

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## Trichlorido[(methyl{2-[methyl(2-pyridyl-methyl)amino]ethyl}amino)acetonitrile]-iron(III) methanol hemisolvate

Anne Nielsen, Christine J. McKenzie and Andrew D. Bond\*

University of Southern Denmark, Department of Physics and Chemistry, Campusvej 55, 5230 Odense M, Denmark

Correspondence e-mail: adb@chem.sdu.dk

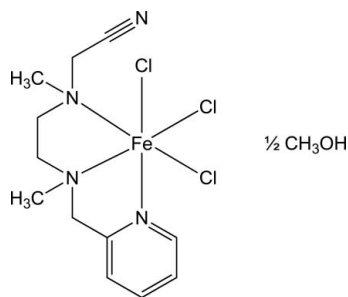
Received 12 October 2009; accepted 22 October 2009

Key indicators: single-crystal X-ray study;  $T = 180$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å; disorder in solvent or counterion;  $R$  factor = 0.040;  $wR$  factor = 0.105; data-to-parameter ratio = 15.0.

The title compound,  $[\text{FeCl}_3(\text{C}_{12}\text{H}_{18}\text{N}_4)] \cdot 0.5\text{CH}_3\text{OH}$ , contains an  $\text{Fe}^{\text{III}}$  ion in a distorted octahedral coordination environment. The neutral  $N,N',N''$ -tridentate ligand adopts a *fac* coordination mode, and chloride ligands lie *trans* to each of the three coordinated N atoms. In the crystal, the complexes form columns extending parallel to the approximate local threefold axes of the  $\text{FeN}_3\text{Cl}_3$  octahedra, and the columns are arranged so that the uncoordinated nitrile groups align in an antiparallel manner and the pyridyl rings form offset face-to-face arrangements [interplanar separations = 2.95 (1) and 3.11 (1) Å; centroid-centroid distances = 5.31 (1) and 4.92 (1) Å]. The methanol solvent molecule is disordered about a twofold rotation axis.

## Related literature

For structures of similar  $\text{Fe}^{\text{III}}$  complexes, see: Cowdell *et al.* (2004); Sundaravel *et al.* (2008); Velusamy *et al.* (2005).



## Experimental

## Crystal data

$[\text{FeCl}_3(\text{C}_{12}\text{H}_{18}\text{N}_4)] \cdot 0.5\text{CH}_3\text{O}$   
 $M_r = 396.53$   
 Monoclinic,  $C2/c$   
 $a = 34.243$  (2) Å  
 $b = 7.1331$  (5) Å  
 $c = 15.4835$  (11) Å  
 $\beta = 116.733$  (3)°

$V = 3377.8$  (4) Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 1.37$  mm<sup>-1</sup>  
 $T = 180$  K  
 $0.18 \times 0.10 \times 0.10$  mm

## Data collection

Bruker-Nonius X8 APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2004)  
 $T_{\text{min}} = 0.744$ ,  $T_{\text{max}} = 0.875$

38084 measured reflections  
 2937 independent reflections  
 2033 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.066$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.105$   
 $S = 1.07$   
 2937 reflections

196 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.48$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.45$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

Fe1—N1	2.186 (3)	Fe1—Cl1	2.2873 (11)
Fe1—N2	2.235 (3)	Fe1—Cl2	2.2908 (11)
Fe1—N3	2.330 (3)	Fe1—Cl3	2.3284 (11)

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

We are grateful to the Danish Natural Sciences Research Council and the Carlsberg Foundation for provision of the X-ray equipment.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5138).

## References

- Bruker (2004). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Cowdell, R., Davies, C. J., Hilton, S. T., Marechal, J.-D., Solan, G. A., Thomas, O. & Fawcett, J. (2004). *Dalton Trans.* pp. 3231–3240.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Sundaravel, K., Dhanalakshmi, T., Suresh, E. & Palaniandavar, M. (2008). *Dalton Trans.* pp. 7012–7025.  
 Velusamy, M., Mayilmurugan, R. & Palaniandavar, M. (2005). *J. Inorg. Biochem.* **99**, 1032–1042.

