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Tetraethylammonium dichlorido[*N*,*N*′-(4,5-dichloro-*o*-phenylene)bis(4-*tert*-butylpyridine-2-carboxamide)-κ^4N]-ferrate(III) acetonitrile solvate

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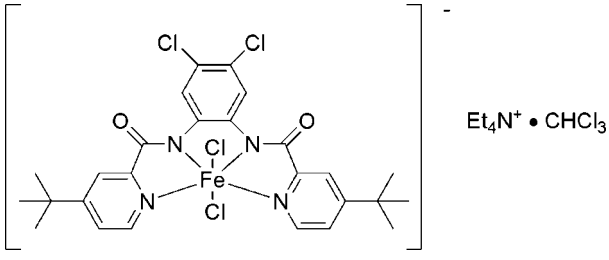
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.064; wR factor = 0.202; data-to-parameter ratio = 21.2.

In the title compound, $(C_8H_{20}N)[Fe(C_{26}H_{26}Cl_2N_4O_2)Cl_2]\cdot CHCl_3$, conventionally abbreviated as $[NEt_4][Fe(bbpc)Cl_2]\cdot CHCl_3$ [H_2bbpc = *N*,*N*′-(4,5-dichloro-*o*-phenylene)bis(4-*tert*-butylpyridine-2-carboxamide)], the Fe^{III} ion is six-coordinate, forming a distorted octahedron with two pyridine and two deprotonated amide N atoms in the equatorial plane, while the two axial sites are occupied by two Cl atoms.

Related literature

For related literature, see: Bartos *et al.* (1995); Che *et al.* (1992); Collins *et al.* (1992); Liu *et al.* (2006); Marlin *et al.* (1999); Patra & Mukherjee (1999); Ray *et al.* (1993); Yan *et al.* (2006); Yang *et al.* (1991).



Experimental

Crystal data

$(C_8H_{20}N)[Fe(C_{26}H_{26}Cl_2N_4O_2)Cl_2]\cdot CHCl_3$
M_r = 873.78
Monoclinic, $P2_1/c$
a = 11.1740 (17) Å
b = 26.598 (4) Å
c = 15.000 (2) Å
β = 108.380 (3)°
V = 4230.9 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.84 mm^{-1}
T = 294 (2) K
0.38 × 0.16 × 0.12 mm

Data collection

Bruker *SMART* CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.742, T_{max} = 0.906
28612 measured reflections
9757 independent reflections
4586 reflections with $I > 2\sigma(I)$
R_{int} = 0.082

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.202
S = 1.01
9757 reflections
461 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2152).

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supplementary materials

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Tetraethylammonium dichlorido[*N*,*N*'-(4,5-dichloro-*o*-phenylene)bis(4-*tert*-butylpyridine-2-carboxamide)-κ^4N]ferrate(III) acetonitrile solvate

X.-B. Xu, L. Yang, Y.-Y. Qu, L.-X. Li and X.-G. Zhou

Comment

There has been a growing interest in the coordination chemistry of peptide complexes of iron in a +3 or higher oxidation state (Marlin *et al.*, 1999; Che *et al.*, 1992). Mascharak and Collins have provided examples of structurally characterized complexes with coordinated carboxamido N atoms as a part of macrocyclic and nonmacrocyclic ligand framework, while high valent iron species have been proposed on the basis of catalytic studies for a variety of oxidation systems (Marlin *et al.*, 1999; Collins *et al.*, 1992; Bartos *et al.*, 1995). As part of our studies on catalysis by N4 non-porphyrin complexes (Liu *et al.*, 2006; Yan *et al.*, 2006), we report here the synthesis and structures of the iron^{III} complex with 1,2-bis(4'-*tert*-butylpyridine-2'-carboxamido)-4,5-dichlorobenzene (H_2bbpc).

In the title compound, the iron^{III} ion is six-coordinated forming a distorted octahedron. Two pyridine and two deprotonated amide nitrogen atoms of the ligand are in the equatorial plane whereas the two axial sites are occupied by Cl atoms (Fig. 1) as usually observed with a Cl(1)—Fe(1)—Cl(2) angle of 151.33°, which is similar to 152.3° of [Et_3NH][Fe(III)(bpb)Cl_2] (Yang *et al.*, 1991). The Fe—*N*(amide) and Fe—*N*(pyridine) distances at 2.0455 (19) Å and 2.170 (2) Å are also comparable to those of [Et_3NH][Fe(III)(bpb)Cl_2] (Yang *et al.*, 1991) and [Fe(III)(bpc)Cl(DMF)] (Patra *et al.*, 1999).

