

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

***trans*-Bis[2,5-bis(pyridin-2-yl)-1,3,4-thiadiazole- $\kappa^2N^2,N^3$ ]bis(methanol- $\kappa O$ )-iron(II) bis(perchlorate)**

Dominic Kaase and Julia Klingele\*

Institut für Anorganische und Analytische Chemie, Albert-Ludwigs-Universität Freiburg, Albertstrasse 21, 79104 Freiburg i. Br., Germany  
Correspondence e-mail: julia.klingele@ac.uni-freiburg.de

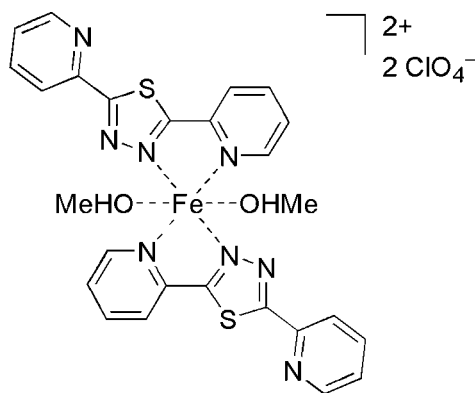
Received 16 May 2014; accepted 2 June 2014

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(C-C) = 0.003$  Å;  $R$  factor = 0.027;  $wR$  factor = 0.062; data-to-parameter ratio = 13.8.

The title compound,  $[Fe(C_{12}H_8N_4S)_2(CH_3OH)_2](ClO_4)_2$ , crystallized in the solvent-free form from a methanol solution. The  $Fe^{II}$  ion is located on a centre of inversion. The distorted  $N_4O_2$  octahedral coordination geometry is formed by two  $N,N'$ -chelating equatorial 2,5-bis(pyridin-2-yl)-1,3,4-thiadiazole ligands and axially coordinating methanol coligands, resulting in the mononuclear *trans*-( $N^2,N^3,O$ )<sub>2</sub> coordination mode. The methanol co-ligand is involved in a hydrogen bond to the perchlorate counter-ion.

## Related literature

For other 3d metal structures of 2,5-bis(pyridin-2-yl)-1,3,4-thiadiazole, see: Klingele *et al.* (2010, 2012); Bentiss, Lagrenee, Mentre *et al.* (2004); Bentiss, Lagrenee, Vezin *et al.* (2004); Zheng *et al.* (2006); Bentiss *et al.* (2002); Wan *et al.* (2007). For related compounds, see: Guionneau *et al.* (2004). For the bridging capability of 4,4'-bispyridine- $N,N'$ -dioxide, see: Jia *et al.* (2008).



## Experimental

## Crystal data

$[Fe(C_{12}H_8N_4S)_2(CH_3OH)_2](ClO_4)_2$   $\gamma = 74.735$  (2) $^\circ$   
 $M_r = 799.40$   $V = 780.61$  (5) Å<sup>3</sup>  
 Triclinic,  $P\bar{1}$   $Z = 1$   
 $a = 8.8410$  (3) Å Mo  $K\alpha$  radiation  
 $b = 9.5579$  (4) Å  $\mu = 0.86$  mm<sup>-1</sup>  
 $c = 9.5875$  (4) Å  $T = 100$  K  
 $\alpha = 87.169$  (2) $^\circ$   $0.18 \times 0.08 \times 0.03$  mm  
 $\beta = 88.945$  (2) $^\circ$

## Data collection

Bruker APEXII CCD area-detector diffractometer 18025 measured reflections  
 3128 independent reflections  
 Absorption correction: multi-scan (SADABS; Bruker, 2001) 2789 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.019$   
 $T_{min} = 0.861$ ,  $T_{max} = 0.975$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$  H atoms treated by a mixture of independent and constrained refinement  
 $wR(F^2) = 0.062$   $\Delta\rho_{max} = 0.49$  e Å<sup>-3</sup>  
 $S = 1.05$   $\Delta\rho_{min} = -0.38$  e Å<sup>-3</sup>  
 3128 reflections  
 227 parameters

Table 1

Hydrogen-bond geometry (Å,  $^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O20-H20\cdots O11$	0.78 (3)	1.91 (3)	2.690 (2)	178 (3)

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: OLEX2.refine (Puschmann *et al.*, 2013); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) and OLEX2.refine (Puschmann *et al.*, 2013); molecular graphics: DIAMOND (Brandenburg & Putz, 2011); software used to prepare material for publication: SHELXL97.

