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# Hexakis(1*H*-imidazole-$\kappa N^3$)iron(II) sulfate–1*H*-imidazole (1/2)

**Alexandra Nistor,[a] Sergiu Shova,[b]* Maria Cazacu[a] and Alina Lazar[a]**

[a]Institute of Macromolecular Chemistry 'Petru Poni', Inorganic Polymers Department, 41A Grigore Ghica Voda Alley, Iasi-700487, Romania, and [b]Institute of Applied Physics of the Academy of Science of Moldova, 5 Academiei Street, Chisinau MD-2028, Republic of Moldova
Correspondence e-mail: shova@usm.md

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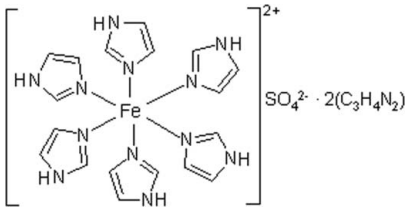
Key indicators: single-crystal X-ray study; $T$ = 200 K; mean $\sigma$(C–C) = 0.009 Å; $R$ factor = 0.065; $wR$ factor = 0.107; data-to-parameter ratio = 13.2.

The asymmetric unit of the title compound, $[Fe(C_3H_4N_2)_6]SO_4\cdot 2C_3H_4N_2$, contains two complex cations, two sulfate anions and four imidazole molecules. In both cations, the $Fe^{II}$ atom is coordinated by six monodentate imidazole ligands and exhibits a slightly distorted octahedral coordination geometry. The Fe—N distances [2.184 (4)–2.218 (4) Å] point to a high-spin state of the $Fe^{2+}$ ions. N—H···O hydrogen bonds between the ionic components generate a three-dimensional framework containing corrugated channels along [001], which are filled by **N—H···N** hydrogen-bonded imidazole chains.

## Related literature

For the crystal structures of other hexakis(imidazole)iron(II) salts, see: Carver *et al.* (2003); Jian *et al.* (2004). For spin crossover in complexes with the $FeN_6$ core, see: Gütlich & Goodwin (2004); Lemercier *et al.* (2006). For the influence of counter-ions and solvent molecules on spin crosover behaviour, see: Bousseksou *et al.* (1996).




## Experimental

### Crystal data

$[Fe(C_3H_4N_2)_6]SO_4\cdot 2C_3H_4N_2$
$M_r$ = 696.57
Triclinic, $P\overline{1}$
$a$ = 15.4091 (8) Å
$b$ = 15.4436 (7) Å
$c$ = 15.9883 (11) Å
$\alpha$ = 69.813 (5)°
$\beta$ = 69.949 (5)°
$\gamma$ = 73.214 (4)°
$V$ = 3291.3 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.58 mm$^{-1}$
$T$ = 200 K
0.20 × 0.15 × 0.15 mm

### Data collection

Oxford Diffraction Xcalibur E diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min}$ = 0.901, $T_{max}$ = 0.917
15987 measured reflections
10955 independent reflections
5185 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.107
$S$ = 0.85
10955 reflections
829 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.63 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.50 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H49···O4 | 0.88 | 1.84 | 2.679 (5) | 160 |
| N4—H50···O5 | 0.88 | 1.89 | 2.762 (5) | 170 |
| N6—H51···O3$^{i}$ | 0.88 | 2.09 | 2.949 (7) | 163 |
| N8—H52···O1$^{ii}$ | 0.88 | 1.94 | 2.822 (5) | 176 |
| N10—H53···O7$^{iii}$ | 0.88 | 1.85 | 2.711 (6) | 166 |
| N12—H54···O7$^{iv}$ | 0.88 | 2.10 | 2.867 (7) | 145 |
| N12—H54···O5$^{iv}$ | 0.88 | 2.61 | 3.434 (7) | 157 |
| N14—H55···O8$^{v}$ | 0.88 | 1.85 | 2.716 (5) | 167 |
| N16—H56···O1$^{vi}$ | 0.88 | 2.22 | 3.074 (6) | 163 |
| N16—H56···O4$^{vi}$ | 0.88 | 2.26 | 2.942 (6) | 135 |
| N18—H57···O6$^{vii}$ | 0.88 | 2.07 | 2.937 (6) | 169 |
| N18—H57···O8$^{vii}$ | 0.88 | 2.47 | 3.100 (6) | 129 |
| N20—H58···O6 | 0.88 | 2.21 | 3.071 (6) | 166 |
| N20—H58···O7 | 0.88 | 2.45 | 3.120 (6) | 133 |
| N22—H59···O2 | 0.88 | 2.18 | 2.939 (6) | 144 |
| N22—H59···O3 | 0.88 | 2.37 | 3.186 (6) | 155 |
| N24—H60···O2$^{viii}$ | 0.88 | 1.84 | 2.710 (6) | 168 |
| N26—H61···N29 | 0.88 | 1.95 | 2.825 (6) | 177 |
| N27—H62···N25 | 0.88 | 2.04 | 2.875 (6) | 158 |
| N30—H63···N32$^{ix}$ | 0.88 | 2.02 | 2.869 (6) | 160 |
| N31—H64···N28 | 0.88 | 1.92 | 2.800 (6) | 174 |

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $x, y+1, z$; (iii) $x+1, y, z$; (iv) $-x+1, -y+1, -z+1$; (v) $x, y-1, z$; (vi) $x-1, y, z$; (vii) $-x+1, -y+1, -z$; (viii) $-x+1, -y, -z+1$; (ix) $x, y, z-1$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2405).

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# supplementary materials

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# Hexakis(1*H*-imidazole-$\kappa N^3$)iron(II) sulfate-1*H*-imidazole (1/2)

**A. Nistor, S. Shova, M. Cazacu and A. Lazar**

## Comment

As part of our research of iron(II) complexes with mono- and polydentate N-containing ligands we report the synthesis and crystal structure of $[Fe(Im)_6]SO_4{\cdot}2Im$, where Im = imidazole. The cationic complexes $[Fe(Im)_6]^{2+}$ characterized by an $[FeN_6]$ coordination core are increasingly investigated as spin crossover (SC) materials, because their bistability (LS, S=0, $^1A_1$ <-> HS, S=2, $^5T_{2\,g}$) is easily triggered thermally, magnetically, by pressure or by light irradiation (Gütlich & Goodwin, 2004; Lemercier *et al.*, 2006).

The presence of sulfate counteranion in the title compound with respect to the earlier studied hexakis(imidazole)iron(II) dinitrate, $[Fe(Im)_6]2(NO_3)$ (Carver *et al.*, 2003) and hexakis(imidazole)iron(II) dichloride tetrahydate,$[Fe(Im)_6]Cl_2\ 4H_2O$ (Jian *et al.*, 2004) is useful as the cationic species could exhibit a large variety of SC behaviors, depending on the non-coordinated counter anions and solvate molecules (Bousseksou *et al.*, 1996). The asymmetric unit of the title compound is depicted in Fig. 1. The $Fe^{II}$ atoms have a slightly distorted octahedral environment, being each coordinated by six monodentate imidazole ligands. The interatomic distances of the $FeN_6$ cores allow to conclude that the $Fe^{2+}$ cation is in a high-spin state. Indeed, the average Fe—N bond lenghs for the two $[Fe(Im)_6]^{2+}$ units A and B is equal to 2.200 (4) Å and 2.210 (4) Å, respectively. The ionic components in the crystal form a three dimensional framework *via* N—H···O hydrogen bonds (Fig. 2a) and the imidazole molecules form hydrogen bonded chains running in the channels of ionic framework (Fig. 2 b). The geometry of hydrogen bonds is listed in Table 1.

## Experimental

Colourless and transparent single crystals of the title compound were obtained as a principal product from the reaction of iron(III) sulfate pentahydrate (0.06 mmol/0.024 g) and imidazole (0.36 mmol/0.0245 g) in the presence of hexamethyldisilazane (0.06 mmol/0.0096 g), terephtalic acid (0.06 mmol/0.00996 g) and dimethylformamide (6 ml) as solvent. The reaction mixture was placed in a glass reactor, which was sealed and kept at 354 K for 24 h. Then, the mixture was cooled to room temperature with a cooling rate of 0.1 K/min and maintained in these conditions for 20 days. Suitable crystals for *X*– ray analysis were separated at the bottom of the flask. Elemental analysis calculated for ($C_{24}H_{32}N_{16}FeSO_4$): C 41.39%, H, 4.63%, N, 32.17%, found: C 41.50%; 4.60%, N 32.11%.

## Refinement

The H atoms were positioned geometrically and refined using a riding model approximation with C—H = 0.95 Å, N—H = 0.88 Å and with $U_{iso}$(H) = 1.2 *x* $U_{eq}$(C,*N*).

