}N_4)_2](PF_6)_2$
M_r = 1069.27
Triclinic, $P\overline{1}$
a = 8.5480 (17) Å
b = 11.280 (2) Å
c = 12.829 (3) Å
α = 115.49 (3)°
β = 107.44 (3)°
γ = 91.52 (3)°
V = 1047.7 (4) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.99 mm^{-1}
T = 293 (2) K
0.43 × 0.28 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.675, T_{max} = 0.811
8741 measured reflections
3903 independent reflections
3622 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.107
S = 1.00
3903 reflections
287 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

The authors thank Liaocheng University for financial support and Professor Jianmin Dou for his help.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2216).

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  doi:10.1107/S1600536807049665

supplementary materials

Acta Cryst. (2007). E**63**, m2722 [**doi:10.1107/S1600536807049665**]

μ-Oxido-bis{chloride[tris(2-pyridylmethyl)amine]iron(III)} bis(hexafluoridophosphate)

Y. Liu, J. Dou, D. Li and X. Zhang

Comment

In recent years, *N*-heterocycle ligands have been widely used as polydentate ligands which show various metal chelation reactions (Scapin *et al.*, 1997; Okabe & Oya, 2000; Serre *et al.*, 2005). In this paper, we report the structure of the title compound, (I).

As shown in Fig. 1, the Fe^{III} atom is chelated by the tetradentate ligand of tris(2-pyridylmethyl)amine *via* four N atoms, and further bonded by one chloride ion and one bridging oxygen atom. Along the axial site, two Fe^{III} atoms are linked into dimer *via* one oxygen atom (Fig. 2).

Experimental

A mixture of iron(III) trichloride (1 mmol) and tris(2-pyridylmethyl)amine (1 mmol) in 20 ml me thanol was refluxed for 2 h. The cooled solution was filterated and the filtrate was evaporated naturally at room temperature. Two days later, brown blocks of (I) were obtained with a yield of 10%. Anal. Calc. for $C_{36}H_{36}Cl_2F_{12}Fe_2N_8OP_2$: C 40.41, H 3.37, N 10.48%; Found: C 40.37, H 3.39, N 10.43%.

Refinement

All H atoms were placed in calculated positions (C—H = 0.93 or 0.97 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

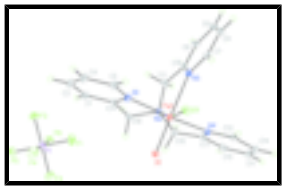

Fig. 1. The asymmetric unit of the title compound, drawn with 30% probability displacement ellipsoids for the non-hydrogen atoms.

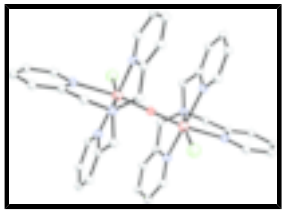

Fig. 2. The dinuclear Fe^{III} complex of the title compound. H atoms have been omitted.

μ-Oxido-bis{chlorido[tris(2-pyridylmethyl)amine]iron(III)} bis(hexafluoridophosphate)

Crystal data

$[Fe_2Cl_2O(C_{18}H_{18}N_4)_2](PF_6)_2$	$Z = 1$
$M_r = 1069.27$	$F_{000} = 540$
Triclinic, $P\overline{1}$	$D_x = 1.695$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.5480$ (17) Å	Cell parameters from 3903 reflections
$b = 11.280$ (2) Å	$\theta = 3.0$–25.5º
$c = 12.829$ (3) Å	$\mu = 0.99$ mm^{-1}
$\alpha = 115.49$ (3)º	$T = 293$ (2) K
$\beta = 107.44$ (3)º	Block, brown
$\gamma = 91.52$ (3)º	0.43 × 0.28 × 0.22 mm
$V = 1047.7$ (4) Å^3	