Financial support by the European Social Fund, by the Ministry of Science, Research and the Arts Baden-Württemberg within the Margarete von Wrangell Program, the Baden-Württemberg Stiftung within the Eliteprogramme for Postdocs, the Fonds der Chemischen Industrie (FCI) and the Universität Freiburg is gratefully acknowledged.

Supporting information for this paper is available from the IUCr electronic archives (Reference: LR2128).

## References

- Bentiss, F., Lagrenee, M., Mentre, O., Conflant, P., Vezin, H., Wignacourt, J. P. & Holt, E. M. (2004). *Inorg. Chem.* **43**, 1865–1873.  
 Bentiss, F., Lagrenee, M., Vezin, H., Wignacourt, J.-P. & Holt, E. M. (2004). *Polyhedron*, **23**, 1903–1907.  
 Bentiss, F., Lagrenee, M., Wignacourt, J. P. & Holt, E. M. (2002). *Polyhedron*, **21**, 403–408.  
 Brandenburg, K. & Putz, H. (2011). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.  
 Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Bruker (2007). *APEX2* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Guionneau, P., Marchivie, M., Bravic, G., Létard, J.-F. & Chasseau, D. (2004). *Top. Curr. Chem.* **234**, 97–128.

- Jia, J., Blake, A. J., Champness, N. R., Hubberstey, P., Wilson, C. & Schröder, M. (2008). *Inorg. Chem.* **47**, 8652–8664.
- Klinge, J., Kaase, D., Klinge, M. H. & Lach, J. (2012). *Dalton Trans.* **41**, 1397–1406.
- Klinge, J., Kaase, D., Klinge, M. H., Lach, J. & Demeshko, S. (2010). *Dalton Trans.* **39**, 1689–1691.
- Puschmann, H., Bourhis, L. J., Dolomanov, O. V., Gildea, R. J. & Howard, J. A. K. (2013). *Acta Cryst. A* **69**, s679.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Wan, X.-S., Ning, A.-M., Hou, S.-C., Niu, C.-Y., Kou, C.-H. & Dang, Y.-L. (2007). *Z. Kristallogr. New Cryst. Struct.* **222**, 153–154.
- Zheng, X.-F., Wan, X.-S., Liu, W., Niu, C.-Y. & Kou, C.-H. (2006). *Z. Kristallogr. New Cryst. Struct.* **221**, 543–544.

## supplementary materials

*Acta Cryst.* (2014). E70, m252–m253 [doi:10.1107/S160053681401277X]

***trans*-Bis[2,5-bis(pyridin-2-yl)-1,3,4-thiadiazole- $\kappa^2N^2,N^3$ ]bis(methanol- $\kappa O$ )iron(II) bis(perchlorate)**

**Dominic Kaase and Julia Klingele**

### 1. Introduction

The ligand 2,5-bis(pyridin-2-yl)-1,3,4-thiadiazole (**L**) is well known to have a suitable ligand field for the preparation of iron(II) spin crossover complexes (Klingele, *et al.*, 2010). Known examples are the 2:1-type complexes  $[\text{Fe}(\text{L})_2(\text{NCS})_2]$ ,  $[\text{Fe}(\text{L})_2(\text{NCSe})_2]$  and  $[\text{Fe}(\text{L})_2(\text{NCBH}_3)_2]$  (Klingele *et al.*, 2012). 4,4'-Bispyridine-*N,N'*-dioxide is able to bridge metal ions to form multidimensional structures (Jia *et al.*, 2008). The title compound  $[\text{Fe}(\text{L})_2(\text{MeOH})_2](\text{ClO}_4)_2$  was obtained unintentionally in the attempt to isolate one-dimensional chains of 4,4'-bispyridine-*N,N'*-dioxide-bridged  $[\text{Fe}(\text{L})_2]^{2+}$  units.

