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Dichlorido[1-(8-quinolyliminomethyl)-2-naphtholato]iron(III)

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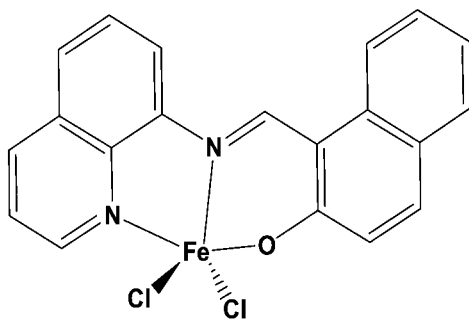
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.037; wR factor = 0.090; data-to-parameter ratio = 16.7.

The Fe^{III} ion in the title complex, $[\text{FeCl}_2(\text{C}_{20}\text{H}_{13}\text{N}_2\text{O})]$, has a distorted square-pyramidal coordination formed by one O atom and two N atoms from a tridentate 1-(8-quinolyliminomethyl)-2-naphtholate ligand and two Cl atoms. In the crystal structure, molecules form a column structure along the a axis through π - π stacking interactions, with centroid-centroid distances of 3.657 (1) and 3.818 (2) Å. Weak $\text{C}-\text{H}\cdots\text{Cl}$ interactions are observed between the columns.

Related literature

For supramolecular self-assembly, see: Crivillers & Furukawa (2009).



Experimental

Crystal data

 $[\text{FeCl}_2(\text{C}_{20}\text{H}_{13}\text{N}_2\text{O})]$ $M_r = 424.07$

Monoclinic, $P2_1/n$
 $a = 7.6177$ (5) Å
 $b = 18.5256$ (11) Å
 $c = 12.2073$ (7) Å
 $\beta = 91.1612$ (16)°
 $V = 1722.37$ (18) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.20$ mm⁻¹
 $T = 293$ K
 $0.80 \times 0.20 \times 0.10$ mm

Data collection

Rigaku R-Axis RAPID
 diffractometer
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 2001)
 $T_{\text{min}} = 0.448$, $T_{\text{max}} = 0.890$

17621 measured reflections
 3934 independent reflections
 3182 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.090$
 $S = 1.08$
 3934 reflections

235 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.44$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}14-\text{H}12\cdots\text{Cl}^{\text{ii}}$	0.93	2.81	3.598 (2)	143
$\text{C}19-\text{H}8\cdots\text{Cl}^{\text{ii}}$	0.93	2.86	3.656 (2)	144

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalClear* (Molecular Structure Corporation and Rigaku, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Yadokari-XG* (Wakita, 2000); software used to prepare material for publication: *SHELXL97*.

This work was supported by 'Development of Molecular Devices in Ferroelectric Metallomesogens' in 2006 of the New Energy and Industrial Technology Development Organization (NEDO) of Japan and Grants-in-Aid for Scientific Research from the Ministry of Education, Culture, Sports, Science and Technology of the Japanese Government (No. 20350028).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2429).

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supplementary materials

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Dichlorido[1-(8-quinolyliminomethyl)-2-naphtholato]iron(III)

D. Urakami, K. Inoue and S. Hayami

Comment

Self-assembly has been recognized as a most efficient process that organizes individual molecular components into highly ordered supramolecular species (Crivillers *et al.*, 2009). The designed construction of supramolecules from molecular building blocks is noted as one of most challenging issues facing synthetic chemistry today. The method by using self-assembly is very important in developing novel molecular compounds with multi-functions. The cooperativity can be achieved by using π - π interactions as well as by using bridging ligands. We focused on an iron(III) complex with a qnal ligand [qnal = 1-(quinolin-8-yliminomethyl)-naphthalen-2-ol] having large π electron system. Here we report the synthesis and crystal structure of the title complex.