**supplementary materials**

*Acta Cryst.* (2009). E65, m1437 [ doi:10.1107/S160053680904375X ]

## Trichlorido[(methyl{2-[methyl(2-pyridylmethyl)amino]ethyl}amino)acetonitrile]iron(III) methanol hemisolvate

A. Nielsen, C. J. McKenzie and A. D. Bond

### Comment

The ligand *N,N'*-dimethyl-*N*-(2-pyridylmethyl)ethylenediamine-*N'*-acetonitrile was prepared as a by-product during synthesis of *N,N'*-dimethyl-*N*-(2-pyridylmethyl)ethylenediamine-*N'*-acetic acid, as a result of contamination of the reagent bromoacetic acid with bromoacetonitrile.

### Experimental

The ligand synthesis was undertaken in three steps:

(i) Picolinal (2.50 ml, 24 mmol) and *N,N'*-dimethylethylenediamine (2.22 ml, 24 mmol) in dry diethylether (20 ml) were stirred overnight under CaCl<sub>2</sub> protection. The solvent was removed under reduced pressure to leave 1,3-dimethyl-2-(2-pyridylmethyl)imidazolidine as a thin yellow oil (3.9 g, yield 92%).

(ii) NaBH<sub>3</sub>CN (1.3925 g, 22 mmol) and CF<sub>3</sub>COOH (3.365 ml, 44 mmol) were added in small portions [CAUTION: possible formation of HCN!] to 1,3-dimethyl-2-(2-pyridylmethyl)imidazolidine (3.8944 g, 22 mmol) in methanol (80 ml) and the reaction mixture was stirred overnight under CaCl<sub>2</sub> protection. NaOH (85 ml of a 4 M aqueous solution) was added. The reaction mixture was stirred overnight and extracted with CHCl<sub>2</sub> (3 × 20 ml), then the organic phase was dried over Na<sub>2</sub>SO<sub>4</sub> and filtered. The filtrate was evaporated *in vacuo* to leave *N,N'*-dimethyl-*N*-(2-pyridylmethyl)ethylenediamine as a thin yellow oil (3.2 g, yield 81%).

(iii) A mixture of *N,N'*-dimethyl-*N*-(2-pyridylmethyl)ethylenediamine (3.1602 g, 18 mmol), bromoacetic acid (2.4499 g, 18 mmol) and triethylamine (2.444 ml, 18 mmol) in absolute ethanol (10 ml) was heated overnight under reflux and N<sub>2</sub>. The solvent was removed under reduced pressure, then the residue was re-dissolved in water, adjusted to pH 8 with conc. NaOH and washed with CH<sub>2</sub>Cl<sub>2</sub> (3 × 15 ml). The aqueous phase was adjusted to pH 4 with conc. HCl then evaporated *in vacuo* to leave a mixture of *N,N'*-dimethyl-*N*-(2-pyridylmethyl)ethylenediamine-*N'*-acetonitrile (*L*) and *N,N'*-dimethyl-*N*-(2-pyridylmethyl)ethylenediamine-*N'*-acetic acid as a brown oil (6.0 g, yield 139% due to impurities of triethylammonium bromide).

The title compound was then prepared as follows:

Anhydrous FeCl<sub>3</sub> (15.8 mg, 0.097 mmol) was added to the mixed ligand product from above (23.3 mg, 0.098 mmol) in methanol (1.75 ml), and a few yellow blocks of (I) were deposited overnight.

## Refinement

H atoms bound to C atoms were placed in idealized positions with C—H = 0.95–0.99 Å and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2$  or  $1.5U_{\text{eq}}(\text{C})$ . The methanol molecule is disordered around a 2-fold rotation axis and all of its atoms have site occupancy factor 0.5. The H atom of the OH group was placed along the O1S—C13<sup>i</sup> vector [symmetry code (i):  $x, 1 - y, 1/2 + z$ ], with O—H = 0.85 Å and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ .

## Figures

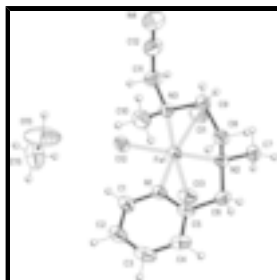


Fig. 1. The molecular structure of (I) with displacement ellipsoids shown at 50% probability for non-H atoms. The methanol molecule (C1S—O1S) is disordered around a 2-fold rotation axis.