### 2. Experimental

#### 2.1. Synthesis and crystallization

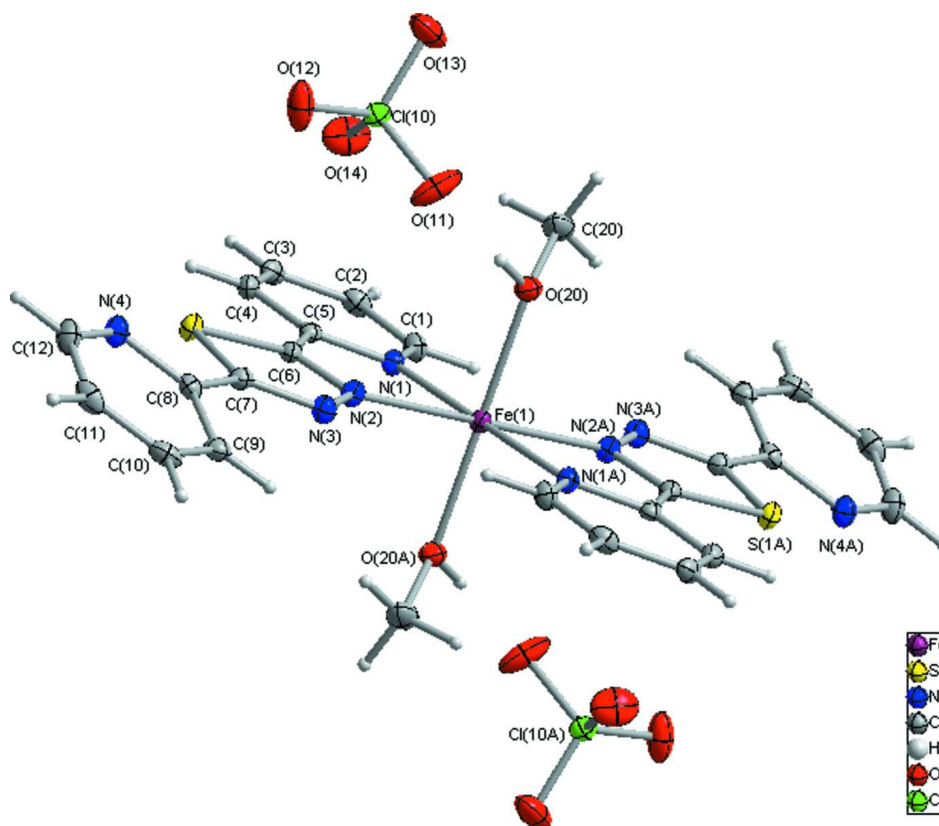
Single crystals suitable for X-ray diffraction of the title compound were obtained unexpectedly by layering a MeOH solution of iron(II) perchlorate with a MeOH solution of 2,5-di(pyridin-2-yl)-1,3,4-thiadiazole and 4,4'-bispyridine-*N,N'*-dioxide in an argon atmosphere.

#### 2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in the Table below. All hydrogen atoms of the ligand and of the methyl hydrogen atoms of the methanol coligand were positioned geometrically and refined using a riding model. The hydrogen atom of the methanol hydroxyl group was located from the difference maps and refined freely and isotropically.

### 3. Results and discussion

The ligand **L** was synthesized according to literature procedure (Klingele *et al.*, 2012). A single-crystal of  $[\text{Fe}(\text{L})_2(\text{MeOH})_2](\text{ClO}_4)_2$  suitable for X-ray diffraction was obtained unexpectedly by layering a MeOH solution of iron(II) perchlorate with a MeOH solution of **L** and 4,4'-bispyridine-*N,N'*-dioxide. The mononuclear complex cation is formed by high-spin iron(II) ion coordinated by two bidentate ligands **L** and two MeOH coligands. The Fe—N [Fe—N<sub>pyr</sub> 2.1516 (15), Fe—N<sub>tda</sub> 2.2015 (15) Å] and Fe—O [2.0886 (13) Å] distances are in the expected range for high-spin iron(II) (Guionneau *et al.*, 2004).