The Fe^{III} ion in the title complex, [Fe(qnal)Cl₂], has a distorted five coordination environment formed by one O atom and two N atoms from a qnal ligand, and two Cl atoms. The Fe—O bond length is shortest and the Fe—Cl bond length is longest. The π - π contacts between the benzene and pyridine rings, Cg1 \cdots Cg3ⁱ and Cg2 \cdots Cg3ⁱⁱ [symmetry codes: (i) $-x, -y, 1 - z$; (ii) $1 - x, -y, 1 - z$, where Cg1, Cg2, Cg3 are centroids of the rings (N1/C1–C4/C9), (C4–C9) and (C11–/C15/C20), respectively] may stabilize the structure, with centroid-centroid distances of 3.657 (1) and 3.818 (2) Å, respectively. The molecules form a column structure by π - π stacking along the *a* axis. Three dimensional network is formed through C—H \cdots Cl interactions between columns.

Experimental

The ligand molecule, qnal, was prepared from 8-aminoquinoline (4.2 mg, 0.03 mmol) and 2-hydroxy-1-naphthaldehyde (5.1 mg, 0.03 mmol), which were mixed in 10 ml methanol and heating on a oil bath for about 2 h under reflux. The title complex was prepared by slow diffusion of qnal (9.0 mg, 0.03 mmol) and FeCl₃ (4.9 mg, 0.03 mmol) in methanol by using a H-form tube. After about one week, single crystals were obtained as black needles.

Refinement

All H atoms were positioned geometrically (C—H = 0.93 Å) and were refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

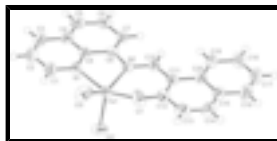


Fig. 1. ORTEP drawing of the title complex, showing 50% probability displacement ellipsoids.

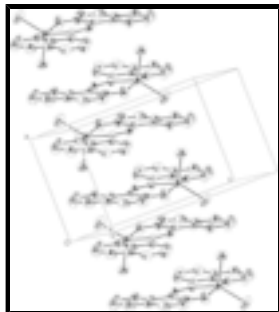


Fig. 2. Column structure for the title complex.



Fig. 3. Part of the crystal structure, showing C—H...Cl interactions as dashed lines.

Dichlorido[1-(8-quinolyliminomethyl)-2-naphtholato]iron(III)

Crystal data

[FeCl₂(C₂₀H₁₃N₂O)]

$M_r = 424.07$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 7.6177$ (5) Å

$b = 18.5256$ (11) Å

$c = 12.2073$ (7) Å

$\beta = 91.1612$ (16)°

$V = 1722.37$ (18) Å³

$Z = 4$

$F_{000} = 860$

$D_x = 1.635$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å

Cell parameters from 15565 reflections

$\theta = 3.1$ – 27.7°

$\mu = 1.20$ mm⁻¹

$T = 293$ K

Needle, black

$0.80 \times 0.20 \times 0.10$ mm

Data collection

Rigaku R-Axis RAPID
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ K

ω scans

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 2001)

$T_{\min} = 0.448$, $T_{\max} = 0.890$

17621 measured reflections

3934 independent reflections

3182 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 27.5^\circ$

$\theta_{\min} = 3.1^\circ$

$h = -9 \rightarrow 9$

$k = -24 \rightarrow 23$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$$R[F^2 > 2\sigma(F^2)] = 0.037$$