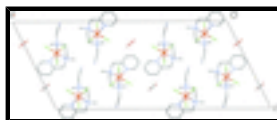


Fig. 2. Unit-cell contents of (I) projected along the *b* axis, which corresponds to the stacking directions of the "columns" referred to in the Abstract.

## Trichlorido[(methyl{2-[methyl(2- pyridylmethyl)amino]ethyl}amino)acetonitrile]iron(III) methanol hemisolvate

### Crystal data

[FeCl<sub>3</sub>(C<sub>12</sub>H<sub>18</sub>N<sub>4</sub>)]·0.5CH<sub>4</sub>O

$M_r = 396.53$

Monoclinic, *C*2/*c*

Hall symbol: -*C* 2yc

$a = 34.243$  (2) Å

$b = 7.1331$  (5) Å

$c = 15.4835$  (11) Å

$\beta = 116.733$  (3)°

$V = 3377.8$  (4) Å<sup>3</sup>

$Z = 8$

$F_{000} = 1632$

$D_x = 1.559$  Mg m<sup>-3</sup>

Mo *K*α radiation,  $\lambda = 0.71073$  Å

Cell parameters from 5350 reflections

$\theta = 2.6$ – $21.6$ °

$\mu = 1.37$  mm<sup>-1</sup>

$T = 180$  K

Block, yellow

$0.18 \times 0.10 \times 0.10$  mm

### Data collection

Bruker–Nonius X8 APEXII CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 180$  K

Thin-slice  $\omega$  and  $\phi$  scans

2937 independent reflections

2033 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.066$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 3.7$ °

Absorption correction: multi-scan  
(SADABS; Bruker, 2004)  $h = -37 \rightarrow 40$   
 $T_{\min} = 0.744$ ,  $T_{\max} = 0.875$   $k = -8 \rightarrow 8$   
 38084 measured reflections  $l = -18 \rightarrow 18$

*Refinement*

Refinement on  $F^2$  Secondary atom site location: difference Fourier map  
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites  
 $R[F^2 > 2\sigma(F^2)] = 0.040$  H-atom parameters constrained  
 $wR(F^2) = 0.105$   $w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 6.352P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $S = 1.07$   $(\Delta/\sigma)_{\max} < 0.001$   
 2937 reflections  $\Delta\rho_{\max} = 0.48 \text{ e } \text{\AA}^{-3}$   
 196 parameters  $\Delta\rho_{\min} = -0.45 \text{ e } \text{\AA}^{-3}$   
 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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Fe1	0.132445 (17)	0.50145 (7)	0.14178 (4)	0.02864 (18)	
Cl1	0.19634 (3)	0.65429 (14)	0.23597 (8)	0.0446 (3)	
Cl2	0.09339 (3)	0.60710 (15)	0.21941 (7)	0.0419 (3)	
Cl3	0.10936 (3)	0.71324 (14)	0.01386 (7)	0.0449 (3)	
N1	0.07912 (9)	0.3232 (4)	0.0431 (2)	0.0303 (7)	
N2	0.16333 (9)	0.3137 (4)	0.0744 (2)	0.0301 (7)	
N3	0.15694 (10)	0.2550 (4)	0.2523 (2)	0.0310 (7)	
C1	0.03944 (12)	0.3100 (5)	0.0400 (3)	0.0362 (10)	
H1A	0.0349	0.3647	0.0907	0.043*	
C2	0.00547 (13)	0.2210 (6)	-0.0332 (3)	0.0425 (11)	
H2A	-0.0225	0.2177	-0.0343	0.051*	
C3	0.01180 (14)	0.1367 (6)	-0.1051 (3)	0.0476 (11)	
H3A	-0.0115	0.0715	-0.1557	0.057*	
C4	0.05248 (14)	0.1471 (6)	-0.1034 (3)	0.0439 (11)	