**Figure 1**

View of the complex  $[\text{Fe}(\text{L})_2(\text{MeOH})_2](\text{ClO}_4)_2$ . Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn with arbitrary radii. [Symmetry code: (A)  $-x, -y+2, -z$ .]

***trans*-Bis[2,5-bis(pyridin-2-yl)-1,3,4-thiadiazole- $\kappa^2$ N<sup>2</sup>,N<sup>3</sup>]bis(methanol- $\kappa$ O)iron(II) bis(perchlorate)**

*Crystal data*

$[\text{Fe}(\text{C}_{12}\text{H}_8\text{N}_4\text{S})_2(\text{CH}_4\text{O})_2](\text{ClO}_4)_2$

$M_r = 799.40$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 8.8410(3)\ \text{\AA}$

$b = 9.5579(4)\ \text{\AA}$

$c = 9.5875(4)\ \text{\AA}$

$\alpha = 87.169(2)^\circ$

$\beta = 88.945(2)^\circ$

$\gamma = 74.735(2)^\circ$

$V = 780.61(5)\ \text{\AA}^3$

$Z = 1$

$F(000) = 408$

$D_x = 1.701\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 9969 reflections

$\theta = 2.2\text{--}30.5^\circ$

$\mu = 0.86\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Plate, red

$0.18 \times 0.08 \times 0.03\ \text{mm}$

*Data collection*

Bruker APEXII CCD area-detector  
diffractometer

Radiation source: microfocus sealed tube  
Multilayer mirror optics monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2001)

$T_{\min} = 0.861, T_{\max} = 0.975$

18025 measured reflections

3128 independent reflections

2789 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\text{max}} = 26.4^\circ, \theta_{\text{min}} = 2.1^\circ$

$h = -11 \rightarrow 11$

$k = -11 \rightarrow 11$

$l = -11 \rightarrow 11$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.062$   
 $S = 1.05$   
 3128 reflections  
 227 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0172P)^2 + 0.9493P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.49 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.0000	1.0000	0.0000	0.01139 (10)
S1	0.22171 (5)	0.51392 (5)	0.06646 (5)	0.01390 (11)
N1	0.18464 (17)	0.89318 (16)	-0.14602 (15)	0.0128 (3)
N2	0.05730 (17)	0.77550 (16)	0.07183 (16)	0.0131 (3)
N3	-0.00347 (18)	0.70371 (17)	0.17709 (16)	0.0144 (3)
N4	0.14375 (19)	0.33511 (17)	0.30380 (17)	0.0171 (3)
C1	0.2469 (2)	0.9547 (2)	-0.25294 (19)	0.0154 (4)
H1	0.2034	1.0550	-0.2760	0.018*
C2	0.3724 (2)	0.8783 (2)	-0.33180 (19)	0.0162 (4)
H2	0.4130	0.9255	-0.4075	0.019*
C3	0.4371 (2)	0.7330 (2)	-0.2986 (2)	0.0154 (4)
H3	0.5228	0.6786	-0.3510	0.018*
C4	0.3748 (2)	0.6677 (2)	-0.18715 (19)	0.0138 (4)
H4	0.4174	0.5679	-0.1617	0.017*
C5	0.2495 (2)	0.75062 (19)	-0.11400 (18)	0.0115 (4)
C6	0.1740 (2)	0.69113 (19)	0.00391 (18)	0.0118 (4)
C7	0.0705 (2)	0.5668 (2)	0.18660 (19)	0.0131 (4)
C8	0.0312 (2)	0.4599 (2)	0.28666 (19)	0.0131 (4)
C9	-0.1131 (2)	0.4898 (2)	0.35465 (19)	0.0158 (4)
H9	-0.1888	0.5801	0.3387	0.019*
C10	-0.1429 (2)	0.3835 (2)	0.4465 (2)	0.0186 (4)
H10	-0.2402	0.3992	0.4949	0.022*
C11	-0.0285 (2)	0.2540 (2)	0.4665 (2)	0.0200 (4)
H11	-0.0462	0.1794	0.5288	0.024*
C12	0.1125 (2)	0.2350 (2)	0.3942 (2)	0.0201 (4)