Experimental

The iron^{III} complex [NEt_4][Fe(bbpc)Cl_2] was prepared according to the literature (Ray *et al.*, 1993). Single crystals suitable for X-ray analysis were obtained by slow diffusion of ether into chloroform solution. Selected IR data (KBr, cm^{-1}): 2968 (*m*), 1626 (amide I band, s), 1596 (amide II band, s), 1375 (*s*), 1171 (*m*), 957 (*m*), 853 (*m*). Anal calculated for $C_{34}H_{46}Cl_4N_5O_2Fe$: C, 54.13; H, 6.15; N, 9.28. Found: C, 53.87; H, 6.36; N, 9.05. MS (FAB): 624 ([Fe(bbpc)Cl_2]$^+$).

Refinement

All H atoms were fixed geometrically and treated as riding on their parent C atoms with C—H = 0.93 Å (aromatic), 0.97 Å (methylene) and 0.96Å (methyl) with U_{iso}(H) = 1.2U_{eq}(aromatic, methylene) or U_{iso}(H) = 1.5U_{eq}(methyl). One of the tertiobutyl group is disordered over two positions with occupancy in the ratio 0.39/0.61. This disorder was treated applying restraints to keep a reasonable chemical structure. The Cl atoms in the solvent molecule present large ellipsoids components but any attempts to modelize a reasonable model failed.

Figures

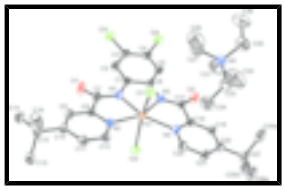


Fig. 1. A view of compound (I) including the complex anion and the cation. The chlrophorm solvent molecule and the H atoms were omitted for clarity. Displacement ellipsoids are drawn at the 30% propability level.

Tetraethylammonium dichlorido[*N*,*N*'-(4,5-dichloro-*o*-phenylene)bis(4-*tert*- butylpyridine-2-carboxamide)-κ^4*N*]iron(III) acetonitrile solvate

Crystal data

$(C_8H_{20}N)[Fe(C_{26}H_{26}Cl_2N_4O_2)Cl_2]\cdot CHCl_3$	F_{000} = 1812
M_r = 873.78	D_x = 1.372 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 8783 reflections
a = 11.1740 (17) Å	θ = 1–27.5º
b = 26.598 (4) Å	μ = 0.84 mm^{-1}
c = 15.000 (2) Å	T = 294 (2) K
β = 108.380 (3)º	Needle, green
V = 4230.9 (11) Å^3	0.38 × 0.16 × 0.12 mm
Z = 4	

Data collection

Bruker CCD area-detector diffractometer	9757 independent reflections
Radiation source: fine-focus sealed tube	4586 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.082
T = 294(2) K	θ_{max} = 27.6º
φ and ω scans	h = −14→14
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	k = −34→24
T_{min} = 0.742, T_{max} = 0.906	l = −19→19
28612 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.064	H-atom parameters constrained
$wR(F^2)$ = 0.202	$w = 1/[\sigma^2(F_o^2) + (0.0902P)^2 + 1.0929P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001

9757 reflections	$\Delta\rho_{max} = 0.82$ e Å^{-3}
461 parameters	$\Delta\rho_{min} = -0.72$ e Å^{-3}
6 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2$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}	Occ. (<1)
Fe1	0.51046 (6)	0.68484 (2)	0.42776 (4)	0.03555 (19)	
Cl1	0.69772 (11)	0.66071 (5)	0.40275 (9)	0.0552 (3)	
Cl2	0.33154 (12)	0.73891 (5)	0.39800 (10)	0.0592 (4)	
Cl3	0.39772 (15)	0.47520 (5)	0.66487 (10)	0.0738 (4)	
Cl4	0.56072 (17)	0.54520 (5)	0.82960 (9)	0.0791 (5)	
O1	0.2920 (4)	0.55604 (14)	0.3533 (2)	0.0769 (12)	
O2	0.6434 (4)	0.71324 (13)	0.7157 (2)	0.0691 (11)	
N1	0.4310 (3)	0.61699 (13)	0.4378 (2)	0.0403 (9)	
N2	0.4232 (3)	0.66324 (13)	0.2817 (2)	0.0384 (8)	
N3	0.5568 (3)	0.67498 (12)	0.5709 (2)	0.0394 (9)	
N4	0.6103 (3)	0.75347 (13)	0.4854 (2)	0.0389 (9)	
C1	0.4538 (4)	0.59680 (16)	0.5284 (3)	0.0392 (10)	
C2	0.4177 (5)	0.54949 (17)	0.5499 (3)	0.0486 (12)	
H2	0.3716	0.5284	0.5019	0.058*	
C3	0.4497 (5)	0.53371 (17)	0.6416 (3)	0.0469 (12)	
C4	0.5199 (5)	0.56432 (17)	0.7140 (3)	0.0494 (12)	
C5	0.5564 (5)	0.61223 (16)	0.6936 (3)	0.0455 (11)	
H5	0.6024	0.6331	0.7421	0.055*	
C6	0.5243 (4)	0.62872 (15)	0.6015 (3)	0.0377 (10)	
C7	0.3580 (5)	0.59411 (18)	0.3598 (3)	0.0487 (12)	
C8	0.3598 (4)	0.61986 (16)	0.2707 (3)	0.0403 (11)	
C9	0.3016 (5)	0.59894 (18)	0.1842 (3)	0.0491 (12)	
H9	0.2568	0.5691	0.1800	0.059*	
C10	0.3088 (4)	0.62196 (17)	0.1025 (3)	0.0460 (11)	
C11	0.3735 (5)	0.66710 (18)	0.1158 (3)	0.0504 (12)	
H11	0.3791	0.6847	0.0638	0.060*	
C12	0.4295 (4)	0.68648 (18)	0.2039 (3)	0.0456 (11)	