**Figures**

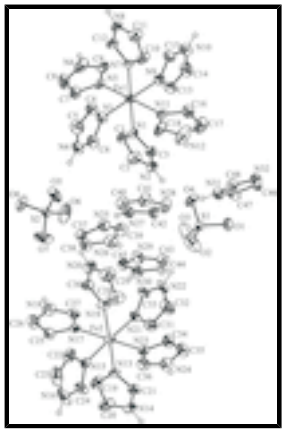


Fig. 1. The structure of the asymmetric unit for $[Fe(C_3H_4N_2)_6]SO_4.2C_3H_4N_2$ with displacement ellipsoids shown at the 50% probability level.

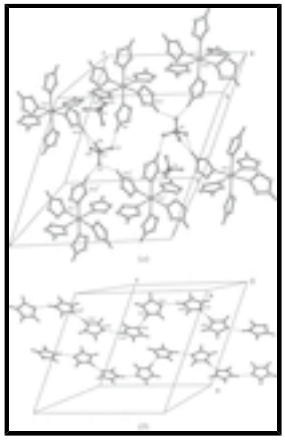


Fig. 2. Crystal packing diagrams showing three-dimensional framework formed by hydrogen-bonded cations and anions (*a*) and the [0 0 1] chain of hydrogen- bonded imidazole molecules (*b*). Only H atoms involved in hydrogen bonding are shown. Hydrogen bonds are shown with dashed lines. Symmetry code: (i)*1 - x, 1 - y, 1 - z*; (ii) *x-1, y, z*.

**Hexakis(1*H*-imidazole-κ*N*$^3$)iron(II) sulfate– 1*H*-imidazole (1/2)**

*Crystal data*

| | |
|---|---|
| $[Fe(C_3H_4N_2)_6]SO_4·2C_3H_4N_2$ | $Z = 4$ |
| $M_r = 696.57$ | $F(000) = 1448$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.406$ Mg m$^{-3}$ |
| $a = 15.4091$ (8) Å | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $b = 15.4436$ (7) Å | Cell parameters from 3265 reflections |
| $c = 15.9883$ (11) Å | $\theta = 2.9$–$29.1°$ |
| $\alpha = 69.813$ (5)° | $\mu = 0.58$ mm$^{-1}$ |
| $\beta = 69.949$ (5)° | $T = 200$ K |
| $\gamma = 73.214$ (4)° | Prism, colourless |
| $V = 3291.3$ (3) Å$^3$ | 0.20 × 0.15 × 0.15 mm |

*Data collection*

| | |
|---|---|
| Oxford Diffraction Xcalibur E diffractometer | 10955 independent reflections |

Radiation source: fine-focus sealed tube
graphite
Detector resolution: 16.1593 pixels $mm^{-1}$
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min}$ = 0.901, $T_{max}$ = 0.917
15987 measured reflections

5185 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.9°
$h$ = −14→18
$k$ = −18→18
$l$ = −19→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.107
$S$ = 0.85
10955 reflections
829 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0001P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.63 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.50 e Å$^{-3}$

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.8831 (4) | 0.5306 (3) | 0.1041 (4) | 0.0406 (14) |
| H1 | 0.8907 | 0.5862 | 0.0547 | 0.049* |
| C2 | 0.8911 (4) | 0.4450 (4) | 0.0941 (4) | 0.0564 (18) |
| H2 | 0.9063 | 0.4293 | 0.0376 | 0.068* |
| C3 | 0.8569 (3) | 0.4366 (3) | 0.2397 (4) | 0.0408 (14) |
| H3 | 0.8431 | 0.4115 | 0.3050 | 0.049* |
| C4 | 0.6680 (4) | 0.7430 (4) | 0.3389 (4) | 0.0506 (17) |
| H4 | 0.6936 | 0.7779 | 0.3601 | 0.061* |
| C5 | 0.5748 (4) | 0.7497 (4) | 0.3535 (4) | 0.0613 (19) |
| H5 | 0.5238 | 0.7872 | 0.3870 | 0.074* |

| | | | | |
|---|---|---|---|---|
| C6 | 0.6578 (4) | 0.6494 (4) | 0.2730 (4) | 0.0485 (15) |
| H6 | 0.6733 | 0.6043 | 0.2394 | 0.058* |
| C7 | 0.8287 (4) | 0.7768 (3) | 0.0738 (4) | 0.0514 (16) |
| H7 | 0.7629 | 0.7785 | 0.0979 | 0.062* |
| C8 | 0.8722 (5) | 0.8207 (4) | −0.0143 (5) | 0.069 (2) |
| H8 | 0.8433 | 0.8580 | −0.0624 | 0.083* |
| C9 | 0.9751 (4) | 0.7461 (3) | 0.0637 (4) | 0.0405 (14) |
| H9 | 1.0337 | 0.7222 | 0.0786 | 0.049* |
| C10 | 0.8820 (4) | 0.7156 (3) | 0.4120 (4) | 0.0486 (16) |
| H10 | 0.8740 | 0.6587 | 0.4596 | 0.058* |
| C11 | 0.8913 (4) | 0.7942 (4) | 0.4237 (4) | 0.0538 (17) |
| H11 | 0.8904 | 0.8035 | 0.4798 | 0.065* |
| C12 | 0.8984 (3) | 0.8172 (3) | 0.2799 (4) | 0.0420 (15) |
| H12 | 0.9041 | 0.8471 | 0.2160 | 0.050* |
| C13 | 1.0812 (4) | 0.5137 (4) | 0.1793 (4) | 0.0497 (17) |
| H13 | 1.0588 | 0.4815 | 0.1522 | 0.060* |
| C14 | 1.1718 (4) | 0.5038 (4) | 0.1747 (5) | 0.066 (2) |
| H14 | 1.2241 | 0.4649 | 0.1442 | 0.079* |
| C15 | 1.0857 (4) | 0.6036 (4) | 0.2509 (4) | 0.0482 (16) |
| H15 | 1.0678 | 0.6492 | 0.2843 | 0.058* |
| C16 | 0.9236 (4) | 0.4531 (4) | 0.4261 (4) | 0.0562 (17) |
| H16 | 0.9892 | 0.4450 | 0.3959 | 0.067* |
| C17 | 0.8826 (5) | 0.3985 (4) | 0.5096 (5) | 0.073 (2) |
| H17 | 0.9134 | 0.3443 | 0.5471 | 0.088* |
| C18 | 0.7769 (5) | 0.5072 (3) | 0.4562 (4) | 0.0522 (17) |
| H18 | 0.7165 | 0.5435 | 0.4516 | 0.063* |
| C19 | 0.3554 (4) | 0.0387 (4) | 0.0896 (4) | 0.0521 (17) |
| H19 | 0.3463 | 0.0980 | 0.0455 | 0.063* |
| C20 | 0.3532 (4) | −0.0454 (4) | 0.0809 (4) | 0.0610 (19) |
| H20 | 0.3413 | −0.0552 | 0.0307 | 0.073* |
| C21 | 0.3814 (3) | −0.0680 (4) | 0.2105 (4) | 0.0495 (17) |
| H21 | 0.3933 | −0.0991 | 0.2692 | 0.059* |
| C22 | 0.1618 (4) | 0.2330 (4) | 0.3056 (4) | 0.0572 (18) |
| H22 | 0.1804 | 0.2809 | 0.3164 | 0.069* |
| C23 | 0.0712 (4) | 0.2272 (5) | 0.3179 (5) | 0.079 (2) |
| H23 | 0.0154 | 0.2689 | 0.3394 | 0.094* |
| C24 | 0.1668 (4) | 0.1106 (4) | 0.2685 (4) | 0.0498 (16) |
| H24 | 0.1892 | 0.0549 | 0.2486 | 0.060* |
| C25 | 0.2971 (4) | 0.2983 (3) | 0.0741 (4) | 0.0421 (14) |
| H25 | 0.2336 | 0.2989 | 0.1105 | 0.050* |
| C26 | 0.3248 (4) | 0.3585 (4) | −0.0112 (4) | 0.0521 (16) |
| H26 | 0.2861 | 0.4083 | −0.0446 | 0.063* |
| C27 | 0.4462 (4) | 0.2595 (3) | 0.0288 (4) | 0.0431 (15) |
| H27 | 0.5095 | 0.2279 | 0.0259 | 0.052* |
| C28 | 0.3728 (4) | 0.2350 (4) | 0.3791 (5) | 0.067 (2) |
| H28 | 0.3601 | 0.1825 | 0.4311 | 0.081* |
| C29 | 0.3862 (5) | 0.3168 (4) | 0.3815 (5) | 0.076 (2) |
| H29 | 0.3849 | 0.3321 | 0.4346 | 0.092* |
| C30 | 0.3982 (4) | 0.3240 (3) | 0.2411 (4) | 0.0489 (17) |