H12	0.1908	0.1463	0.4098	0.024*
C20	-0.2991 (2)	0.9010 (2)	-0.0662 (2)	0.0234 (5)
H20A	-0.3466	0.8617	-0.1416	0.035*
H20B	-0.3792	0.9775	-0.0223	0.035*
H20C	-0.2552	0.8232	0.0036	0.035*
O20	-0.17589 (15)	0.96075 (15)	-0.12275 (15)	0.0173 (3)
H20	-0.212 (3)	1.021 (3)	-0.179 (3)	0.026*
Cl10	-0.46099 (5)	1.25579 (5)	-0.29619 (5)	0.01521 (11)
O11	-0.30426 (17)	1.16340 (19)	-0.32171 (16)	0.0353 (4)
O12	-0.47275 (19)	1.39590 (16)	-0.36152 (17)	0.0320 (4)
O13	-0.57261 (18)	1.19384 (18)	-0.35684 (18)	0.0339 (4)
O14	-0.48818 (18)	1.26721 (19)	-0.14843 (15)	0.0327 (4)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Fe1	0.01072 (18)	0.00969 (19)	0.01282 (19)	-0.00126 (14)	0.00130 (14)	0.00030 (14)
S1	0.0141 (2)	0.0101 (2)	0.0162 (2)	-0.00128 (17)	0.00275 (17)	0.00076 (18)
N1	0.0137 (8)	0.0124 (8)	0.0118 (8)	-0.0027 (6)	0.0007 (6)	-0.0007 (6)
N2	0.0133 (7)	0.0125 (8)	0.0128 (8)	-0.0024 (6)	0.0018 (6)	0.0005 (6)
N3	0.0159 (8)	0.0134 (8)	0.0136 (8)	-0.0042 (6)	0.0017 (6)	0.0012 (6)
N4	0.0202 (8)	0.0136 (8)	0.0171 (8)	-0.0041 (7)	0.0023 (7)	0.0009 (7)
C1	0.0184 (9)	0.0129 (9)	0.0153 (9)	-0.0048 (8)	0.0005 (7)	-0.0001 (7)
C2	0.0177 (9)	0.0198 (10)	0.0134 (9)	-0.0090 (8)	0.0025 (7)	-0.0020 (8)
C3	0.0124 (9)	0.0184 (10)	0.0163 (9)	-0.0047 (7)	0.0026 (7)	-0.0070 (8)
C4	0.0137 (9)	0.0115 (9)	0.0157 (9)	-0.0021 (7)	-0.0010 (7)	-0.0026 (7)
C5	0.0119 (8)	0.0128 (9)	0.0104 (8)	-0.0041 (7)	-0.0022 (7)	-0.0014 (7)
C6	0.0117 (8)	0.0109 (9)	0.0126 (9)	-0.0026 (7)	-0.0025 (7)	-0.0009 (7)
C7	0.0115 (9)	0.0152 (9)	0.0127 (9)	-0.0036 (7)	-0.0007 (7)	-0.0015 (7)
C8	0.0159 (9)	0.0129 (9)	0.0118 (9)	-0.0060 (7)	-0.0007 (7)	-0.0015 (7)
C9	0.0152 (9)	0.0170 (10)	0.0161 (9)	-0.0057 (8)	-0.0035 (7)	-0.0003 (8)
C10	0.0184 (10)	0.0262 (11)	0.0153 (10)	-0.0130 (8)	0.0002 (8)	-0.0026 (8)
C11	0.0301 (11)	0.0198 (10)	0.0144 (10)	-0.0145 (9)	0.0005 (8)	0.0011 (8)
C12	0.0272 (11)	0.0131 (10)	0.0189 (10)	-0.0037 (8)	-0.0001 (8)	0.0015 (8)
C20	0.0144 (10)	0.0255 (11)	0.0320 (12)	-0.0074 (8)	0.0032 (8)	-0.0068 (9)
O20	0.0145 (7)	0.0178 (7)	0.0192 (7)	-0.0037 (6)	-0.0026 (5)	0.0009 (6)
Cl10	0.0138 (2)	0.0166 (2)	0.0151 (2)	-0.00390 (17)	0.00130 (17)	0.00016 (18)
O11	0.0177 (8)	0.0490 (11)	0.0267 (9)	0.0102 (7)	0.0051 (6)	0.0129 (8)
O12	0.0452 (10)	0.0162 (8)	0.0349 (9)	-0.0095 (7)	0.0194 (7)	-0.0019 (7)
O13	0.0289 (9)	0.0383 (10)	0.0423 (10)	-0.0201 (7)	0.0035 (7)	-0.0160 (8)
O14	0.0312 (9)	0.0527 (11)	0.0135 (7)	-0.0105 (8)	0.0059 (6)	-0.0004 (7)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Fe1—O20	2.0886 (13)	C1—C2	1.391 (3)
Fe1—O20 <sup>i</sup>	2.0886 (13)	C2—C3	1.379 (3)
Fe1—N2 <sup>i</sup>	2.1516 (15)	C3—C4	1.389 (3)
Fe1—N2	2.1516 (15)	C4—C5	1.382 (3)
Fe1—N1 <sup>i</sup>	2.2015 (15)	C5—C6	1.468 (2)
Fe1—N1	2.2015 (15)	C7—C8	1.471 (2)