H12	0.4733	0.7167	0.2099	0.055*	
C13	0.2490 (5)	0.59941 (19)	0.0053 (3)	0.0570 (13)	
C14	0.1764 (6)	0.5507 (2)	0.0082 (4)	0.083 (2)	
H14A	0.1423	0.5374	−0.0542	0.124*	
H14B	0.1090	0.5577	0.0332	0.124*	
H14C	0.2326	0.5265	0.0474	0.124*	
C15	0.1525 (6)	0.6372 (2)	−0.0550 (4)	0.0752 (17)	
H15A	0.1097	0.6225	−0.1150	0.113*	
H15B	0.1955	0.6671	−0.0638	0.113*	
H15C	0.0922	0.6455	−0.0238	0.113*	
C16	0.3521 (6)	0.5890 (3)	−0.0374 (5)	0.110 (3)	
H16A	0.4069	0.5633	−0.0019	0.166*	
H16B	0.3997	0.6192	−0.0364	0.166*	
H16C	0.3153	0.5780	−0.1011	0.166*	
C17	0.6155 (4)	0.71209 (16)	0.6296 (3)	0.0424 (11)	
C18	0.6447 (4)	0.75677 (15)	0.5787 (3)	0.0384 (10)	
C19	0.7036 (4)	0.79860 (16)	0.6265 (3)	0.0416 (11)	
H19	0.7253	0.7996	0.6917	0.050*	
C20	0.7311 (4)	0.83949 (16)	0.5783 (3)	0.0405 (10)	
C21	0.6945 (5)	0.83492 (18)	0.4817 (3)	0.0506 (12)	
H21	0.7103	0.8612	0.4460	0.061*	
C22	0.6354 (5)	0.79231 (17)	0.4377 (3)	0.0500 (12)	
H22	0.6121	0.7904	0.3725	0.060*	
C23	0.7961 (5)	0.88641 (18)	0.6282 (3)	0.0519 (13)	
C24A	0.9179 (14)	0.8686 (6)	0.7044 (11)	0.0870 (19)	0.39
H24A	0.9733	0.8532	0.6748	0.131*	0.39
H24B	0.8966	0.8446	0.7448	0.131*	0.39
H24C	0.9593	0.8969	0.7409	0.131*	0.39
C25A	0.7102 (15)	0.9105 (6)	0.6776 (12)	0.0870 (19)	0.39
H25A	0.7130	0.8912	0.7322	0.131*	0.39
H25B	0.6253	0.9114	0.6355	0.131*	0.39
H25C	0.7383	0.9442	0.6960	0.131*	0.39
C26A	0.8306 (18)	0.9228 (6)	0.5640 (11)	0.0870 (19)	0.39
H26A	0.8734	0.9052	0.5271	0.131*	0.39
H26B	0.8849	0.9483	0.6007	0.131*	0.39
H26C	0.7554	0.9381	0.5232	0.131*	0.39
C24B	0.8573 (12)	0.8799 (4)	0.7337 (6)	0.0870 (19)	0.61
H24D	0.9155	0.9069	0.7576	0.131*	0.61
H24E	0.9016	0.8484	0.7459	0.131*	0.61
H24F	0.7934	0.8802	0.7640	0.131*	0.61
C25B	0.6950 (10)	0.9287 (4)	0.6160 (8)	0.0870 (19)	0.61
H25D	0.7333	0.9576	0.6524	0.131*	0.61
H25E	0.6277	0.9167	0.6372	0.131*	0.61
H25F	0.6621	0.9379	0.5509	0.131*	0.61
C26B	0.8916 (10)	0.9059 (4)	0.5842 (8)	0.0870 (19)	0.61
H26D	0.8513	0.9111	0.5182	0.131*	0.61
H26E	0.9584	0.8818	0.5933	0.131*	0.61
H26F	0.9258	0.9371	0.6133	0.131*	0.61
N5	1.0897 (4)	0.69071 (17)	0.5774 (3)	0.0616 (12)	