$$wR(F^2) = 0.090$$

$$S = 1.08$$

3934 reflections

235 parameters

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.7323P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.44 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.16122 (4)	0.072865 (16)	0.67792 (2)	0.03433 (11)
C1	0.1279 (3)	-0.04180 (15)	0.8607 (2)	0.0498 (6)
H1	0.0834	-0.0042	0.9022	0.060*
C2	0.1519 (4)	-0.10907 (17)	0.9103 (2)	0.0599 (7)
H2	0.1216	-0.1161	0.9830	0.072*
C3	0.2195 (4)	-0.16364 (16)	0.8515 (2)	0.0562 (7)
H3	0.2361	-0.2086	0.8839	0.067*
C4	0.2653 (3)	-0.15321 (13)	0.74142 (19)	0.0409 (5)
C5	0.3412 (3)	-0.20622 (12)	0.6755 (2)	0.0485 (6)
H4	0.3632	-0.2521	0.7035	0.058*
C6	0.3825 (3)	-0.19030 (12)	0.5702 (2)	0.0451 (6)
H5	0.4356	-0.2254	0.5276	0.054*
C7	0.3470 (3)	-0.12257 (12)	0.52462 (19)	0.0401 (5)
H6	0.3743	-0.1136	0.4520	0.048*
C8	0.2723 (3)	-0.06922 (10)	0.58598 (17)	0.0306 (4)
C9	0.2340 (3)	-0.08413 (11)	0.69642 (17)	0.0329 (4)
C10	0.2480 (3)	0.01983 (11)	0.44882 (17)	0.0321 (4)
H7	0.2835	-0.0167	0.4020	0.038*
C11	0.2188 (3)	0.08832 (11)	0.40091 (17)	0.0327 (4)
C12	0.1502 (3)	0.14570 (12)	0.46229 (19)	0.0399 (5)
C13	0.1253 (3)	0.21425 (13)	0.4125 (2)	0.0495 (6)
H13	0.0767	0.2516	0.4526	0.059*
C14	0.1709 (3)	0.22598 (14)	0.3084 (2)	0.0524 (7)
H12	0.1570	0.2719	0.2789	0.063*

supplementary materials

C15	0.2392 (3)	0.17054 (13)	0.24256 (19)	0.0441 (6)
C16	0.2835 (4)	0.18369 (17)	0.1329 (2)	0.0600 (8)
H11	0.2733	0.2303	0.1052	0.072*
C17	0.3403 (4)	0.13057 (19)	0.0673 (2)	0.0655 (8)
H10	0.3656	0.1400	-0.0055	0.079*
C18	0.3606 (4)	0.06130 (17)	0.1099 (2)	0.0586 (7)
H9	0.3989	0.0244	0.0647	0.070*
C19	0.3254 (3)	0.04645 (14)	0.21726 (19)	0.0450 (6)
H8	0.3441	0.0001	0.2444	0.054*
C20	0.2612 (3)	0.10040 (12)	0.28673 (18)	0.0354 (5)
N1	0.1657 (2)	-0.02918 (10)	0.75694 (14)	0.0366 (4)
N2	0.2306 (2)	0.00218 (9)	0.55195 (14)	0.0305 (4)
O1	0.1072 (3)	0.13872 (9)	0.56416 (14)	0.0559 (5)
Cl1	0.40989 (8)	0.11675 (4)	0.74667 (6)	0.05373 (18)