## supplementary materials

H4A	0.0576	0.0897	-0.1527	0.053*	
C5	0.08561 (12)	0.2433 (5)	-0.0281 (3)	0.0334 (9)	
C6	0.12956 (13)	0.2725 (6)	-0.0253 (3)	0.0373 (10)	
H6A	0.1379	0.1584	-0.0494	0.045*	
H6B	0.1279	0.3779	-0.0684	0.045*	
C7	0.20174 (12)	0.3989 (6)	0.0690 (3)	0.0399 (10)	
H7A	0.2138	0.3098	0.0392	0.060*	
H7B	0.2241	0.4296	0.1344	0.060*	
H7C	0.1927	0.5134	0.0300	0.060*	
C8	0.17688 (12)	0.1338 (5)	0.1299 (3)	0.0339 (9)	
H8A	0.2007	0.0754	0.1197	0.041*	
H8B	0.1518	0.0460	0.1058	0.041*	
C9	0.19227 (12)	0.1686 (5)	0.2357 (3)	0.0310 (9)	
H9A	0.2012	0.0485	0.2714	0.037*	
H9B	0.2180	0.2528	0.2604	0.037*	
C10	0.12260 (14)	0.1127 (6)	0.2399 (3)	0.0462 (11)	
H10A	0.1354	0.0147	0.2891	0.069*	
H10B	0.1114	0.0564	0.1754	0.069*	
H10C	0.0986	0.1739	0.2471	0.069*	
C11	0.17554 (14)	0.3223 (6)	0.3534 (3)	0.0466 (11)	
H11A	0.1968	0.4236	0.3625	0.056*	
H11B	0.1518	0.3759	0.3655	0.056*	
C12	0.19758 (15)	0.1724 (7)	0.4245 (3)	0.0495 (12)	
N4	0.21605 (14)	0.0568 (6)	0.4803 (3)	0.0660 (12)	
C1S	0.0000	0.3867 (12)	0.2500	0.081 (2)	
H1S1	-0.0264	0.3589	0.1902	0.122*	0.50
H1S2	0.0177	0.4799	0.2369	0.122*	0.50
H1S3	-0.0084	0.4363	0.2983	0.122*	0.50
O1S	0.0231 (3)	0.2310 (11)	0.2834 (5)	0.103 (3)	0.50
H1S	0.0438	0.2424	0.3402	0.155*	0.50

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Fe1	0.0251 (3)	0.0238 (3)	0.0325 (3)	-0.0011 (2)	0.0090 (2)	-0.0001 (2)
Cl1	0.0361 (6)	0.0348 (6)	0.0520 (7)	-0.0047 (5)	0.0100 (5)	-0.0027 (5)
Cl2	0.0341 (6)	0.0442 (6)	0.0442 (6)	0.0051 (5)	0.0150 (5)	-0.0080 (5)
Cl3	0.0434 (6)	0.0320 (6)	0.0460 (6)	-0.0021 (5)	0.0083 (5)	0.0112 (5)
N1	0.0254 (18)	0.0280 (18)	0.0329 (18)	0.0023 (14)	0.0090 (14)	0.0036 (14)
N2	0.0262 (18)	0.0366 (19)	0.0250 (17)	-0.0030 (14)	0.0095 (14)	-0.0019 (14)
N3	0.0283 (18)	0.0358 (19)	0.0262 (17)	-0.0009 (14)	0.0098 (15)	-0.0019 (14)
C1	0.028 (2)	0.033 (2)	0.045 (2)	-0.0015 (18)	0.014 (2)	0.0012 (19)
C2	0.029 (2)	0.040 (2)	0.048 (3)	0.0003 (19)	0.008 (2)	0.013 (2)
C3	0.033 (3)	0.046 (3)	0.042 (3)	-0.007 (2)	-0.002 (2)	0.004 (2)
C4	0.044 (3)	0.045 (3)	0.029 (2)	-0.004 (2)	0.0046 (19)	-0.0008 (19)
C5	0.032 (2)	0.033 (2)	0.027 (2)	-0.0023 (17)	0.0048 (19)	0.0036 (18)
C6	0.038 (2)	0.046 (2)	0.027 (2)	-0.0031 (19)	0.0134 (19)	-0.0022 (18)
C7	0.035 (2)	0.048 (3)	0.042 (2)	-0.006 (2)	0.022 (2)	0.000 (2)