C27	1.0491 (7)	0.6444 (3)	0.5164 (6)	0.098 (2)
H27A	1.0520	0.6156	0.5568	0.117*
H27B	0.9622	0.6488	0.4773	0.117*
C28	1.1258 (8)	0.6334 (4)	0.4559 (6)	0.139 (3)
H28A	1.2126	0.6301	0.4937	0.208*
H28B	1.1178	0.6604	0.4118	0.208*
H28C	1.0976	0.6026	0.4225	0.208*
C29	1.2224 (6)	0.6877 (3)	0.6422 (5)	0.093 (2)
H29A	1.2785	0.6860	0.6044	0.112*
H29B	1.2413	0.7187	0.6782	0.112*
C30	1.2517 (8)	0.6448 (3)	0.7091 (6)	0.125 (3)
H30A	1.2313	0.6139	0.6748	0.188*
H30B	1.2029	0.6477	0.7513	0.188*
H30C	1.3399	0.6452	0.7444	0.188*
C31	0.9954 (6)	0.6931 (3)	0.6309 (6)	0.108 (3)
H31A	0.9115	0.6951	0.5857	0.129*
H31B	1.0004	0.6618	0.6652	0.129*
C32	1.0105 (10)	0.7346 (5)	0.6971 (8)	0.196 (6)
H32A	0.9316	0.7410	0.7079	0.294*
H32B	1.0367	0.7642	0.6716	0.294*
H32C	1.0731	0.7260	0.7554	0.294*
C33	1.0820 (6)	0.7370 (3)	0.5178 (5)	0.094 (2)
H33A	1.1056	0.7658	0.5592	0.112*
H33B	1.1438	0.7339	0.4851	0.112*
C34	0.9560 (7)	0.7475 (3)	0.4466 (6)	0.118 (3)
H34A	0.8935	0.7511	0.4778	0.177*
H34B	0.9331	0.7202	0.4027	0.177*
H34C	0.9606	0.7780	0.4136	0.177*
Cl11	0.19736 (18)	0.43278 (9)	0.41748 (17)	0.1219 (8)
C35A	0.1169 (9)	0.4704 (3)	0.3228 (7)	0.162 (5)
H35A	0.1603	0.5028	0.3277	0.194*
Cl2A	−0.0355 (3)	0.48061 (12)	0.3158 (4)	0.248 (2)
Cl3A	0.1157 (4)	0.4405 (2)	0.2175 (3)	0.271 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0450 (4)	0.0311 (3)	0.0310 (3)	−0.0062 (3)	0.0126 (3)	−0.0013 (3)
Cl1	0.0479 (7)	0.0577 (8)	0.0614 (8)	−0.0011 (6)	0.0194 (6)	−0.0077 (6)
Cl2	0.0567 (8)	0.0543 (8)	0.0667 (9)	0.0088 (6)	0.0197 (7)	−0.0022 (6)
Cl3	0.1042 (12)	0.0443 (8)	0.0731 (10)	−0.0178 (7)	0.0286 (9)	0.0186 (7)
Cl4	0.1320 (14)	0.0584 (9)	0.0447 (8)	−0.0082 (8)	0.0248 (9)	0.0152 (6)
O1	0.109 (3)	0.065 (2)	0.050 (2)	−0.056 (2)	0.016 (2)	−0.0018 (18)
O2	0.128 (3)	0.048 (2)	0.0294 (18)	−0.022 (2)	0.021 (2)	−0.0039 (15)
N1	0.048 (2)	0.038 (2)	0.033 (2)	−0.0066 (17)	0.0103 (17)	0.0019 (16)
N2	0.043 (2)	0.039 (2)	0.034 (2)	−0.0063 (17)	0.0134 (17)	−0.0026 (16)
N3	0.052 (2)	0.030 (2)	0.038 (2)	−0.0045 (16)	0.0169 (18)	−0.0001 (15)
N4	0.052 (2)	0.035 (2)	0.0294 (19)	−0.0059 (16)	0.0126 (17)	0.0005 (15)