Cl2	-0.06535 (9)	0.11031 (4)	0.77771 (7)	0.0629 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.03643 (18)	0.03247 (18)	0.03430 (17)	0.00072 (13)	0.00555 (13)	-0.00741 (13)
C1	0.0496 (15)	0.0636 (17)	0.0365 (12)	-0.0030 (13)	0.0074 (11)	0.0006 (12)
C2	0.0656 (18)	0.076 (2)	0.0387 (14)	-0.0091 (16)	0.0045 (13)	0.0150 (14)
C3	0.0592 (17)	0.0552 (16)	0.0539 (15)	-0.0136 (13)	-0.0071 (13)	0.0222 (13)
C4	0.0372 (12)	0.0376 (12)	0.0476 (13)	-0.0079 (10)	-0.0082 (10)	0.0090 (10)
C5	0.0510 (15)	0.0290 (12)	0.0650 (16)	-0.0031 (11)	-0.0136 (13)	0.0054 (11)
C6	0.0474 (14)	0.0306 (11)	0.0569 (15)	0.0025 (10)	-0.0076 (12)	-0.0099 (10)
C7	0.0459 (13)	0.0345 (12)	0.0400 (12)	0.0007 (10)	0.0006 (10)	-0.0067 (9)
C8	0.0299 (10)	0.0264 (10)	0.0354 (11)	-0.0023 (8)	-0.0013 (8)	-0.0019 (8)
C9	0.0282 (10)	0.0336 (11)	0.0367 (11)	-0.0055 (9)	-0.0026 (8)	0.0006 (9)
C10	0.0328 (11)	0.0313 (11)	0.0322 (10)	0.0007 (9)	0.0034 (8)	-0.0047 (8)
C11	0.0314 (11)	0.0317 (11)	0.0348 (11)	-0.0002 (9)	-0.0015 (9)	-0.0010 (8)
C12	0.0397 (12)	0.0377 (12)	0.0420 (12)	0.0075 (10)	-0.0076 (10)	-0.0035 (10)
C13	0.0530 (15)	0.0365 (13)	0.0583 (16)	0.0138 (11)	-0.0138 (12)	-0.0061 (11)
C14	0.0528 (16)	0.0383 (13)	0.0654 (18)	0.0043 (11)	-0.0165 (13)	0.0139 (12)
C15	0.0390 (13)	0.0461 (14)	0.0466 (13)	-0.0034 (11)	-0.0099 (10)	0.0126 (11)
C16	0.0582 (17)	0.0689 (19)	0.0525 (16)	-0.0071 (15)	-0.0085 (14)	0.0291 (14)
C17	0.0636 (19)	0.092 (2)	0.0408 (14)	-0.0143 (17)	0.0042 (13)	0.0187 (15)
C18	0.0532 (16)	0.080 (2)	0.0431 (14)	-0.0083 (14)	0.0114 (12)	-0.0013 (14)
C19	0.0465 (14)	0.0495 (14)	0.0391 (12)	-0.0061 (11)	0.0077 (11)	0.0021 (11)
C20	0.0307 (11)	0.0381 (11)	0.0372 (11)	-0.0044 (9)	-0.0033 (9)	0.0049 (9)
N1	0.0371 (10)	0.0410 (10)	0.0317 (9)	-0.0036 (8)	0.0040 (8)	-0.0004 (8)
N2	0.0339 (9)	0.0276 (8)	0.0301 (9)	0.0004 (7)	0.0020 (7)	-0.0019 (7)
O1	0.0798 (13)	0.0474 (10)	0.0406 (9)	0.0282 (9)	0.0024 (9)	-0.0061 (8)
Cl1	0.0437 (3)	0.0501 (4)	0.0674 (4)	-0.0116 (3)	0.0018 (3)	-0.0121 (3)
Cl2	0.0552 (4)	0.0489 (4)	0.0858 (5)	0.0031 (3)	0.0354 (4)	-0.0098 (3)