Data collection

Bruker APEXII CCD area-detector diffractometer	3903 independent reflections
Radiation source: fine-focus sealed tube	3622 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.021$
$T = 293$(2) K	$\theta_{max} = 25.5$º
φ and ω scans	$\theta_{min} = 3.0$º
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	$h = -8 \rightarrow 10$
$T_{min} = 0.675$, $T_{max} = 0.811$	$k = -13 \rightarrow 13$
8741 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.029$	H-atom parameters constrained
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0834P)^2 + 0.2007P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
3903 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
287 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	1.03741 (3)	0.34679 (3)	0.39270 (2)	0.03262 (13)
C1	0.9133 (4)	0.5294 (2)	0.1545 (2)	0.0555 (6)
H1	0.8141	0.5476	0.1144	0.067*
C2	1.0635 (4)	0.5737 (3)	0.1516 (2)	0.0667 (8)
H2	1.0659	0.6246	0.1109	0.080*
C3	0.7462 (3)	0.1753 (2)	0.1448 (2)	0.0520 (6)
H3A	0.7443	0.2011	0.0815	0.062*
H3B	0.6367	0.1253	0.1211	0.062*
C4	1.2074 (4)	0.5439 (3)	0.2073 (3)	0.0668 (7)
H4	1.3081	0.5725	0.2041	0.080*
C5	1.2014 (3)	0.4705 (3)	0.2686 (2)	0.0558 (6)
H5	1.2994	0.4493	0.3069	0.067*
C6	0.9542 (4)	−0.1238 (2)	0.0605 (2)	0.0563 (6)
H6	0.9321	−0.2120	0.0001	0.068*
C7	1.1346 (3)	0.0609 (2)	0.2407 (2)	0.0483 (5)
H7	1.2380	0.0967	0.3031	0.058*
C8	0.8350 (3)	−0.0449 (2)	0.0586 (2)	0.0479 (5)
H8	0.7308	−0.0793	−0.0031	0.057*
C9	1.1065 (3)	−0.0703 (2)	0.1529 (2)	0.0559 (6)
H9	1.1894	−0.1217	0.1563	0.067*
C10	0.6720 (3)	0.2875 (2)	0.3311 (2)	0.0448 (5)
H10A	0.5634	0.2354	0.2732	0.054*
H10B	0.6561	0.3761	0.3820	0.054*
C11	0.7289 (3)	0.1177 (3)	0.5343 (2)	0.0550 (6)
H11	0.6661	0.0789	0.5631	0.066*
C12	0.9878 (3)	0.1900 (2)	0.53063 (19)	0.0408 (4)
H12	1.1033	0.1989	0.5572	0.049*
C13	0.9147 (3)	0.4569 (2)	0.21892 (17)	0.0419 (5)
C14	0.8988 (3)	0.1304 (2)	0.5744 (2)	0.0490 (5)
H14	0.9529	0.0995	0.6297	0.059*
C15	0.6513 (3)	0.1628 (2)	0.4510 (2)	0.0507 (5)
H15	0.5357	0.1526	0.4218	0.061*
C16	0.7606 (3)	0.4151 (2)	0.23721 (19)	0.0426 (5)