C1	0.047 (3)	0.035 (2)	0.037 (2)	−0.001 (2)	0.016 (2)	0.002 (2)
C2	0.064 (3)	0.039 (3)	0.044 (3)	−0.011 (2)	0.019 (2)	0.002 (2)
C3	0.060 (3)	0.033 (3)	0.052 (3)	−0.005 (2)	0.024 (3)	0.006 (2)
C4	0.071 (3)	0.038 (3)	0.042 (3)	0.009 (2)	0.023 (3)	0.010 (2)
C5	0.065 (3)	0.034 (3)	0.038 (3)	−0.003 (2)	0.017 (2)	0.001 (2)
C6	0.048 (3)	0.029 (2)	0.040 (2)	0.0003 (19)	0.019 (2)	0.0040 (19)
C7	0.057 (3)	0.045 (3)	0.044 (3)	−0.020 (2)	0.015 (2)	−0.005 (2)
C8	0.046 (3)	0.036 (3)	0.037 (2)	−0.008 (2)	0.010 (2)	0.0014 (19)
C9	0.060 (3)	0.042 (3)	0.042 (3)	−0.015 (2)	0.012 (2)	−0.006 (2)
C10	0.053 (3)	0.044 (3)	0.038 (3)	0.001 (2)	0.010 (2)	−0.003 (2)
C11	0.065 (3)	0.048 (3)	0.036 (3)	−0.006 (2)	0.014 (2)	0.003 (2)
C12	0.052 (3)	0.045 (3)	0.038 (3)	−0.013 (2)	0.012 (2)	0.002 (2)
C13	0.070 (4)	0.053 (3)	0.039 (3)	0.004 (3)	0.005 (3)	−0.008 (2)
C14	0.120 (5)	0.049 (4)	0.055 (4)	−0.005 (3)	−0.007 (3)	−0.006 (3)
C15	0.091 (4)	0.067 (4)	0.051 (3)	−0.007 (3)	−0.002 (3)	−0.001 (3)
C16	0.094 (5)	0.171 (8)	0.068 (4)	0.013 (5)	0.029 (4)	−0.047 (5)
C17	0.058 (3)	0.035 (3)	0.035 (3)	−0.004 (2)	0.015 (2)	−0.004 (2)
C18	0.048 (3)	0.033 (2)	0.033 (2)	−0.0030 (19)	0.012 (2)	0.0000 (19)
C19	0.054 (3)	0.038 (3)	0.033 (2)	−0.002 (2)	0.014 (2)	−0.0028 (19)
C20	0.049 (3)	0.030 (2)	0.043 (3)	−0.0030 (19)	0.014 (2)	−0.002 (2)
C21	0.072 (3)	0.038 (3)	0.042 (3)	−0.017 (2)	0.018 (2)	0.001 (2)
C22	0.074 (3)	0.043 (3)	0.032 (2)	−0.015 (2)	0.015 (2)	0.001 (2)
C23	0.066 (3)	0.040 (3)	0.050 (3)	−0.017 (2)	0.019 (3)	−0.004 (2)
C24A	0.117 (5)	0.056 (4)	0.085 (4)	−0.029 (3)	0.027 (4)	−0.025 (3)
C25A	0.117 (5)	0.056 (4)	0.085 (4)	−0.029 (3)	0.027 (4)	−0.025 (3)
C26A	0.117 (5)	0.056 (4)	0.085 (4)	−0.029 (3)	0.027 (4)	−0.025 (3)
C24B	0.117 (5)	0.056 (4)	0.085 (4)	−0.029 (3)	0.027 (4)	−0.025 (3)
C25B	0.117 (5)	0.056 (4)	0.085 (4)	−0.029 (3)	0.027 (4)	−0.025 (3)
C26B	0.117 (5)	0.056 (4)	0.085 (4)	−0.029 (3)	0.027 (4)	−0.025 (3)
N5	0.052 (3)	0.065 (3)	0.068 (3)	−0.004 (2)	0.020 (2)	0.002 (2)
C27	0.077 (5)	0.086 (5)	0.122 (6)	−0.009 (4)	0.020 (5)	0.009 (5)
C28	0.107 (6)	0.184 (9)	0.121 (7)	0.043 (6)	0.030 (6)	−0.041 (6)
C29	0.067 (4)	0.111 (6)	0.099 (5)	0.012 (4)	0.023 (4)	0.001 (4)
C30	0.111 (6)	0.156 (8)	0.107 (6)	0.049 (6)	0.030 (5)	0.034 (6)
C31	0.073 (5)	0.147 (8)	0.108 (6)	0.016 (5)	0.035 (5)	0.013 (5)
C32	0.133 (9)	0.295 (16)	0.146 (9)	0.091 (9)	0.024 (7)	−0.072 (10)
C33	0.081 (5)	0.080 (5)	0.117 (6)	−0.013 (4)	0.026 (4)	0.016 (4)
C34	0.089 (5)	0.107 (6)	0.135 (7)	−0.014 (4)	0.001 (5)	0.051 (5)
Cl11	0.0844 (13)	0.1178 (17)	0.1413 (19)	−0.0121 (11)	0.0038 (12)	0.0392 (14)
C35A	0.135 (8)	0.128 (7)	0.158 (9)	−0.092 (6)	−0.047 (6)	0.059 (6)
Cl2A	0.136 (2)	0.145 (3)	0.414 (6)	0.027 (2)	0.017 (3)	0.137 (3)
Cl3A	0.187 (4)	0.410 (7)	0.150 (3)	−0.071 (4)	−0.040 (3)	−0.039 (4)

Geometric parameters (Å, °)

Fe1—N1	2.038 (3)	C23—C26A	1.498 (13)
Fe1—N3	2.060 (3)	C23—C26B	1.512 (10)
Fe1—N4	2.172 (3)	C23—C24B	1.522 (10)
Fe1—N2	2.176 (3)	C23—C25A	1.526 (13)