| | | | | |
|---|---|---|---|---|
| H30 | 0.4074 | 0.3474 | 0.1760 | 0.059* |
| C31 | 0.5881 (4) | 0.0190 (4) | 0.1647 (4) | 0.0497 (16) |
| H31 | 0.5665 | −0.0214 | 0.1461 | 0.060* |
| C32 | 0.6772 (4) | 0.0084 (4) | 0.1642 (5) | 0.073 (2) |
| H32 | 0.7297 | −0.0375 | 0.1445 | 0.088* |
| C33 | 0.5884 (4) | 0.1284 (4) | 0.2165 (4) | 0.0484 (16) |
| H33 | 0.5696 | 0.1808 | 0.2413 | 0.058* |
| C34 | 0.4485 (4) | −0.0236 (4) | 0.4049 (4) | 0.0511 (16) |
| H34 | 0.5128 | −0.0252 | 0.3715 | 0.061* |
| C35 | 0.4172 (4) | −0.0696 (4) | 0.4943 (4) | 0.0550 (17) |
| H35 | 0.4547 | −0.1095 | 0.5345 | 0.066* |
| C36 | 0.2995 (4) | 0.0082 (4) | 0.4388 (4) | 0.0494 (16) |
| H36 | 0.2366 | 0.0327 | 0.4346 | 0.059* |
| C37 | 0.5915 (4) | 0.4256 (3) | 0.0643 (4) | 0.0439 (15) |
| H37 | 0.5389 | 0.4761 | 0.0614 | 0.053* |
| C38 | 0.6337 (4) | 0.3757 (4) | 0.0021 (4) | 0.0449 (15) |
| H38 | 0.6162 | 0.3853 | −0.0524 | 0.054* |
| C39 | 0.7043 (4) | 0.3218 (4) | 0.1094 (4) | 0.0529 (16) |
| H39 | 0.7474 | 0.2842 | 0.1446 | 0.063* |
| C40 | 0.5647 (4) | 0.4377 (4) | 0.3840 (4) | 0.0502 (16) |
| H40 | 0.5211 | 0.4956 | 0.3745 | 0.060* |
| C41 | 0.5914 (4) | 0.3905 (4) | 0.4620 (4) | 0.0500 (16) |
| H41 | 0.5674 | 0.4089 | 0.5178 | 0.060* |
| C42 | 0.6655 (4) | 0.3135 (4) | 0.3643 (4) | 0.0457 (15) |
| H42 | 0.7057 | 0.2660 | 0.3359 | 0.055* |
| C43 | 0.8919 (4) | 0.0898 (3) | −0.0391 (4) | 0.0453 (15) |
| H43 | 0.9103 | 0.0628 | 0.0168 | 0.054* |
| C44 | 0.9266 (4) | 0.0552 (3) | −0.1126 (4) | 0.0410 (14) |
| H44 | 0.9729 | −0.0001 | −0.1180 | 0.049* |
| C45 | 0.8238 (4) | 0.1831 (4) | −0.1424 (4) | 0.0462 (16) |
| H45 | 0.7855 | 0.2346 | −0.1747 | 0.055* |
| C46 | 0.9204 (4) | 0.0970 (3) | 0.5650 (4) | 0.0405 (14) |
| H46 | 0.9791 | 0.0543 | 0.5581 | 0.049* |
| C47 | 0.8726 (4) | 0.1345 (4) | 0.5007 (4) | 0.0469 (15) |
| H47 | 0.8898 | 0.1239 | 0.4415 | 0.056* |
| C48 | 0.7976 (4) | 0.1849 (3) | 0.6218 (4) | 0.0427 (15) |
| H48 | 0.7503 | 0.2179 | 0.6624 | 0.051* |
| N1 | 0.8625 (3) | 0.5252 (2) | 0.1959 (3) | 0.0332 (11) |
| N2 | 0.8731 (3) | 0.3863 (3) | 0.1803 (3) | 0.0449 (13) |
| H49 | 0.8722 | 0.3261 | 0.1950 | 0.054* |
| N3 | 0.7205 (3) | 0.6795 (3) | 0.2899 (3) | 0.0378 (11) |
| N4 | 0.5702 (3) | 0.6900 (3) | 0.3088 (3) | 0.0463 (12) |
| H50 | 0.5185 | 0.6803 | 0.3047 | 0.056* |
| N5 | 0.8931 (3) | 0.7305 (2) | 0.1219 (3) | 0.0352 (11) |
| N6 | 0.9645 (4) | 0.8004 (3) | −0.0195 (3) | 0.0593 (15) |
| H51 | 1.0099 | 0.8194 | −0.0687 | 0.071* |
| N7 | 0.8855 (3) | 0.7292 (2) | 0.3222 (3) | 0.0322 (11) |
| N8 | 0.9024 (3) | 0.8574 (3) | 0.3397 (3) | 0.0417 (12) |
| H52 | 0.9107 | 0.9151 | 0.3267 | 0.050* |