C8	0.030 (2)	0.034 (2)	0.035 (2)	0.0039 (18)	0.0116 (18)	-0.0020 (18)
C9	0.029 (2)	0.028 (2)	0.036 (2)	0.0025 (17)	0.0140 (18)	0.0016 (17)
C10	0.042 (3)	0.042 (3)	0.055 (3)	-0.002 (2)	0.022 (2)	0.016 (2)
C11	0.051 (3)	0.049 (3)	0.034 (2)	0.011 (2)	0.014 (2)	-0.002 (2)
C12	0.056 (3)	0.052 (3)	0.041 (3)	0.004 (2)	0.022 (2)	0.005 (2)
N4	0.071 (3)	0.071 (3)	0.054 (3)	0.007 (2)	0.027 (2)	0.017 (2)
C1S	0.073 (6)	0.071 (6)	0.119 (7)	0.000	0.061 (5)	0.000
O1S	0.121 (7)	0.052 (5)	0.074 (6)	0.008 (5)	-0.012 (5)	0.008 (4)

*Geometric parameters (Å, °)*

Fe1—N1	2.186 (3)	C6—H6A	0.990
Fe1—N2	2.235 (3)	C6—H6B	0.990
Fe1—N3	2.330 (3)	C7—H7A	0.980
Fe1—Cl1	2.2873 (11)	C7—H7B	0.980
Fe1—Cl2	2.2908 (11)	C7—H7C	0.980
Fe1—Cl3	2.3284 (11)	C8—C9	1.500 (5)
N1—C1	1.341 (5)	C8—H8A	0.990
N1—C5	1.345 (5)	C8—H8B	0.990
N2—C6	1.484 (5)	C9—H9A	0.990
N2—C7	1.484 (4)	C9—H9B	0.990
N2—C8	1.497 (5)	C10—H10A	0.980
N3—C9	1.479 (4)	C10—H10B	0.980
N3—C11	1.480 (5)	C10—H10C	0.980
N3—C10	1.499 (5)	C11—C12	1.476 (6)
C1—C2	1.362 (5)	C11—H11A	0.990
C1—H1A	0.950	C11—H11B	0.990
C2—C3	1.365 (6)	C12—N4	1.155 (5)
C2—H2A	0.950	C1S—O1S	1.326 (9)
C3—C4	1.383 (6)	C1S—H1S1	0.980
C3—H3A	0.950	C1S—H1S2	0.980
C4—C5	1.388 (5)	C1S—H1S3	0.980
C4—H4A	0.950	O1S—O1S <sup>i</sup>	1.449 (16)
C5—C6	1.501 (5)	O1S—H1S	0.850
N1—Fe1—N2	75.33 (11)	N2—C6—H6A	109.4
N1—Fe1—Cl1	169.08 (9)	C5—C6—H6A	109.4
N2—Fe1—Cl1	93.76 (8)	N2—C6—H6B	109.4
N1—Fe1—Cl2	93.27 (8)	C5—C6—H6B	109.4
N2—Fe1—Cl2	162.33 (8)	H6A—C6—H6B	108.0
Cl1—Fe1—Cl2	97.20 (4)	N2—C7—H7A	109.5
N1—Fe1—Cl3	85.73 (8)	N2—C7—H7B	109.5
N2—Fe1—Cl3	92.44 (8)	H7A—C7—H7B	109.5
Cl1—Fe1—Cl3	95.42 (4)	N2—C7—H7C	109.5
Cl2—Fe1—Cl3	100.25 (4)	H7A—C7—H7C	109.5
N1—Fe1—N3	89.21 (10)	H7B—C7—H7C	109.5
N2—Fe1—N3	78.53 (10)	N2—C8—C9	110.6 (3)
Cl1—Fe1—N3	88.06 (8)	N2—C8—H8A	109.5
Cl2—Fe1—N3	88.00 (8)	C9—C8—H8A	109.5
Cl3—Fe1—N3	170.54 (8)	N2—C8—H8B	109.5