Geometric parameters (\AA , $^\circ$)

Fe1—O1	1.8876 (17)	C9—N1	1.367 (3)
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Fe1—N2	2.0957 (17)	C10—C11	1.413 (3)
Fe1—N1	2.1223 (19)	C10—N2	1.310 (3)
Fe1—C11	2.2111 (7)	C10—H7	0.9300
Fe1—C12	2.2426 (7)	C11—C12	1.407 (3)
C1—C2	1.396 (4)	C11—C20	1.454 (3)
C1—N1	1.325 (3)	C12—C13	1.419 (3)
C1—H1	0.9300	C12—O1	1.299 (3)
C2—C3	1.349 (4)	C13—C14	1.342 (4)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.408 (3)	C14—C15	1.410 (4)
C3—H3	0.9300	C14—H12	0.9300
C4—C5	1.402 (3)	C15—C16	1.408 (4)
C4—C9	1.411 (3)	C15—C20	1.416 (3)
C5—C6	1.362 (4)	C16—C17	1.346 (4)
C5—H4	0.9300	C16—H11	0.9300
C6—H5	0.9300	C17—H10	0.9300
C6—C7	1.397 (3)	C18—C17	1.392 (4)
C7—C8	1.371 (3)	C18—H9	0.9300
C7—H6	0.9300	C19—C18	1.371 (3)
C8—C9	1.412 (3)	C19—H8	0.9300
C8—N2	1.420 (3)	C20—C19	1.405 (3)
Fe1—N1—C1	126.14 (17)	C11—C20—C15	118.6 (2)
Fe1—N1—C9	114.86 (14)	C11—C20—C19	123.8 (2)
Fe1—N2—C8	115.18 (13)	C12—C11—C20	119.1 (2)
Fe1—N2—C10	125.53 (14)	C12—C13—H13	119.5
Fe1—O1—C12	135.52 (15)	C13—C12—O1	117.7 (2)
C1—C2—C3	119.1 (2)	C13—C14—C15	121.8 (2)
C1—C2—H2	120.5	C13—C14—H12	119.1
C1—N1—C9	118.5 (2)	C14—C13—C12	121.1 (2)
C2—C1—N1	122.9 (3)	C14—C13—H13	119.5
C2—C1—H1	118.5	C14—C15—C20	119.6 (2)
C2—C3—C4	120.7 (2)	C15—C14—H12	119.1
C2—C3—H3	119.7	C15—C16—H11	119.2
C3—C2—H2	120.5	C15—C20—C19	117.6 (2)
C3—C4—C5	124.4 (2)	C16—C15—C14	120.9 (2)
C3—C4—C9	116.9 (2)	C16—C15—C20	119.5 (2)
C4—C3—H3	119.7	C16—C17—C18	119.1 (3)
C4—C5—C6	119.8 (2)	C16—C17—H10	120.4
C4—C5—H4	120.1	C17—C16—C15	121.7 (3)
C4—C9—C8	120.8 (2)	C17—C16—H11	119.2
C4—C9—N1	121.9 (2)	C17—C18—C19	121.3 (3)
C5—C4—C9	118.7 (2)	C17—C18—H9	119.3
C5—C6—C7	121.6 (2)	C18—C17—H10	120.4
C5—C6—H5	119.2	C18—C19—C20	120.8 (3)
C6—C5—H4	120.1	C18—C19—H8	119.6
C6—C7—C8	120.6 (2)	C19—C18—H9	119.3
C6—C7—H6	119.7	C20—C19—H8	119.6
C7—C6—H5	119.2	N1—Fe1—N2	77.00 (7)
C7—C8—C9	118.47 (19)	N1—Fe1—O1	155.70 (8)

supplementary materials

C7—C8—N2	127.17 (19)	N1—Fe1—Cl1	98.59 (5)
C8—C7—H6	119.7	N1—Fe1—Cl2	91.95 (5)
C8—C9—N1	117.29 (19)	N1—C1—H1	118.5
C8—N2—C10	119.16 (17)	N2—Fe1—O1	85.32 (7)
C9—C8—N2	114.34 (18)	N2—Fe1—Cl1	106.34 (5)
C10—C11—C12	121.0 (2)	N2—Fe1—Cl2	143.16 (5)
C10—C11—C20	119.91 (19)	N2—C10—H7	116.4
C11—C10—N2	127.14 (19)	O1—Fe1—Cl1	102.35 (7)
C11—C10—H7	116.4	O1—Fe1—Cl2	92.36 (6)
C11—C12—C13	119.7 (2)	Cl1—Fe1—Cl2	110.06 (3)
C11—C12—O1	122.6 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C14—H12 \cdots C11 ⁱ	0.93	2.81	3.598 (2)	143
C19—H8 \cdots C11 ⁱⁱ	0.93	2.86	3.656 (2)	144

Symmetry codes: (i) $x-1/2, -y+1/2, z-1/2$; (ii) $-x+1, -y, -z+1$.