| | | | | |
|---|---|---|---|---|
| N9 | 1.0254 (3) | 0.5760 (3) | 0.2280 (3) | 0.0394 (11) |
| N10 | 1.1739 (3) | 0.5608 (3) | 0.2223 (4) | 0.0571 (14) |
| H53 | 1.2240 | 0.5678 | 0.2321 | 0.068* |
| N11 | 0.8552 (3) | 0.5217 (3) | 0.3926 (3) | 0.0360 (11) |
| N12 | 0.7920 (4) | 0.4350 (3) | 0.5287 (4) | 0.0783 (19) |
| H54 | 0.7491 | 0.4153 | 0.5800 | 0.094* |
| N13 | 0.3731 (3) | 0.0232 (3) | 0.1728 (3) | 0.0383 (12) |
| N14 | 0.3711 (3) | −0.1118 (3) | 0.1568 (3) | 0.0463 (13) |
| H55 | 0.3753 | −0.1727 | 0.1687 | 0.056* |
| N15 | 0.2210 (3) | 0.1589 (3) | 0.2754 (3) | 0.0389 (11) |
| N16 | 0.0771 (4) | 0.1505 (4) | 0.2931 (4) | 0.0773 (18) |
| H56 | 0.0293 | 0.1301 | 0.2933 | 0.093* |
| N17 | 0.3736 (3) | 0.2371 (3) | 0.0996 (3) | 0.0355 (11) |
| N18 | 0.4197 (3) | 0.3317 (3) | −0.0379 (3) | 0.0476 (12) |
| H57 | 0.4574 | 0.3577 | −0.0906 | 0.057* |
| N19 | 0.3804 (3) | 0.2397 (3) | 0.2897 (3) | 0.0443 (13) |
| N20 | 0.4017 (4) | 0.3723 (3) | 0.2939 (4) | 0.0691 (18) |
| H58 | 0.4122 | 0.4299 | 0.2751 | 0.083* |
| N21 | 0.5318 (3) | 0.0937 (3) | 0.1948 (3) | 0.0361 (11) |
| N22 | 0.6758 (3) | 0.0780 (3) | 0.1980 (4) | 0.0661 (16) |
| H59 | 0.7249 | 0.0886 | 0.2065 | 0.079* |
| N23 | 0.3744 (3) | 0.0256 (3) | 0.3694 (3) | 0.0376 (11) |
| N24 | 0.3226 (3) | −0.0481 (3) | 0.5157 (3) | 0.0526 (13) |
| H60 | 0.2835 | −0.0675 | 0.5701 | 0.063* |
| N25 | 0.6377 (3) | 0.3910 (3) | 0.1336 (3) | 0.0469 (13) |
| N26 | 0.7052 (3) | 0.3096 (3) | 0.0299 (3) | 0.0536 (14) |
| H61 | 0.7441 | 0.2676 | 0.0018 | 0.064* |
| N27 | 0.6113 (3) | 0.3877 (3) | 0.3215 (3) | 0.0506 (13) |
| H62 | 0.6067 | 0.4013 | 0.2648 | 0.061* |
| N28 | 0.6587 (3) | 0.3115 (3) | 0.4493 (3) | 0.0561 (14) |
| N29 | 0.8255 (3) | 0.1704 (3) | −0.0570 (3) | 0.0486 (13) |
| N30 | 0.8833 (3) | 0.1136 (3) | −0.1778 (3) | 0.0452 (12) |
| H63 | 0.8922 | 0.1074 | −0.2330 | 0.054* |
| N31 | 0.7945 (3) | 0.1907 (3) | 0.5383 (3) | 0.0460 (12) |
| H64 | 0.7496 | 0.2252 | 0.5119 | 0.055* |
| N32 | 0.8733 (3) | 0.1284 (3) | 0.6417 (3) | 0.0452 (12) |
| O1 | 0.9397 (2) | 0.0386 (2) | 0.2964 (3) | 0.0546 (11) |
| O2 | 0.7783 (3) | 0.1092 (3) | 0.3067 (3) | 0.0821 (14) |
| O3 | 0.8813 (3) | 0.1121 (4) | 0.1626 (3) | 0.1085 (18) |
| O4 | 0.8938 (3) | 0.1995 (2) | 0.2527 (4) | 0.113 (2) |
| O5 | 0.4011 (3) | 0.6542 (2) | 0.3180 (3) | 0.0648 (13) |
| O6 | 0.4728 (3) | 0.5586 (2) | 0.2131 (3) | 0.0780 (14) |
| O7 | 0.3098 (3) | 0.5833 (3) | 0.2806 (3) | 0.0946 (17) |
| O8 | 0.3793 (3) | 0.7069 (2) | 0.1671 (3) | 0.0575 (12) |
| S1 | 0.87346 (9) | 0.11509 (8) | 0.25340 (9) | 0.0254 (3) |
| S2 | 0.39014 (9) | 0.62584 (8) | 0.24501 (9) | 0.0286 (3) |
| Fe1 | 0.87273 (5) | 0.62872 (4) | 0.25814 (5) | 0.03015 (19) |
| Fe2 | 0.37628 (5) | 0.12942 (5) | 0.23314 (6) | 0.0351 (2) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.057 (4) | 0.037 (3) | 0.027 (3) | −0.016 (3) | −0.011 (3) | −0.003 (3) |
| C2 | 0.079 (5) | 0.054 (4) | 0.040 (4) | −0.018 (3) | −0.013 (4) | −0.016 (3) |
| C3 | 0.051 (4) | 0.040 (3) | 0.036 (4) | −0.015 (3) | −0.014 (3) | −0.009 (3) |
| C4 | 0.042 (4) | 0.048 (4) | 0.064 (5) | −0.009 (3) | −0.017 (4) | −0.015 (3) |
| C5 | 0.050 (4) | 0.062 (4) | 0.075 (6) | 0.003 (3) | −0.014 (4) | −0.036 (4) |
| C6 | 0.049 (4) | 0.061 (4) | 0.034 (4) | −0.009 (3) | −0.012 (3) | −0.012 (3) |
| C7 | 0.056 (4) | 0.052 (4) | 0.037 (4) | −0.020 (3) | −0.019 (4) | 0.012 (3) |
| C8 | 0.074 (5) | 0.077 (5) | 0.051 (5) | −0.015 (4) | −0.038 (4) | 0.012 (4) |
| C9 | 0.046 (4) | 0.033 (3) | 0.033 (4) | −0.013 (3) | −0.001 (3) | −0.004 (3) |
| C10 | 0.087 (5) | 0.034 (3) | 0.030 (4) | −0.023 (3) | −0.014 (3) | −0.008 (3) |
| C11 | 0.078 (5) | 0.049 (4) | 0.039 (4) | −0.016 (3) | −0.010 (4) | −0.021 (3) |
| C12 | 0.057 (4) | 0.032 (3) | 0.043 (4) | −0.017 (3) | −0.015 (3) | −0.009 (3) |
| C13 | 0.036 (4) | 0.053 (4) | 0.071 (5) | 0.000 (3) | −0.015 (3) | −0.037 (4) |
| C14 | 0.050 (4) | 0.061 (4) | 0.084 (6) | 0.009 (4) | −0.023 (4) | −0.028 (4) |
| C15 | 0.040 (4) | 0.055 (4) | 0.059 (5) | −0.008 (3) | −0.020 (3) | −0.020 (3) |
| C16 | 0.055 (4) | 0.062 (4) | 0.049 (5) | −0.019 (4) | −0.019 (4) | −0.001 (3) |
| C17 | 0.103 (6) | 0.057 (4) | 0.048 (5) | −0.013 (5) | −0.023 (5) | 0.002 (4) |
| C18 | 0.079 (5) | 0.034 (3) | 0.029 (4) | −0.009 (3) | −0.007 (4) | 0.000 (3) |
| C19 | 0.091 (5) | 0.039 (3) | 0.027 (4) | −0.021 (3) | −0.018 (4) | 0.000 (3) |
| C20 | 0.101 (6) | 0.050 (4) | 0.042 (4) | −0.032 (4) | −0.009 (4) | −0.020 (3) |
| C21 | 0.049 (4) | 0.043 (3) | 0.071 (5) | −0.006 (3) | −0.021 (4) | −0.030 (4) |
| C22 | 0.045 (4) | 0.043 (4) | 0.067 (5) | 0.000 (3) | −0.005 (4) | −0.013 (3) |
| C23 | 0.029 (4) | 0.096 (6) | 0.066 (6) | 0.018 (4) | 0.001 (4) | −0.007 (5) |
| C24 | 0.041 (4) | 0.084 (4) | 0.029 (4) | −0.032 (4) | −0.008 (3) | −0.005 (3) |
| C25 | 0.035 (3) | 0.053 (3) | 0.028 (3) | −0.018 (3) | −0.004 (3) | 0.004 (3) |
| C26 | 0.033 (4) | 0.064 (4) | 0.045 (4) | −0.007 (3) | −0.003 (3) | −0.007 (3) |
| C27 | 0.029 (3) | 0.048 (4) | 0.059 (5) | −0.010 (3) | −0.007 (3) | −0.025 (3) |
| C28 | 0.084 (5) | 0.078 (5) | 0.048 (5) | −0.039 (4) | 0.003 (4) | −0.028 (4) |
| C29 | 0.115 (7) | 0.067 (5) | 0.072 (6) | −0.020 (4) | −0.036 (5) | −0.037 (4) |
| C30 | 0.058 (4) | 0.040 (3) | 0.063 (5) | −0.013 (3) | −0.023 (4) | −0.022 (3) |
| C31 | 0.035 (4) | 0.060 (4) | 0.055 (5) | −0.011 (3) | −0.003 (3) | −0.025 (3) |
| C32 | 0.038 (4) | 0.091 (5) | 0.092 (7) | −0.004 (4) | −0.017 (4) | −0.034 (5) |
| C33 | 0.045 (4) | 0.061 (4) | 0.052 (4) | −0.016 (3) | −0.027 (3) | −0.012 (3) |
| C34 | 0.045 (4) | 0.061 (4) | 0.041 (4) | −0.011 (3) | −0.012 (3) | −0.005 (3) |
| C35 | 0.041 (4) | 0.069 (4) | 0.048 (5) | −0.006 (3) | −0.014 (4) | −0.011 (4) |
| C36 | 0.047 (4) | 0.063 (4) | 0.031 (4) | −0.026 (3) | −0.003 (3) | 0.001 (3) |
| C37 | 0.052 (4) | 0.041 (3) | 0.044 (4) | −0.010 (3) | −0.019 (3) | −0.010 (3) |
| C38 | 0.051 (4) | 0.065 (4) | 0.021 (3) | −0.019 (3) | −0.012 (3) | −0.007 (3) |
| C39 | 0.073 (5) | 0.063 (4) | 0.024 (4) | −0.020 (4) | −0.013 (3) | −0.008 (3) |
| C40 | 0.062 (4) | 0.050 (4) | 0.040 (4) | −0.010 (3) | −0.012 (4) | −0.016 (3) |
| C41 | 0.063 (4) | 0.046 (4) | 0.032 (4) | −0.006 (3) | −0.006 (3) | −0.010 (3) |
| C42 | 0.045 (4) | 0.038 (3) | 0.052 (5) | 0.005 (3) | −0.023 (3) | −0.012 (3) |
| C43 | 0.066 (4) | 0.049 (4) | 0.029 (4) | −0.018 (3) | −0.017 (3) | −0.011 (3) |
| C44 | 0.044 (4) | 0.030 (3) | 0.042 (4) | 0.001 (3) | −0.014 (3) | −0.004 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C45 | 0.046 (4) | 0.037 (3) | 0.059 (5) | 0.004 (3) | −0.023 (3) | −0.018 (3) |
| C46 | 0.029 (3) | 0.047 (3) | 0.047 (4) | 0.003 (3) | −0.013 (3) | −0.021 (3) |
| C47 | 0.045 (4) | 0.081 (4) | 0.023 (4) | −0.016 (3) | −0.005 (3) | −0.025 (3) |
| C48 | 0.047 (4) | 0.039 (3) | 0.038 (4) | −0.001 (3) | −0.010 (3) | −0.014 (3) |
| N1 | 0.038 (3) | 0.025 (2) | 0.036 (3) | −0.007 (2) | −0.014 (2) | −0.003 (2) |
| N2 | 0.057 (3) | 0.027 (2) | 0.058 (4) | −0.005 (2) | −0.023 (3) | −0.016 (3) |
| N3 | 0.040 (3) | 0.046 (3) | 0.025 (3) | −0.014 (2) | −0.006 (2) | −0.005 (2) |
| N4 | 0.035 (3) | 0.063 (3) | 0.042 (3) | −0.013 (3) | −0.014 (3) | −0.009 (3) |
| N5 | 0.047 (3) | 0.024 (2) | 0.031 (3) | −0.010 (2) | −0.010 (2) | 0.000 (2) |
| N6 | 0.088 (4) | 0.045 (3) | 0.031 (3) | −0.022 (3) | −0.003 (3) | 0.001 (3) |
| N7 | 0.036 (3) | 0.031 (2) | 0.034 (3) | −0.015 (2) | −0.007 (2) | −0.009 (2) |
| N8 | 0.060 (3) | 0.024 (2) | 0.047 (3) | −0.011 (2) | −0.017 (3) | −0.012 (2) |
| N9 | 0.040 (3) | 0.043 (3) | 0.034 (3) | −0.015 (2) | −0.009 (2) | −0.003 (2) |
| N10 | 0.045 (3) | 0.060 (3) | 0.070 (4) | −0.013 (3) | −0.020 (3) | −0.017 (3) |
| N11 | 0.053 (3) | 0.029 (2) | 0.031 (3) | −0.017 (2) | −0.011 (3) | −0.006 (2) |
| N12 | 0.119 (5) | 0.054 (3) | 0.022 (3) | −0.010 (4) | 0.017 (4) | −0.004 (3) |
| N13 | 0.040 (3) | 0.029 (2) | 0.050 (3) | −0.013 (2) | −0.013 (3) | −0.010 (2) |
| N14 | 0.057 (3) | 0.031 (3) | 0.055 (4) | −0.015 (2) | −0.010 (3) | −0.018 (3) |
| N15 | 0.035 (3) | 0.033 (2) | 0.040 (3) | −0.015 (2) | −0.012 (2) | 0.010 (2) |
| N16 | 0.037 (4) | 0.137 (5) | 0.052 (4) | −0.022 (4) | −0.020 (3) | −0.007 (4) |
| N17 | 0.036 (3) | 0.034 (2) | 0.038 (3) | −0.015 (2) | −0.010 (2) | −0.005 (2) |
| N18 | 0.060 (4) | 0.057 (3) | 0.024 (3) | −0.023 (3) | −0.004 (3) | −0.007 (2) |
| N19 | 0.048 (3) | 0.044 (3) | 0.048 (4) | −0.022 (2) | −0.002 (3) | −0.021 (3) |
| N20 | 0.099 (5) | 0.033 (3) | 0.097 (5) | 0.001 (3) | −0.055 (4) | −0.028 (3) |
| N21 | 0.028 (3) | 0.052 (3) | 0.029 (3) | −0.014 (2) | −0.006 (2) | −0.009 (2) |
| N22 | 0.046 (4) | 0.073 (4) | 0.088 (5) | −0.016 (3) | −0.044 (3) | −0.003 (3) |
| N23 | 0.042 (3) | 0.037 (3) | 0.035 (3) | −0.011 (2) | −0.010 (2) | −0.010 (2) |
| N24 | 0.048 (3) | 0.063 (3) | 0.040 (4) | −0.012 (3) | −0.006 (3) | −0.012 (3) |
| N25 | 0.052 (3) | 0.048 (3) | 0.049 (4) | −0.002 (3) | −0.017 (3) | −0.026 (3) |
| N26 | 0.049 (3) | 0.066 (3) | 0.048 (4) | −0.006 (3) | −0.005 (3) | −0.031 (3) |
| N27 | 0.054 (3) | 0.061 (3) | 0.046 (4) | −0.009 (3) | −0.020 (3) | −0.021 (3) |
| N28 | 0.067 (4) | 0.046 (3) | 0.041 (4) | 0.004 (3) | −0.016 (3) | −0.006 (3) |
| N29 | 0.044 (3) | 0.058 (3) | 0.049 (4) | 0.002 (3) | −0.017 (3) | −0.027 (3) |
| N30 | 0.047 (3) | 0.047 (3) | 0.049 (4) | −0.011 (2) | −0.016 (3) | −0.017 (3) |
| N31 | 0.055 (3) | 0.051 (3) | 0.036 (3) | −0.016 (3) | −0.019 (3) | −0.006 (2) |
| N32 | 0.055 (3) | 0.044 (3) | 0.039 (3) | −0.008 (3) | −0.018 (3) | −0.010 (2) |
| O1 | 0.064 (3) | 0.038 (2) | 0.070 (3) | 0.0048 (19) | −0.043 (3) | −0.012 (2) |
| O2 | 0.040 (3) | 0.154 (4) | 0.042 (3) | −0.025 (3) | 0.001 (2) | −0.022 (3) |
| O3 | 0.079 (4) | 0.205 (5) | 0.046 (3) | 0.028 (3) | −0.031 (3) | −0.071 (4) |
| O4 | 0.103 (4) | 0.019 (2) | 0.229 (7) | −0.003 (2) | −0.091 (4) | −0.009 (3) |
| O5 | 0.063 (3) | 0.089 (3) | 0.070 (3) | 0.007 (2) | −0.042 (3) | −0.049 (3) |
| O6 | 0.079 (3) | 0.059 (2) | 0.051 (3) | 0.025 (2) | 0.002 (3) | −0.013 (2) |
| O7 | 0.092 (4) | 0.138 (4) | 0.071 (4) | −0.092 (3) | −0.017 (3) | 0.003 (3) |
| O8 | 0.092 (3) | 0.033 (2) | 0.038 (3) | 0.001 (2) | −0.022 (3) | −0.0052 (19) |
| S1 | 0.0245 (7) | 0.0285 (7) | 0.0213 (7) | −0.0020 (6) | −0.0054 (6) | −0.0080 (6) |
| S2 | 0.0324 (8) | 0.0315 (7) | 0.0235 (8) | −0.0044 (6) | −0.0084 (6) | −0.0099 (6) |
| Fe1 | 0.0341 (5) | 0.0300 (4) | 0.0259 (5) | −0.0140 (3) | −0.0048 (4) | −0.0040 (3) |
| Fe2 | 0.0347 (5) | 0.0354 (4) | 0.0384 (5) | −0.0168 (4) | −0.0051 (4) | −0.0107 (4) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.351 (6) | C31—H31 | 0.9500 |
| C1—N1 | 1.369 (6) | C32—N22 | 1.353 (6) |
| C1—H1 | 0.9500 | C32—H32 | 0.9500 |
| C2—N2 | 1.349 (6) | C33—N21 | 1.332 (5) |
| C2—H2 | 0.9500 | C33—N22 | 1.337 (6) |
| C3—N1 | 1.318 (5) | C33—H33 | 0.9500 |
| C3—N2 | 1.342 (6) | C34—C35 | 1.342 (7) |
| C3—H3 | 0.9500 | C34—N23 | 1.360 (6) |
| C4—C5 | 1.353 (7) | C34—H34 | 0.9500 |
| C4—N3 | 1.366 (6) | C35—N24 | 1.345 (6) |
| C4—H4 | 0.9500 | C35—H35 | 0.9500 |
| C5—N4 | 1.375 (6) | C36—N23 | 1.315 (6) |
| C5—H5 | 0.9500 | C36—N24 | 1.334 (7) |
| C6—N3 | 1.323 (6) | C36—H36 | 0.9500 |
| C6—N4 | 1.330 (6) | C37—C38 | 1.342 (6) |
| C6—H6 | 0.9500 | C37—N25 | 1.391 (6) |
| C7—N5 | 1.349 (6) | C37—H37 | 0.9500 |
| C7—C8 | 1.360 (8) | C38—N26 | 1.352 (6) |
| C7—H7 | 0.9500 | C38—H38 | 0.9500 |
| C8—N6 | 1.344 (7) | C39—N25 | 1.313 (6) |
| C8—H8 | 0.9500 | C39—N26 | 1.343 (6) |
| C9—N5 | 1.318 (6) | C39—H39 | 0.9500 |
| C9—N6 | 1.341 (6) | C40—C41 | 1.344 (7) |
| C9—H9 | 0.9500 | C40—N27 | 1.352 (6) |
| C10—C11 | 1.345 (6) | C40—H40 | 0.9500 |
| C10—N7 | 1.362 (6) | C41—N28 | 1.379 (6) |
| C10—H10 | 0.9500 | C41—H41 | 0.9500 |
| C11—N8 | 1.349 (6) | C42—N28 | 1.316 (7) |
| C11—H11 | 0.9500 | C42—N27 | 1.334 (6) |
| C12—N7 | 1.331 (5) | C42—H42 | 0.9500 |
| C12—N8 | 1.333 (6) | C43—C44 | 1.338 (6) |
| C12—H12 | 0.9500 | C43—N29 | 1.379 (6) |
| C13—C14 | 1.339 (7) | C43—H43 | 0.9500 |
| C13—N9 | 1.361 (6) | C44—N30 | 1.352 (6) |
| C13—H13 | 0.9500 | C44—H44 | 0.9500 |
| C14—N10 | 1.361 (6) | C45—N29 | 1.319 (7) |
| C14—H14 | 0.9500 | C45—N30 | 1.344 (6) |
| C15—N9 | 1.324 (5) | C45—H45 | 0.9500 |
| C15—N10 | 1.325 (6) | C46—C47 | 1.341 (7) |
| C15—H15 | 0.9500 | C46—N32 | 1.365 (6) |
| C16—C17 | 1.359 (8) | C46—H46 | 0.9500 |
| C16—N11 | 1.368 (6) | C47—N31 | 1.350 (6) |
| C16—H16 | 0.9500 | C47—H47 | 0.9500 |
| C17—N12 | 1.317 (7) | C48—N32 | 1.303 (6) |
| C17—H17 | 0.9500 | C48—N31 | 1.323 (6) |
| C18—N11 | 1.302 (6) | C48—H48 | 0.9500 |