H16A	0.7431	0.4888	0.3056	0.051*
H16B	0.6645	0.3919	0.1640	0.051*
C17	0.7467 (2)	0.22294 (19)	0.41166 (19)	0.0376 (4)
C18	0.8712 (3)	0.0858 (2)	0.14890 (18)	0.0376 (4)
Cl1	1.31395 (6)	0.35084 (6)	0.48437 (5)	0.05048 (18)
F1	0.5830 (3)	0.6609 (2)	0.1918 (2)	0.1015 (7)
F2	0.6042 (3)	0.7201 (2)	0.04869 (17)	0.0865 (6)
F3	0.4872 (2)	0.8487 (2)	0.27954 (15)	0.0822 (5)
F4	0.7327 (2)	0.8614 (2)	0.24929 (18)	0.0928 (7)
F5	0.5110 (2)	0.90780 (17)	0.13816 (17)	0.0758 (5)
F6	0.36000 (19)	0.71100 (16)	0.08030 (14)	0.0660 (4)
N1	1.0560 (2)	0.42830 (18)	0.27430 (16)	0.0430 (4)
N2	0.7779 (2)	0.29828 (16)	0.26251 (15)	0.0373 (4)
N3	1.0195 (2)	0.13923 (16)	0.23996 (15)	0.0388 (4)
N4	0.9131 (2)	0.23558 (16)	0.45116 (15)	0.0359 (4)
O1	1.0000	0.5000	0.5000	0.0375 (4)
P1	0.54888 (7)	0.78429 (6)	0.16556 (5)	0.04801 (18)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.02598 (19)	0.03690 (19)	0.03504 (19)	0.00687 (11)	0.00891 (12)	0.01758 (13)
C1	0.0802 (18)	0.0457 (12)	0.0422 (12)	0.0145 (11)	0.0187 (11)	0.0230 (10)
C2	0.105 (2)	0.0507 (13)	0.0512 (14)	0.0005 (14)	0.0321 (14)	0.0270 (11)
C3	0.0475 (13)	0.0445 (11)	0.0411 (11)	0.0094 (10)	−0.0014 (9)	0.0106 (9)
C4	0.081 (2)	0.0650 (16)	0.0624 (15)	−0.0025 (14)	0.0340 (14)	0.0309 (13)
C5	0.0541 (14)	0.0630 (15)	0.0572 (14)	0.0042 (11)	0.0247 (11)	0.0303 (12)
C6	0.0718 (17)	0.0396 (11)	0.0564 (13)	0.0117 (10)	0.0302 (12)	0.0155 (10)
C7	0.0428 (12)	0.0498 (12)	0.0541 (13)	0.0145 (9)	0.0183 (10)	0.0241 (10)
C8	0.0520 (13)	0.0426 (11)	0.0436 (11)	0.0015 (9)	0.0160 (9)	0.0159 (9)
C9	0.0580 (15)	0.0516 (13)	0.0672 (15)	0.0246 (11)	0.0326 (12)	0.0273 (11)
C10	0.0268 (9)	0.0536 (12)	0.0556 (12)	0.0086 (8)	0.0088 (8)	0.0301 (10)
C11	0.0557 (14)	0.0597 (14)	0.0606 (14)	0.0039 (11)	0.0261 (11)	0.0336 (12)
C12	0.0378 (11)	0.0424 (10)	0.0424 (10)	0.0088 (8)	0.0112 (8)	0.0212 (8)
C13	0.0544 (13)	0.0383 (10)	0.0302 (9)	0.0091 (9)	0.0141 (8)	0.0136 (8)
C14	0.0553 (14)	0.0507 (12)	0.0479 (12)	0.0100 (10)	0.0176 (10)	0.0289 (10)
C15	0.0356 (12)	0.0546 (13)	0.0648 (14)	0.0052 (9)	0.0182 (10)	0.0294 (11)
C16	0.0445 (11)	0.0442 (11)	0.0380 (10)	0.0153 (9)	0.0083 (8)	0.0214 (9)
C17	0.0293 (9)	0.0367 (9)	0.0429 (10)	0.0050 (7)	0.0115 (8)	0.0157 (8)
C18	0.0387 (10)	0.0392 (10)	0.0361 (9)	0.0050 (8)	0.0134 (8)	0.0181 (8)
Cl1	0.0289 (3)	0.0560 (3)	0.0553 (3)	0.0120 (2)	0.0086 (2)	0.0194 (3)
F1	0.0956 (15)	0.1158 (16)	0.158 (2)	0.0535 (13)	0.0633 (15)	0.1043 (16)
F2	0.0924 (13)	0.0893 (12)	0.0793 (11)	0.0075 (10)	0.0537 (10)	0.0245 (10)
F3	0.0766 (12)	0.1077 (14)	0.0562 (9)	0.0051 (10)	0.0312 (8)	0.0274 (9)
F4	0.0410 (9)	0.1444 (19)	0.0804 (12)	−0.0076 (10)	0.0008 (8)	0.0552 (12)
F5	0.0739 (11)	0.0658 (10)	0.0922 (12)	0.0048 (8)	0.0181 (9)	0.0476 (9)
F6	0.0484 (8)	0.0654 (9)	0.0676 (9)	−0.0062 (7)	0.0112 (7)	0.0228 (7)
N1	0.0468 (10)	0.0444 (9)	0.0411 (9)	0.0069 (8)	0.0173 (7)	0.0214 (7)

N2	0.0316 (8)	0.0391 (8)	0.0376 (8)	0.0087 (7)	0.0065 (6)	0.0178 (7)
N3	0.0356 (9)	0.0411 (9)	0.0406 (9)	0.0097 (7)	0.0135 (7)	0.0191 (7)
N4	0.0304 (8)	0.0377 (8)	0.0397 (8)	0.0072 (6)	0.0109 (7)	0.0187 (7)
O1	0.0333 (10)	0.0385 (10)	0.0382 (10)	0.0076 (8)	0.0095 (8)	0.0172 (8)
P1	0.0379 (3)	0.0613 (4)	0.0485 (3)	0.0062 (3)	0.0134 (2)	0.0296 (3)