S1—C6	1.7144 (18)	C8—C9	1.389 (3)
S1—C7	1.7364 (19)	C9—C10	1.386 (3)
N1—C1	1.338 (2)	C10—C11	1.384 (3)
N1—C5	1.354 (2)	C11—C12	1.389 (3)
N2—C6	1.315 (2)	C20—O20	1.443 (2)
N2—N3	1.372 (2)	Cl10—O13	1.4232 (15)
N3—C7	1.299 (2)	Cl10—O12	1.4292 (15)
N4—C12	1.339 (2)	Cl10—O14	1.4372 (15)
N4—C8	1.342 (2)	Cl10—O11	1.4574 (15)
O20—Fe1—O20 <sup>i</sup>	180.00 (4)	C2—C3—C4	118.88 (17)
O20—Fe1—N2 <sup>i</sup>	91.48 (6)	C5—C4—C3	118.73 (17)
O20 <sup>i</sup> —Fe1—N2 <sup>i</sup>	88.52 (6)	N1—C5—C4	122.90 (16)
O20—Fe1—N2	88.52 (6)	N1—C5—C6	114.25 (16)
O20 <sup>i</sup> —Fe1—N2	91.48 (6)	C4—C5—C6	122.85 (16)
N2 <sup>i</sup> —Fe1—N2	180.0	N2—C6—C5	120.39 (16)
O20—Fe1—N1 <sup>i</sup>	87.99 (5)	N2—C6—S1	113.48 (13)
O20 <sup>i</sup> —Fe1—N1 <sup>i</sup>	92.01 (5)	C5—C6—S1	126.13 (14)
N2 <sup>i</sup> —Fe1—N1 <sup>i</sup>	76.37 (6)	N3—C7—C8	124.64 (17)
N2—Fe1—N1 <sup>i</sup>	103.63 (6)	N3—C7—S1	114.73 (13)
O20—Fe1—N1	92.01 (5)	C8—C7—S1	120.61 (14)
O20 <sup>i</sup> —Fe1—N1	87.99 (5)	N4—C8—C9	124.49 (17)
N2 <sup>i</sup> —Fe1—N1	103.63 (6)	N4—C8—C7	114.70 (16)
N2—Fe1—N1	76.37 (6)	C9—C8—C7	120.81 (17)
N1 <sup>i</sup> —Fe1—N1	180.0	C10—C9—C8	117.74 (18)
C6—S1—C7	86.84 (9)	C11—C10—C9	118.93 (18)
C1—N1—C5	117.61 (16)	C10—C11—C12	118.91 (18)
C1—N1—Fe1	127.76 (12)	N4—C12—C11	123.43 (19)
C5—N1—Fe1	114.39 (11)	C20—O20—Fe1	123.03 (12)
C6—N2—N3	113.63 (15)	O13—Cl10—O12	109.10 (10)
C6—N2—Fe1	114.27 (12)	O13—Cl10—O14	110.30 (10)
N3—N2—Fe1	132.09 (12)	O12—Cl10—O14	110.33 (10)
C7—N3—N2	111.33 (15)	O13—Cl10—O11	108.75 (10)
C12—N4—C8	116.48 (17)	O12—Cl10—O11	108.62 (9)
N1—C1—C2	122.82 (17)	O14—Cl10—O11	109.71 (9)
C3—C2—C1	119.05 (17)		
O20—Fe1—N1—C1	92.67 (15)	Fe1—N2—C6—C5	-2.5 (2)
O20 <sup>i</sup> —Fe1—N1—C1	-87.33 (15)	N3—N2—C6—S1	-0.96 (19)
N2 <sup>i</sup> —Fe1—N1—C1	0.65 (16)	Fe1—N2—C6—S1	178.04 (8)
N2—Fe1—N1—C1	-179.35 (16)	N1—C5—C6—N2	-2.2 (2)
O20—Fe1—N1—C5	-93.28 (12)	C4—C5—C6—N2	178.55 (16)
O20 <sup>i</sup> —Fe1—N1—C5	86.72 (12)	N1—C5—C6—S1	177.18 (13)
N2 <sup>i</sup> —Fe1—N1—C5	174.71 (12)	C4—C5—C6—S1	-2.0 (3)
N2—Fe1—N1—C5	-5.29 (12)	C7—S1—C6—N2	0.74 (14)
O20—Fe1—N2—C6	96.46 (13)	C7—S1—C6—C5	-178.71 (16)
O20 <sup>i</sup> —Fe1—N2—C6	-83.54 (13)	N2—N3—C7—C8	-178.46 (16)
N1 <sup>i</sup> —Fe1—N2—C6	-175.97 (12)	N2—N3—C7—S1	-0.07 (19)
N1—Fe1—N2—C6	4.03 (12)	C6—S1—C7—N3	-0.37 (15)