## supplementary materials

---

C1—N1—C5	118.7 (3)	C9—C8—H8B	109.5
C1—N1—Fe1	125.4 (3)	H8A—C8—H8B	108.1
C5—N1—Fe1	115.2 (2)	N3—C9—C8	110.2 (3)
C6—N2—C7	108.6 (3)	N3—C9—H9A	109.6
C6—N2—C8	108.8 (3)	C8—C9—H9A	109.6
C7—N2—C8	109.2 (3)	N3—C9—H9B	109.6
C6—N2—Fe1	107.1 (2)	C8—C9—H9B	109.6
C7—N2—Fe1	113.4 (2)	H9A—C9—H9B	108.1
C8—N2—Fe1	109.6 (2)	N3—C10—H10A	109.5
C9—N3—C11	108.8 (3)	N3—C10—H10B	109.5
C9—N3—C10	110.5 (3)	H10A—C10—H10B	109.5
C11—N3—C10	107.1 (3)	N3—C10—H10C	109.5
C9—N3—Fe1	103.9 (2)	H10A—C10—H10C	109.5
C11—N3—Fe1	111.9 (2)	H10B—C10—H10C	109.5
C10—N3—Fe1	114.5 (2)	C12—C11—N3	112.8 (3)
N1—C1—C2	122.3 (4)	C12—C11—H11A	109.0
N1—C1—H1A	118.9	N3—C11—H11A	109.0
C2—C1—H1A	118.9	C12—C11—H11B	109.0
C1—C2—C3	119.6 (4)	N3—C11—H11B	109.0
C1—C2—H2A	120.2	H11A—C11—H11B	107.8
C3—C2—H2A	120.2	N4—C12—C11	177.8 (5)
C2—C3—C4	119.3 (4)	O1S—C1S—H1S1	109.5
C2—C3—H3A	120.4	O1S—C1S—H1S2	109.5
C4—C3—H3A	120.4	H1S1—C1S—H1S2	109.5
C3—C4—C5	118.5 (4)	O1S—C1S—H1S3	109.5
C3—C4—H4A	120.7	H1S1—C1S—H1S3	109.5
C5—C4—H4A	120.7	H1S2—C1S—H1S3	109.5
N1—C5—C4	121.6 (4)	C1S—O1S—O1S <sup>i</sup>	56.9 (4)
N1—C5—C6	116.8 (3)	C1S—O1S—H1S	113.4
C4—C5—C6	121.5 (4)	O1S <sup>i</sup> —O1S—H1S	150.8
N2—C6—C5	111.1 (3)		
N2—Fe1—N1—C1	168.7 (3)	Cl2—Fe1—N3—C11	-51.8 (2)
Cl1—Fe1—N1—C1	165.9 (3)	N1—Fe1—N3—C10	-22.9 (3)
Cl2—Fe1—N1—C1	2.4 (3)	N2—Fe1—N3—C10	-98.1 (3)
Cl3—Fe1—N1—C1	-97.6 (3)	Cl1—Fe1—N3—C10	167.7 (3)
N3—Fe1—N1—C1	90.4 (3)	Cl2—Fe1—N3—C10	70.4 (2)
N2—Fe1—N1—C5	-21.2 (2)	C5—N1—C1—C2	-0.9 (5)
Cl1—Fe1—N1—C5	-24.1 (6)	Fe1—N1—C1—C2	168.8 (3)
Cl2—Fe1—N1—C5	172.5 (2)	N1—C1—C2—C3	2.0 (6)
Cl3—Fe1—N1—C5	72.4 (2)	C1—C2—C3—C4	-1.6 (6)
N3—Fe1—N1—C5	-99.6 (3)	C2—C3—C4—C5	0.1 (6)
N1—Fe1—N2—C6	32.1 (2)	C1—N1—C5—C4	-0.6 (5)
Cl1—Fe1—N2—C6	-148.5 (2)	Fe1—N1—C5—C4	-171.3 (3)
Cl2—Fe1—N2—C6	83.2 (3)	C1—N1—C5—C6	176.0 (3)
Cl3—Fe1—N2—C6	-52.9 (2)	Fe1—N1—C5—C6	5.3 (4)
N3—Fe1—N2—C6	124.3 (2)	C3—C4—C5—N1	1.0 (6)
N1—Fe1—N2—C7	151.8 (3)	C3—C4—C5—C6	-175.5 (4)
Cl1—Fe1—N2—C7	-28.7 (2)	C7—N2—C6—C5	-162.3 (3)