Geometric parameters (Å, °)

Fe1—O1	1.7969 (7)	C10—N2	1.474 (3)
Fe1—N4	2.1173 (18)	C10—C17	1.511 (3)
Fe1—N1	2.1254 (19)	C10—H10A	0.9700
Fe1—N2	2.2223 (19)	C10—H10B	0.9700
Fe1—N3	2.2798 (19)	C11—C14	1.368 (4)
Fe1—Cl1	2.2923 (9)	C11—C15	1.379 (4)
C1—C2	1.382 (4)	C11—H11	0.9300
C1—C13	1.388 (3)	C12—N4	1.338 (3)
C1—H1	0.9300	C12—C14	1.378 (3)
C2—C4	1.356 (5)	C12—H12	0.9300
C2—H2	0.9300	C13—N1	1.331 (3)
C3—N2	1.487 (3)	C13—C16	1.503 (3)
C3—C18	1.494 (3)	C14—H14	0.9300
C3—H3A	0.9700	C15—C17	1.376 (3)
C3—H3B	0.9700	C15—H15	0.9300
C4—C5	1.374 (4)	C16—N2	1.488 (3)
C4—H4	0.9300	C16—H16A	0.9700
C5—N1	1.352 (3)	C16—H16B	0.9700
C5—H5	0.9300	C17—N4	1.340 (3)
C6—C9	1.372 (4)	C18—N3	1.343 (3)
C6—C8	1.372 (4)	F1—P1	1.581 (2)
C6—H6	0.9300	F2—P1	1.5824 (18)
C7—N3	1.340 (3)	F3—P1	1.5847 (18)
C7—C9	1.379 (3)	F4—P1	1.5782 (19)
C7—H7	0.9300	F5—P1	1.5959 (17)
C8—C18	1.380 (3)	F6—P1	1.6059 (17)
C8—H8	0.9300	O1—Fe1^{i}	1.7969 (7)
C9—H9	0.9300		
O1—Fe1—N4	90.93 (5)	N4—C12—H12	119.0
O1—Fe1—N1	92.68 (5)	C14—C12—H12	119.0
N4—Fe1—N1	154.66 (7)	N1—C13—C1	121.1 (2)
O1—Fe1—N2	91.68 (6)	N1—C13—C16	116.61 (18)
N4—Fe1—N2	78.42 (7)	C1—C13—C16	122.2 (2)
N1—Fe1—N2	76.41 (7)	C11—C14—C12	118.3 (2)
O1—Fe1—N3	166.76 (5)	C11—C14—H14	120.8
N4—Fe1—N3	82.06 (7)	C12—C14—H14	120.8
N1—Fe1—N3	89.03 (7)	C17—C15—C11	119.2 (2)
N2—Fe1—N3	75.96 (7)	C17—C15—H15	120.4
O1—Fe1—Cl1	103.05 (5)	C11—C15—H15	120.4
N4—Fe1—Cl1	103.64 (5)	N2—C16—C13	110.29 (17)
N1—Fe1—Cl1	99.94 (6)	N2—C16—H16A	109.6