O20—Fe1—N2—N3	-84.78 (15)	C6—S1—C7—C8	178.09 (15)
O20 <sup>i</sup> —Fe1—N2—N3	95.22 (15)	C12—N4—C8—C9	-0.7 (3)
N1 <sup>i</sup> —Fe1—N2—N3	2.79 (16)	C12—N4—C8—C7	179.92 (17)
N1—Fe1—N2—N3	-177.21 (16)	N3—C7—C8—N4	-163.82 (17)
C6—N2—N3—C7	0.7 (2)	S1—C7—C8—N4	17.9 (2)
Fe1—N2—N3—C7	-178.11 (13)	N3—C7—C8—C9	16.8 (3)
C5—N1—C1—C2	0.7 (3)	S1—C7—C8—C9	-161.50 (14)
Fe1—N1—C1—C2	174.61 (13)	N4—C8—C9—C10	0.0 (3)
N1—C1—C2—C3	-0.5 (3)	C7—C8—C9—C10	179.33 (17)
C1—C2—C3—C4	-0.1 (3)	C8—C9—C10—C11	0.3 (3)
C2—C3—C4—C5	0.3 (3)	C9—C10—C11—C12	0.1 (3)
C1—N1—C5—C4	-0.4 (3)	C8—N4—C12—C11	1.2 (3)
Fe1—N1—C5—C4	-175.14 (13)	C10—C11—C12—N4	-0.9 (3)
C1—N1—C5—C6	-179.67 (15)	N2 <sup>i</sup> —Fe1—O20—C20	-124.06 (14)
Fe1—N1—C5—C6	5.64 (19)	N2—Fe1—O20—C20	55.94 (14)
C3—C4—C5—N1	-0.1 (3)	N1 <sup>i</sup> —Fe1—O20—C20	-47.76 (14)
C3—C4—C5—C6	179.08 (16)	N1—Fe1—O20—C20	132.24 (14)
N3—N2—C6—C5	178.53 (15)		

Symmetry code: (i)  $-x, -y+2, -z$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O20—H20 $\cdots$ O11	0.78 (3)	1.91 (3)	2.690 (2)	178 (3)