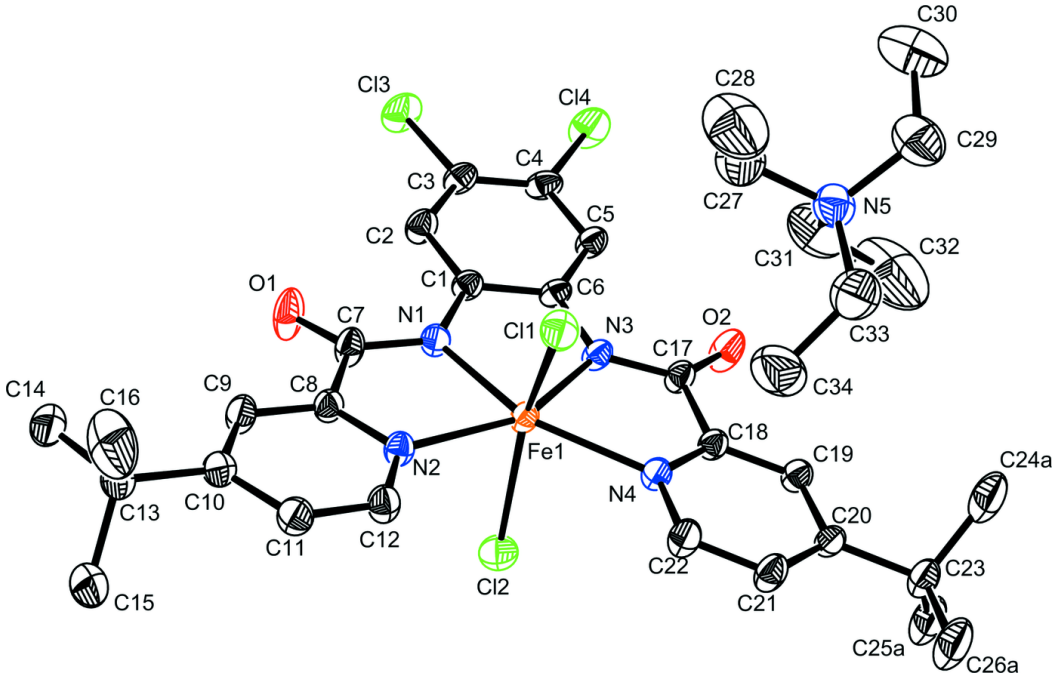
Fe1—Cl1	2.3294 (14)	C23—C24A	1.550 (13)
Fe1—Cl2	2.3879 (14)	C23—C25B	1.564 (10)
Cl3—C3	1.735 (5)	C24A—H24A	0.9600
Cl4—C4	1.724 (5)	C24A—H24B	0.9600
O1—C7	1.238 (5)	C24A—H24C	0.9600
O2—C17	1.231 (5)	C25A—H25A	0.9600
N1—C7	1.342 (5)	C25A—H25B	0.9600
N1—C1	1.407 (5)	C25A—H25C	0.9600
N2—C8	1.337 (5)	C26A—H26A	0.9600
N2—C12	1.342 (5)	C26A—H26B	0.9600
N3—C17	1.347 (5)	C26A—H26C	0.9600
N3—C6	1.401 (5)	C24B—H24D	0.9600
N4—C18	1.331 (5)	C24B—H24E	0.9600
N4—C22	1.336 (5)	C24B—H24F	0.9600
C1—C2	1.390 (6)	C25B—H25D	0.9600
C1—C6	1.415 (6)	C25B—H25E	0.9600
C2—C3	1.373 (6)	C25B—H25F	0.9600
C2—H2	0.9300	C26B—H26D	0.9600
C3—C4	1.387 (7)	C26B—H26E	0.9600
C4—C5	1.401 (6)	C26B—H26F	0.9600
C5—C6	1.384 (6)	N5—C29	1.497 (7)
C5—H5	0.9300	N5—C33	1.508 (7)
C7—C8	1.507 (6)	N5—C31	1.513 (8)
C8—C9	1.374 (6)	N5—C27	1.516 (8)
C9—C10	1.394 (6)	C27—C28	1.461 (10)
C9—H9	0.9300	C27—H27A	0.9700
C10—C11	1.383 (6)	C27—H27B	0.9700
C10—C13	1.523 (6)	C28—H28A	0.9600
C11—C12	1.372 (6)	C28—H28B	0.9600
C11—H11	0.9300	C28—H28C	0.9600
C12—H12	0.9300	C29—C30	1.485 (9)
C13—C16	1.510 (8)	C29—H29A	0.9700
C13—C14	1.537 (7)	C29—H29B	0.9700
C13—C15	1.542 (7)	C30—H30A	0.9600
C14—H14A	0.9600	C30—H30B	0.9600
C14—H14B	0.9600	C30—H30C	0.9600
C14—H14C	0.9600	C31—C32	1.459 (11)
C15—H15A	0.9600	C31—H31A	0.9700
C15—H15B	0.9600	C31—H31B	0.9700
C15—H15C	0.9600	C32—H32A	0.9600
C16—H16A	0.9600	C32—H32B	0.9600
C16—H16B	0.9600	C32—H32C	0.9600
C16—H16C	0.9600	C33—C34	1.502 (9)
C17—C18	1.503 (6)	C33—H33A	0.9700
C18—C19	1.374 (6)	C33—H33B	0.9700
C19—C20	1.393 (6)	C34—H34A	0.9600
C19—H19	0.9300	C34—H34B	0.9600
C20—C21	1.381 (6)	C34—H34C	0.9600
C20—C23	1.517 (6)	Cl11—C35A	1.738 (8)