| | | | |
|---|---|---|---|
| C18—N12 | 1.336 (7) | N1—Fe1 | 2.218 (4) |
| C18—H18 | 0.9500 | N2—H49 | 0.8800 |
| C19—C20 | 1.363 (6) | N3—Fe1 | 2.185 (4) |
| C19—N13 | 1.377 (6) | N4—H50 | 0.8800 |
| C19—H19 | 0.9500 | N5—Fe1 | 2.184 (4) |
| C20—N14 | 1.344 (7) | N6—H51 | 0.8800 |
| C20—H20 | 0.9500 | N7—Fe1 | 2.215 (3) |
| C21—N13 | 1.314 (6) | N8—H52 | 0.8800 |
| C21—N14 | 1.332 (6) | N9—Fe1 | 2.195 (4) |
| C21—H21 | 0.9500 | N10—H53 | 0.8800 |
| C22—N15 | 1.361 (6) | N11—Fe1 | 2.199 (4) |
| C22—C23 | 1.367 (7) | N12—H54 | 0.8800 |
| C22—H22 | 0.9500 | N13—Fe2 | 2.189 (4) |
| C23—N16 | 1.344 (7) | N14—H55 | 0.8800 |
| C23—H23 | 0.9500 | N15—Fe2 | 2.205 (4) |
| C24—N15 | 1.321 (5) | N16—H56 | 0.8800 |
| C24—N16 | 1.322 (6) | N17—Fe2 | 2.213 (4) |
| C24—H24 | 0.9500 | N18—H57 | 0.8800 |
| C25—C26 | 1.364 (7) | N19—Fe2 | 2.211 (4) |
| C25—N17 | 1.368 (6) | N20—H58 | 0.8800 |
| C25—H25 | 0.9500 | N21—Fe2 | 2.210 (4) |
| C26—N18 | 1.355 (6) | N22—H59 | 0.8800 |
| C26—H26 | 0.9500 | N23—Fe2 | 2.211 (4) |
| C27—N17 | 1.318 (6) | N24—H60 | 0.8800 |
| C27—N18 | 1.328 (6) | N26—H61 | 0.8800 |
| C27—H27 | 0.9500 | N27—H62 | 0.8800 |
| C28—C29 | 1.353 (6) | N30—H63 | 0.8800 |
| C28—N19 | 1.370 (7) | N31—H64 | 0.8800 |
| C28—H28 | 0.9500 | O1—S1 | 1.460 (3) |
| C29—N20 | 1.347 (7) | O2—S1 | 1.433 (4) |
| C29—H29 | 0.9500 | O3—S1 | 1.431 (4) |
| C30—N19 | 1.318 (6) | O4—S1 | 1.422 (3) |
| C30—N20 | 1.328 (6) | O5—S2 | 1.454 (3) |
| C30—H30 | 0.9500 | O6—S2 | 1.454 (4) |
| C31—C32 | 1.332 (7) | O7—S2 | 1.421 (3) |
| C31—N21 | 1.355 (5) | O8—S2 | 1.446 (4) |
| C2—C1—N1 | 110.0 (5) | N32—C46—H46 | 124.6 |
| C2—C1—H1 | 125.0 | C46—C47—N31 | 105.0 (5) |
| N1—C1—H1 | 125.0 | C46—C47—H47 | 127.5 |
| N2—C2—C1 | 106.3 (5) | N31—C47—H47 | 127.5 |
| N2—C2—H2 | 126.9 | N32—C48—N31 | 111.9 (5) |
| C1—C2—H2 | 126.9 | N32—C48—H48 | 124.1 |
| N1—C3—N2 | 111.4 (5) | N31—C48—H48 | 124.1 |
| N1—C3—H3 | 124.3 | C3—N1—C1 | 104.8 (4) |
| N2—C3—H3 | 124.3 | C3—N1—Fe1 | 125.1 (4) |
| C5—C4—N3 | 111.3 (5) | C1—N1—Fe1 | 128.3 (3) |
| C5—C4—H4 | 124.4 | C3—N2—C2 | 107.4 (4) |
| N3—C4—H4 | 124.4 | C3—N2—H49 | 126.3 |
| C4—C5—N4 | 104.5 (5) | C2—N2—H49 | 126.3 |