N2—Fe1—Cl1	165.03 (5)	C13—C16—H16A	109.6
N3—Fe1—Cl1	89.56 (6)	N2—C16—H16B	109.6
C2—C1—C13	118.2 (3)	C13—C16—H16B	109.6
C2—C1—H1	120.9	H16A—C16—H16B	108.1
C13—C1—H1	120.9	N4—C17—C15	120.9 (2)
C1—C2—C4	120.7 (2)	N4—C17—C10	116.74 (18)
C1—C2—H2	119.6	C15—C17—C10	122.17 (19)
C4—C2—H2	119.6	N3—C18—C8	122.3 (2)
N2—C3—C18	114.64 (17)	N3—C18—C3	117.46 (18)
N2—C3—H3A	108.6	C8—C18—C3	120.20 (19)
C18—C3—H3A	108.6	C13—N1—C5	119.9 (2)
N2—C3—H3B	108.6	C13—N1—Fe1	114.83 (15)
C18—C3—H3B	108.6	C5—N1—Fe1	124.38 (17)
H3A—C3—H3B	107.6	C10—N2—C3	112.59 (18)
C5—C4—C2	118.7 (3)	C10—N2—C16	112.68 (16)
C5—C4—H4	120.7	C3—N2—C16	109.10 (17)
C2—C4—H4	120.7	C10—N2—Fe1	104.81 (12)
N1—C5—C4	121.4 (3)	C3—N2—Fe1	113.27 (13)
N1—C5—H5	119.3	C16—N2—Fe1	104.09 (12)
C4—C5—H5	119.3	C7—N3—C18	117.61 (18)
C9—C6—C8	118.9 (2)	C7—N3—Fe1	126.10 (15)
C9—C6—H6	120.5	C18—N3—Fe1	115.74 (14)
C8—C6—H6	120.5	C12—N4—C17	119.66 (18)
N3—C7—C9	123.0 (2)	C12—N4—Fe1	125.24 (14)
N3—C7—H7	118.5	C17—N4—Fe1	114.81 (14)
C9—C7—H7	118.5	Fe1—O1—Fe1^{i}	180.000 (17)
C18—C8—C6	119.4 (2)	F3—P1—F1	90.06 (13)
C18—C8—H8	120.3	F3—P1—F4	91.04 (11)
C6—C8—H8	120.3	F1—P1—F4	91.70 (14)
C6—C9—C7	118.8 (2)	F3—P1—F2	177.94 (12)
C6—C9—H9	120.6	F1—P1—F2	91.05 (12)
C7—C9—H9	120.6	F4—P1—F2	90.66 (12)
N2—C10—C17	112.31 (17)	F3—P1—F5	89.67 (11)
N2—C10—H10A	109.1	F1—P1—F5	178.99 (11)
C17—C10—H10A	109.1	F4—P1—F5	89.27 (12)
N2—C10—H10B	109.1	F2—P1—F5	89.19 (12)
C17—C10—H10B	109.1	F3—P1—F6	88.48 (10)
H10A—C10—H10B	107.9	F1—P1—F6	90.43 (12)
C14—C11—C15	119.8 (2)	F4—P1—F6	177.81 (12)
C14—C11—H11	120.1	F2—P1—F6	89.78 (10)
C15—C11—H11	120.1	F5—P1—F6	88.59 (10)
N4—C12—C14	122.0 (2)		

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1.

Fig. 1

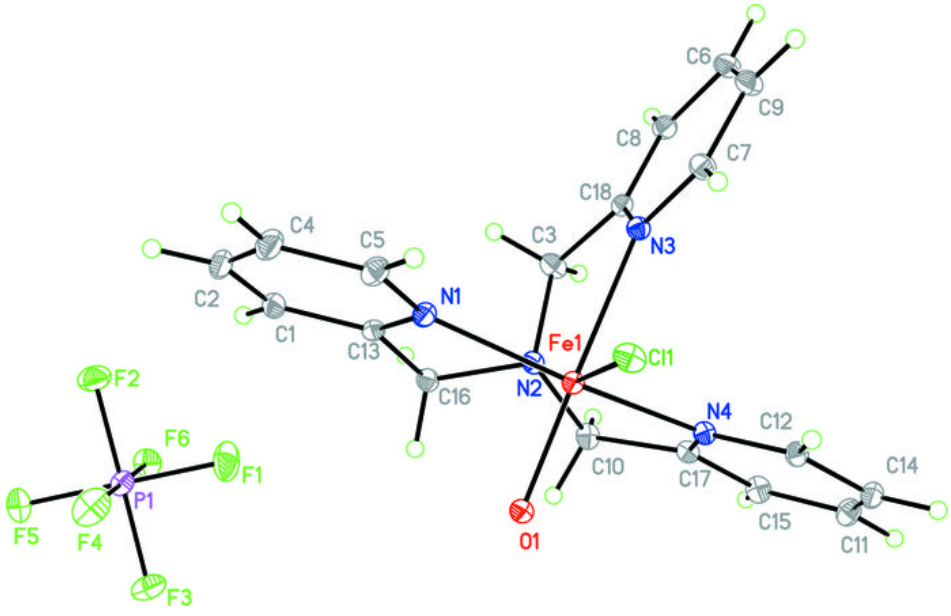
C6
C9
C8
C7
C18
N3
C3
C4
C5
C2
C1
N1
C13
C16
Fe1
N2
Cl1
N4
C12
C10
C17
C14
C15
C11
O1
F2
F6
F1
P1
F5
F4
F3

Fig. 2