C12—Fe1—N2—C7	-157.0 (2)	C8—N2—C6—C5	78.9 (4)
C13—Fe1—N2—C7	66.9 (2)	Fe1—N2—C6—C5	-39.5 (4)
N3—Fe1—N2—C7	-116.0 (2)	N1—C5—C6—N2	24.1 (5)
N1—Fe1—N2—C8	-85.8 (2)	C4—C5—C6—N2	-159.3 (4)
C11—Fe1—N2—C8	93.7 (2)	C6—N2—C8—C9	-152.1 (3)
C12—Fe1—N2—C8	-34.7 (4)	C7—N2—C8—C9	89.5 (4)
C13—Fe1—N2—C8	-170.7 (2)	Fe1—N2—C8—C9	-35.3 (3)
N3—Fe1—N2—C8	6.4 (2)	C11—N3—C9—C8	-168.7 (3)
N1—Fe1—N3—C9	97.7 (2)	C10—N3—C9—C8	74.0 (4)
N2—Fe1—N3—C9	22.5 (2)	Fe1—N3—C9—C8	-49.3 (3)
C11—Fe1—N3—C9	-71.7 (2)	N2—C8—C9—N3	59.5 (4)
C12—Fe1—N3—C9	-169.0 (2)	C9—N3—C11—C12	-56.6 (4)
N1—Fe1—N3—C11	-145.1 (3)	C10—N3—C11—C12	62.9 (4)
N2—Fe1—N3—C11	139.7 (3)	Fe1—N3—C11—C12	-170.8 (3)
C11—Fe1—N3—C11	45.5 (2)		

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

Fig. 1

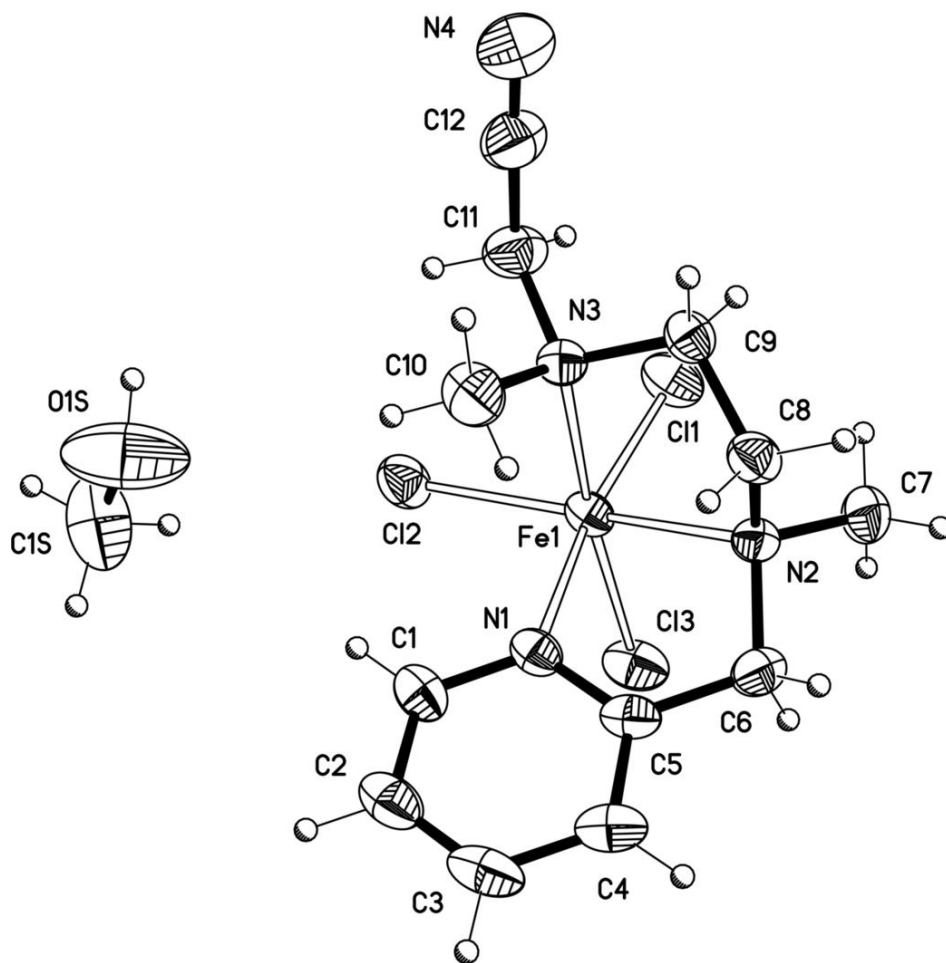


Fig. 2