Fig. 1

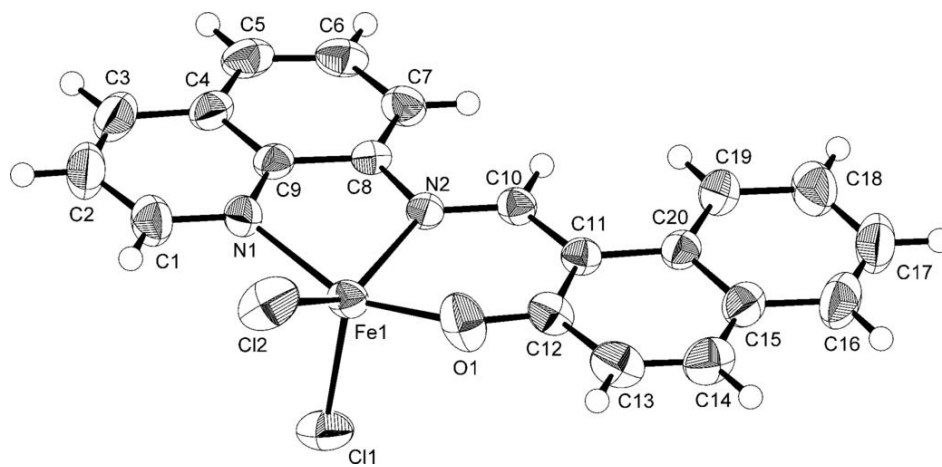


Fig. 2

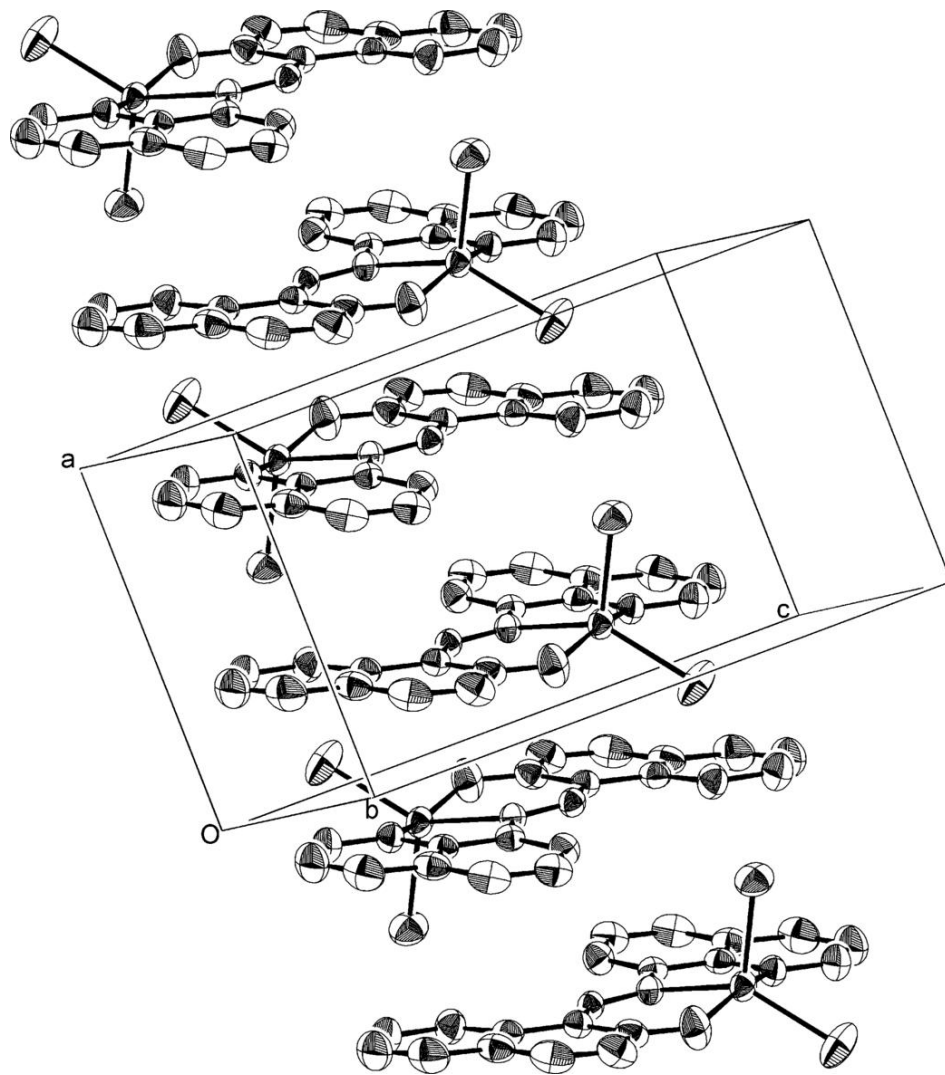


Fig. 3