| | | | |
|---|---|---|---|
| C4—C5—H5 | 127.7 | C6—N3—C4 | 104.6 (4) |
| N4—C5—H5 | 127.7 | C6—N3—Fe1 | 127.4 (4) |
| N3—C6—N4 | 111.6 (5) | C4—N3—Fe1 | 127.8 (3) |
| N3—C6—H6 | 124.2 | C6—N4—C5 | 108.1 (5) |
| N4—C6—H6 | 124.2 | C6—N4—H50 | 126.0 |
| N5—C7—C8 | 109.8 (5) | C5—N4—H50 | 126.0 |
| N5—C7—H7 | 125.1 | C9—N5—C7 | 105.8 (5) |
| C8—C7—H7 | 125.1 | C9—N5—Fe1 | 125.4 (4) |
| N6—C8—C7 | 106.0 (6) | C7—N5—Fe1 | 127.8 (4) |
| N6—C8—H8 | 127.0 | C9—N6—C8 | 107.7 (5) |
| C7—C8—H8 | 127.0 | C9—N6—H51 | 126.1 |
| N5—C9—N6 | 110.7 (5) | C8—N6—H51 | 126.1 |
| N5—C9—H9 | 124.7 | C12—N7—C10 | 105.0 (4) |
| N6—C9—H9 | 124.7 | C12—N7—Fe1 | 126.7 (4) |
| C11—C10—N7 | 110.3 (5) | C10—N7—Fe1 | 128.3 (3) |
| C11—C10—H10 | 124.9 | C12—N8—C11 | 108.2 (4) |
| N7—C10—H10 | 124.9 | C12—N8—H52 | 125.9 |
| C10—C11—N8 | 106.1 (5) | C11—N8—H52 | 125.9 |
| C10—C11—H11 | 127.0 | C15—N9—C13 | 103.7 (4) |
| N8—C11—H11 | 127.0 | C15—N9—Fe1 | 125.9 (4) |
| N7—C12—N8 | 110.6 (5) | C13—N9—Fe1 | 130.4 (3) |
| N7—C12—H12 | 124.7 | C15—N10—C14 | 106.4 (5) |
| N8—C12—H12 | 124.7 | C15—N10—H53 | 126.8 |
| C14—C13—N9 | 110.8 (5) | C14—N10—H53 | 126.8 |
| C14—C13—H13 | 124.6 | C18—N11—C16 | 104.8 (5) |
| N9—C13—H13 | 124.6 | C18—N11—Fe1 | 127.6 (4) |
| C13—C14—N10 | 106.3 (5) | C16—N11—Fe1 | 127.5 (4) |
| C13—C14—H14 | 126.9 | C17—N12—C18 | 107.7 (6) |
| N10—C14—H14 | 126.8 | C17—N12—H54 | 126.1 |
| N9—C15—N10 | 112.7 (5) | C18—N12—H54 | 126.1 |
| N9—C15—H15 | 123.7 | C21—N13—C19 | 105.0 (4) |
| N10—C15—H15 | 123.7 | C21—N13—Fe2 | 127.7 (4) |
| C17—C16—N11 | 108.9 (6) | C19—N13—Fe2 | 127.1 (3) |
| C17—C16—H16 | 125.6 | C21—N14—C20 | 107.0 (4) |
| N11—C16—H16 | 125.6 | C21—N14—H55 | 126.5 |
| N12—C17—C16 | 106.8 (6) | C20—N14—H55 | 126.5 |
| N12—C17—H17 | 126.6 | C24—N15—C22 | 106.0 (4) |
| C16—C17—H17 | 126.6 | C24—N15—Fe2 | 125.6 (4) |
| N11—C18—N12 | 111.7 (6) | C22—N15—Fe2 | 128.2 (3) |
| N11—C18—H18 | 124.2 | C24—N16—C23 | 108.7 (5) |
| N12—C18—H18 | 124.2 | C24—N16—H56 | 125.7 |
| C20—C19—N13 | 108.6 (5) | C23—N16—H56 | 125.7 |
| C20—C19—H19 | 125.7 | C27—N17—C25 | 104.8 (5) |
| N13—C19—H19 | 125.7 | C27—N17—Fe2 | 127.4 (4) |
| N14—C20—C19 | 107.1 (5) | C25—N17—Fe2 | 127.8 (4) |
| N14—C20—H20 | 126.5 | C27—N18—C26 | 108.5 (5) |
| C19—C20—H20 | 126.5 | C27—N18—H57 | 125.8 |
| N13—C21—N14 | 112.2 (5) | C26—N18—H57 | 125.8 |
| N13—C21—H21 | 123.9 | C30—N19—C28 | 104.8 (4) |