C21—C22	1.372 (6)	C35A—Cl2A	1.694 (11)
C21—H21	0.9300	C35A—Cl3A	1.764 (12)
C22—H22	0.9300	C35A—H35A	0.9800
N1—Fe1—N3	77.44 (13)	C26B—C23—C20	111.1 (5)
N1—Fe1—N4	153.65 (13)	C26A—C23—C24B	128.2 (9)
N3—Fe1—N4	76.30 (13)	C26B—C23—C24B	110.2 (7)
N1—Fe1—N2	76.90 (13)	C20—C23—C24B	114.0 (5)
N3—Fe1—N2	154.33 (14)	C26A—C23—C25A	111.6 (10)
N4—Fe1—N2	129.33 (13)	C26B—C23—C25A	134.9 (8)
N1—Fe1—Cl1	101.69 (11)	C20—C23—C25A	107.9 (7)
N3—Fe1—Cl1	100.91 (11)	C24B—C23—C25A	71.9 (9)
N4—Fe1—Cl1	85.46 (10)	C26A—C23—C24A	109.1 (10)
N2—Fe1—Cl1	84.56 (10)	C26B—C23—C24A	81.4 (9)
N1—Fe1—Cl2	100.86 (11)	C20—C23—C24A	106.5 (7)
N3—Fe1—Cl2	101.07 (11)	C24B—C23—C24A	36.6 (7)
N4—Fe1—Cl2	82.12 (10)	C25A—C23—C24A	108.1 (10)
N2—Fe1—Cl2	83.56 (10)	C26A—C23—C25B	77.4 (9)
Cl1—Fe1—Cl2	151.34 (5)	C26B—C23—C25B	106.6 (7)
C7—N1—C1	122.9 (4)	C20—C23—C25B	108.6 (5)
C7—N1—Fe1	119.8 (3)	C24B—C23—C25B	105.9 (7)
C1—N1—Fe1	117.3 (3)	C25A—C23—C25B	38.1 (7)
C8—N2—C12	117.5 (4)	C24A—C23—C25B	137.7 (8)
C8—N2—Fe1	113.8 (3)	C23—C24A—H24A	109.5
C12—N2—Fe1	128.6 (3)	C23—C24A—H24B	109.5
C17—N3—C6	123.5 (4)	C23—C24A—H24C	109.5
C17—N3—Fe1	119.9 (3)	C23—C25A—H25A	109.5
C6—N3—Fe1	116.6 (3)	C23—C25A—H25B	109.5
C18—N4—C22	118.2 (4)	C23—C25A—H25C	109.5
C18—N4—Fe1	114.6 (3)	C23—C26A—H26A	109.5
C22—N4—Fe1	127.2 (3)	C23—C26A—H26B	109.5
C2—C1—N1	126.3 (4)	C23—C26A—H26C	109.5
C2—C1—C6	119.7 (4)	C23—C24B—H24D	109.5
N1—C1—C6	114.0 (4)	C23—C24B—H24E	109.5
C3—C2—C1	120.4 (4)	H24D—C24B—H24E	109.5
C3—C2—H2	119.8	C23—C24B—H24F	109.5
C1—C2—H2	119.8	H24D—C24B—H24F	109.5
C2—C3—C4	120.7 (4)	H24E—C24B—H24F	109.5
C2—C3—Cl3	118.6 (4)	C23—C25B—H25D	109.5
C4—C3—Cl3	120.7 (4)	C23—C25B—H25E	109.5
C3—C4—C5	119.6 (4)	H25D—C25B—H25E	109.5
C3—C4—Cl4	121.3 (4)	C23—C25B—H25F	109.5
C5—C4—Cl4	119.1 (4)	H25D—C25B—H25F	109.5
C6—C5—C4	120.4 (4)	H25E—C25B—H25F	109.5
C6—C5—H5	119.8	C23—C26B—H26D	109.5
C4—C5—H5	119.8	C23—C26B—H26E	109.5
C5—C6—N3	126.4 (4)	H26D—C26B—H26E	109.5
C5—C6—C1	119.3 (4)	C23—C26B—H26F	109.5
N3—C6—C1	114.3 (4)	H26D—C26B—H26F	109.5
O1—C7—N1	128.5 (4)	H26E—C26B—H26F	109.5