| | | | |
|---|---|---|---|
| N14—C21—H21 | 123.9 | C30—N19—Fe2 | 125.8 (4) |
| N15—C22—C23 | 108.8 (5) | C28—N19—Fe2 | 129.2 (4) |
| N15—C22—H22 | 125.6 | C30—N20—C29 | 107.3 (5) |
| C23—C22—H22 | 125.6 | C30—N20—H58 | 126.3 |
| N16—C23—C22 | 105.9 (6) | C29—N20—H58 | 126.3 |
| N16—C23—H23 | 127.0 | C33—N21—C31 | 104.5 (4) |
| C22—C23—H23 | 127.0 | C33—N21—Fe2 | 126.1 (4) |
| N15—C24—N16 | 110.7 (5) | C31—N21—Fe2 | 127.8 (3) |
| N15—C24—H24 | 124.7 | C33—N22—C32 | 109.6 (5) |
| N16—C24—H24 | 124.7 | C33—N22—H59 | 125.2 |
| C26—C25—N17 | 110.3 (5) | C32—N22—H59 | 125.2 |
| C26—C25—H25 | 124.9 | C36—N23—C34 | 104.6 (5) |
| N17—C25—H25 | 124.9 | C36—N23—Fe2 | 126.5 (4) |
| N18—C26—C25 | 104.9 (5) | C34—N23—Fe2 | 128.3 (4) |
| N18—C26—H26 | 127.5 | C36—N24—C35 | 106.9 (5) |
| C25—C26—H26 | 127.5 | C36—N24—H60 | 126.6 |
| N17—C27—N18 | 111.5 (5) | C35—N24—H60 | 126.6 |
| N17—C27—H27 | 124.2 | C39—N25—C37 | 104.4 (5) |
| N18—C27—H27 | 124.2 | C39—N26—C38 | 105.3 (5) |
| C29—C28—N19 | 109.3 (6) | C39—N26—H61 | 127.3 |
| C29—C28—H28 | 125.3 | C38—N26—H61 | 127.3 |
| N19—C28—H28 | 125.3 | C42—N27—C40 | 105.2 (5) |
| N20—C29—C28 | 106.6 (6) | C42—N27—H62 | 127.4 |
| N20—C29—H29 | 126.7 | C40—N27—H62 | 127.4 |
| C28—C29—H29 | 126.7 | C42—N28—C41 | 102.7 (5) |
| N19—C30—N20 | 111.9 (6) | C45—N29—C43 | 104.9 (4) |
| N19—C30—H30 | 124.0 | C45—N30—C44 | 107.0 (5) |
| N20—C30—H30 | 124.0 | C45—N30—H63 | 126.5 |
| C32—C31—N21 | 112.4 (5) | C44—N30—H63 | 126.5 |
| C32—C31—H31 | 123.8 | C48—N31—C47 | 108.0 (5) |
| N21—C31—H31 | 123.8 | C48—N31—H64 | 126.0 |
| C31—C32—N22 | 104.1 (5) | C47—N31—H64 | 126.0 |
| C31—C32—H32 | 128.0 | C48—N32—C46 | 104.3 (5) |
| N22—C32—H32 | 128.0 | O4—S1—O3 | 112.3 (3) |
| N21—C33—N22 | 109.4 (5) | O4—S1—O2 | 109.2 (3) |
| N21—C33—H33 | 125.3 | O3—S1—O2 | 106.9 (3) |
| N22—C33—H33 | 125.3 | O4—S1—O1 | 105.9 (2) |
| C35—C34—N23 | 110.0 (5) | O3—S1—O1 | 111.7 (2) |
| C35—C34—H34 | 125.0 | O2—S1—O1 | 110.9 (2) |
| N23—C34—H34 | 125.0 | O7—S2—O8 | 110.4 (3) |
| C34—C35—N24 | 106.6 (6) | O7—S2—O5 | 108.7 (3) |
| C34—C35—H35 | 126.7 | O8—S2—O5 | 109.9 (2) |
| N24—C35—H35 | 126.7 | O7—S2—O6 | 109.2 (3) |
| N23—C36—N24 | 111.8 (5) | O8—S2—O6 | 108.1 (2) |
| N23—C36—H36 | 124.1 | O5—S2—O6 | 110.5 (2) |
| N24—C36—H36 | 124.1 | N5—Fe1—N3 | 91.34 (16) |
| C38—C37—N25 | 108.5 (5) | N5—Fe1—N9 | 89.46 (16) |
| C38—C37—H37 | 125.7 | N3—Fe1—N9 | 179.19 (16) |
| N25—C37—H37 | 125.7 | N5—Fe1—N11 | 177.69 (15) |

| | | | |
|---|---|---|---|
| C37—C38—N26 | 108.6 (5) | N3—Fe1—N11 | 89.94 (16) |
| C37—C38—H38 | 125.7 | N9—Fe1—N11 | 89.25 (16) |
| N26—C38—H38 | 125.7 | N5—Fe1—N7 | 91.76 (15) |
| N25—C39—N26 | 113.1 (5) | N3—Fe1—N7 | 89.64 (15) |
| N25—C39—H39 | 123.4 | N9—Fe1—N7 | 90.47 (14) |
| N26—C39—H39 | 123.4 | N11—Fe1—N7 | 90.17 (14) |
| C41—C40—N27 | 107.5 (5) | N5—Fe1—N1 | 89.24 (15) |
| C41—C40—H40 | 126.3 | N3—Fe1—N1 | 91.41 (15) |
| N27—C40—H40 | 126.3 | N9—Fe1—N1 | 88.46 (15) |
| C40—C41—N28 | 110.2 (5) | N11—Fe1—N1 | 88.80 (14) |
| C40—C41—H41 | 124.9 | N7—Fe1—N1 | 178.53 (15) |
| N28—C41—H41 | 124.9 | N13—Fe2—N15 | 88.93 (15) |
| N28—C42—N27 | 114.4 (5) | N13—Fe2—N21 | 89.99 (15) |
| N28—C42—H42 | 122.8 | N15—Fe2—N21 | 177.53 (15) |
| N27—C42—H42 | 122.8 | N13—Fe2—N19 | 178.24 (18) |
| C44—C43—N29 | 109.6 (5) | N15—Fe2—N19 | 91.41 (16) |
| C44—C43—H43 | 125.2 | N21—Fe2—N19 | 89.74 (15) |
| N29—C43—H43 | 125.2 | N13—Fe2—N23 | 91.88 (15) |
| C43—C44—N30 | 107.2 (5) | N15—Fe2—N23 | 88.94 (15) |
| C43—C44—H44 | 126.4 | N21—Fe2—N23 | 88.88 (16) |
| N30—C44—H44 | 126.4 | N19—Fe2—N23 | 89.85 (16) |
| N29—C45—N30 | 111.3 (5) | N13—Fe2—N17 | 89.60 (15) |
| N29—C45—H45 | 124.3 | N15—Fe2—N17 | 89.37 (16) |
| N30—C45—H45 | 124.3 | N21—Fe2—N17 | 92.84 (15) |
| C47—C46—N32 | 110.8 (5) | N19—Fe2—N17 | 88.68 (16) |
| C47—C46—H46 | 124.6 | N23—Fe2—N17 | 177.73 (16) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H49···O4 | 0.88 | 1.84 | 2.679 (5) | 160 |
| N4—H50···O5 | 0.88 | 1.89 | 2.762 (5) | 170 |
| N6—H51···O3$^{i}$ | 0.88 | 2.09 | 2.949 (7) | 163 |
| N8—H52···O1$^{ii}$ | 0.88 | 1.94 | 2.822 (5) | 176 |
| N10—H53···O7$^{iii}$ | 0.88 | 1.85 | 2.711 (6) | 166 |
| N12—H54···O7$^{iv}$ | 0.88 | 2.10 | 2.867 (7) | 145 |
| N12—H54···O5$^{iv}$ | 0.88 | 2.61 | 3.434 (7) | 157 |
| N14—H55···O8$^{v}$ | 0.88 | 1.85 | 2.716 (5) | 167 |
| N16—H56···O1$^{vi}$ | 0.88 | 2.22 | 3.074 (6) | 163 |
| N16—H56···O4$^{vi}$ | 0.88 | 2.26 | 2.942 (6) | 135 |
| N18—H57···O6$^{vii}$ | 0.88 | 2.07 | 2.937 (6) | 169 |
| N18—H57···O8$^{vii}$ | 0.88 | 2.47 | 3.100 (6) | 129 |
| N20—H58···O6 | 0.88 | 2.21 | 3.071 (6) | 166 |
| N20—H58···O7 | 0.88 | 2.45 | 3.120 (6) | 133 |
| N22—H59···O2 | 0.88 | 2.18 | 2.939 (6) | 144 |
| N22—H59···O3 | 0.88 | 2.37 | 3.186 (6) | 155 |
| N24—H60···O2$^{viii}$ | 0.88 | 1.84 | 2.710 (6) | 168 |

| | | | | |
|---|---|---|---|---|
| N26—H61···N29 | 0.88 | 1.95 | 2.825 (6) | 177 |
| N27—H62···N25 | 0.88 | 2.04 | 2.875 (6) | 158 |
| N30—H63···N32$^{ix}$ | 0.88 | 2.02 | 2.869 (6) | 160 |
| N31—H64···N28 | 0.88 | 1.92 | 2.800 (6) | 174 |

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*; (ii) *x*, *y*+1, *z*; (iii) *x*+1, *y*, *z*; (iv) −*x*+1, −*y*+1, −*z*+1; (v) *x*, *y*−1, *z*; (vi) *x*−1, *y*, *z*; (vii) −*x*+1, −*y*+1, −*z*; (viii) −*x*+1, −*y*, −*z*+1; (ix) *x*, *y*, *z*−1.