O1—C7—C8	118.4 (4)	C29—N5—C33	106.8 (5)
N1—C7—C8	113.1 (4)	C29—N5—C31	111.8 (5)
N2—C8—C9	122.7 (4)	C33—N5—C31	111.1 (5)
N2—C8—C7	116.0 (4)	C29—N5—C27	113.3 (5)
C9—C8—C7	121.3 (4)	C33—N5—C27	110.8 (5)
C8—C9—C10	120.8 (4)	C31—N5—C27	103.2 (5)
C8—C9—H9	119.6	C28—C27—N5	114.5 (6)
C10—C9—H9	119.6	C28—C27—H27A	108.6
C11—C10—C9	115.3 (4)	N5—C27—H27A	108.6
C11—C10—C13	122.1 (4)	C28—C27—H27B	108.6
C9—C10—C13	122.6 (4)	N5—C27—H27B	108.6
C12—C11—C10	121.7 (4)	H27A—C27—H27B	107.6
C12—C11—H11	119.2	C27—C28—H28A	109.5
C10—C11—H11	119.2	C27—C28—H28B	109.5
N2—C12—C11	122.0 (4)	H28A—C28—H28B	109.5
N2—C12—H12	119.0	C27—C28—H28C	109.5
C11—C12—H12	119.0	H28A—C28—H28C	109.5
C16—C13—C10	108.4 (5)	H28B—C28—H28C	109.5
C16—C13—C14	109.7 (5)	C30—C29—N5	116.2 (6)
C10—C13—C14	112.4 (4)	C30—C29—H29A	108.2
C16—C13—C15	110.9 (5)	N5—C29—H29A	108.2
C10—C13—C15	108.6 (4)	C30—C29—H29B	108.2
C14—C13—C15	106.8 (5)	N5—C29—H29B	108.2
C13—C14—H14A	109.5	H29A—C29—H29B	107.4
C13—C14—H14B	109.5	C29—C30—H30A	109.5
H14A—C14—H14B	109.5	C29—C30—H30B	109.5
C13—C14—H14C	109.5	H30A—C30—H30B	109.5
H14A—C14—H14C	109.5	C29—C30—H30C	109.5
H14B—C14—H14C	109.5	H30A—C30—H30C	109.5
C13—C15—H15A	109.5	H30B—C30—H30C	109.5
C13—C15—H15B	109.5	C32—C31—N5	116.3 (8)
H15A—C15—H15B	109.5	C32—C31—H31A	108.2
C13—C15—H15C	109.5	N5—C31—H31A	108.2
H15A—C15—H15C	109.5	C32—C31—H31B	108.2
H15B—C15—H15C	109.5	N5—C31—H31B	108.2
C13—C16—H16A	109.5	H31A—C31—H31B	107.4
C13—C16—H16B	109.5	C31—C32—H32A	109.5
H16A—C16—H16B	109.5	C31—C32—H32B	109.5
C13—C16—H16C	109.5	H32A—C32—H32B	109.5
H16A—C16—H16C	109.5	C31—C32—H32C	109.5
H16B—C16—H16C	109.5	H32A—C32—H32C	109.5
O2—C17—N3	127.9 (4)	H32B—C32—H32C	109.5
O2—C17—C18	119.3 (4)	C34—C33—N5	116.1 (5)
N3—C17—C18	112.7 (4)	C34—C33—H33A	108.3
N4—C18—C19	122.3 (4)	N5—C33—H33A	108.3
N4—C18—C17	116.4 (4)	C34—C33—H33B	108.3
C19—C18—C17	121.3 (4)	N5—C33—H33B	108.3
C18—C19—C20	120.6 (4)	H33A—C33—H33B	107.4
C18—C19—H19	119.7	C33—C34—H34A	109.5

C20—C19—H19	119.7	C33—C34—H34B	109.5
C21—C20—C19	115.8 (4)	H34A—C34—H34B	109.5
C21—C20—C23	121.8 (4)	C33—C34—H34C	109.5
C19—C20—C23	122.4 (4)	H34A—C34—H34C	109.5
C22—C21—C20	121.1 (4)	H34B—C34—H34C	109.5
C22—C21—H21	119.5	Cl2A—C35A—Cl11	113.2 (6)
C20—C21—H21	119.5	Cl2A—C35A—Cl3A	107.0 (5)
N4—C22—C21	122.1 (4)	Cl11—C35A—Cl3A	109.2 (7)
N4—C22—H22	119.0	Cl2A—C35A—H35A	109.1
C21—C22—H22	119.0	Cl11—C35A—H35A	109.1
C26A—C23—C26B	30.5 (8)	Cl3A—C35A—H35A	109.1
C26A—C23—C20	113.2 (8)		

Fig. 1


C30
C28
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Cl4
C29
C4
C3
C27
N5
C5
C2
C31
C32
O1
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C7
N1
Cl1
N3
O2
C33
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C14
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C24a
N2
N4
C13
C10
C20
C11
C12
C22
C23
Cl2
C21
C15
C25a
C26a