**Fig. 1**

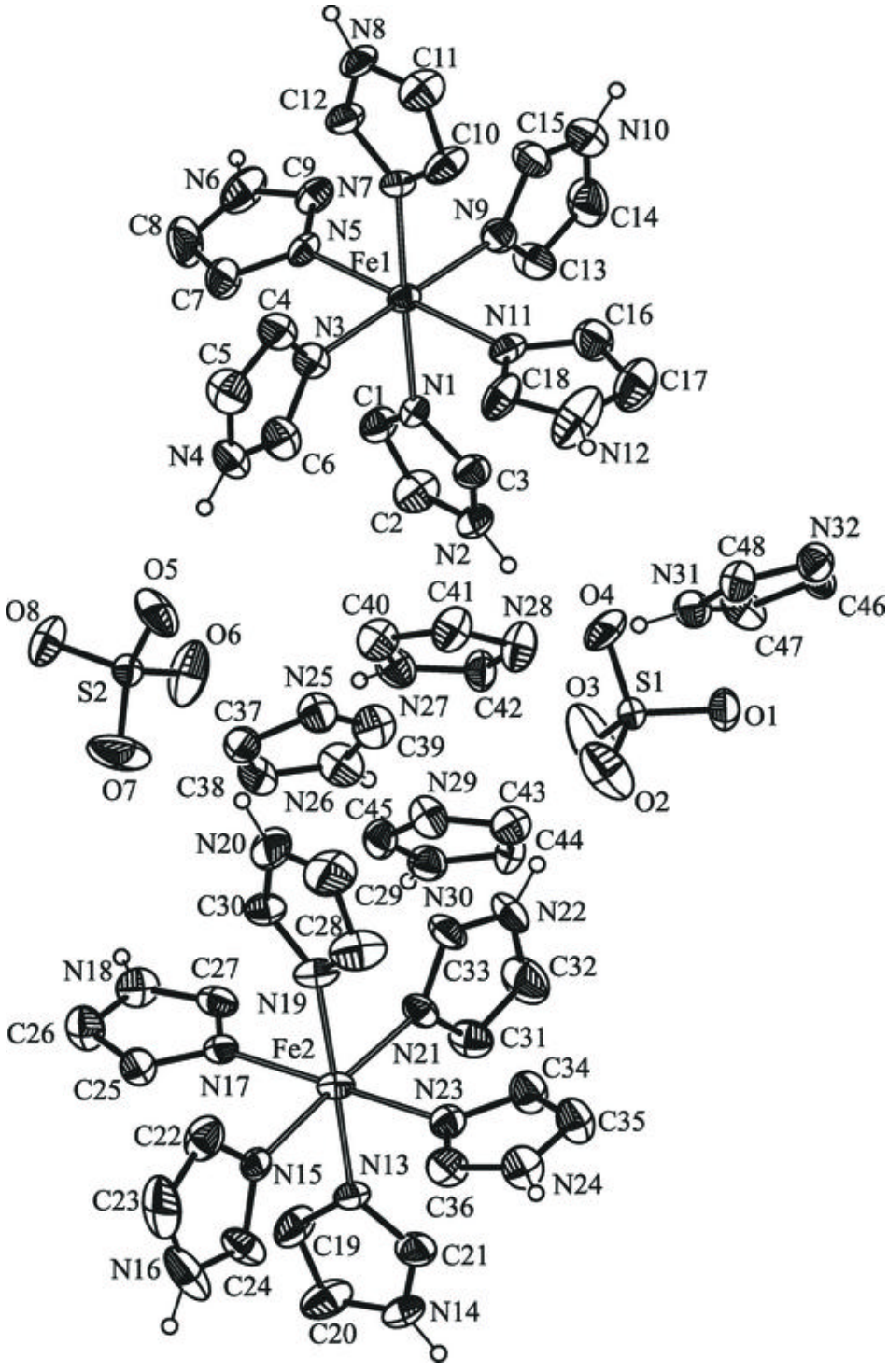
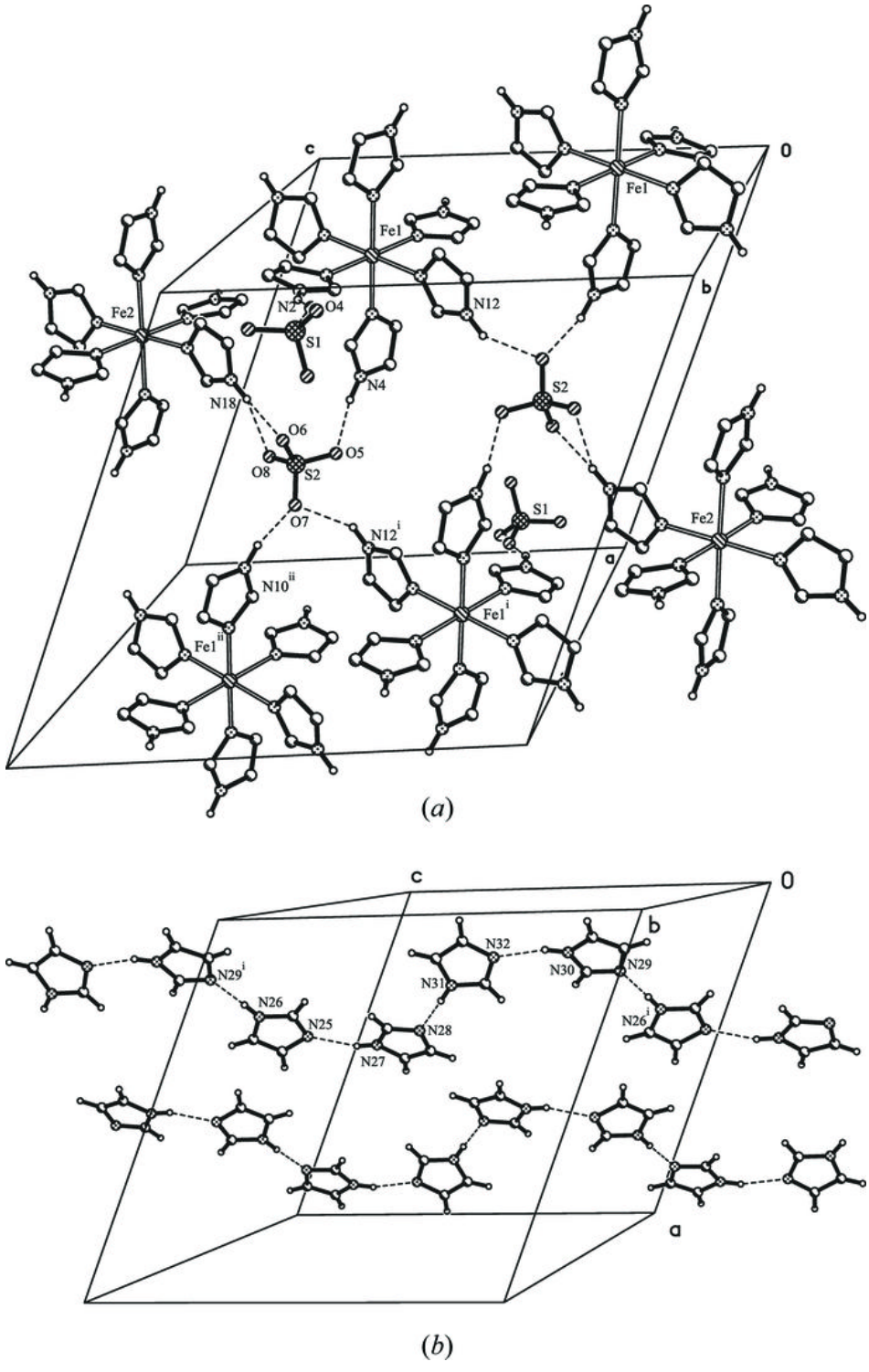

N8
C11
C12
C10
C15
N10
N6
C9
N7
N9
C14
C8
N5
Fe1
C13
C7
C4
N3
N11
C16
C5
N1
C18
C17
C1
N4
C6
N12
C3
C2
N2
N32
C48
O5
N31
O4
C46
O8
C40
C41
N28
O6
C47
S2
N25
O3
S1
C37
N27
C42
O1
C39
O7
C38
N26
C45
N29
C43
O2
N20
C44
C29
N30
C30
N22
C28
N18
C27
C33
C32
N19
C26
Fe2
N21
C31
C25
N17
C34
N23
C35
C22
N15
N13
N24
C23
C36
C19
C21
N16
C24
N14
C20

**Fig. 2**


c
0
b
a
Fe1
Fe2
N2
O4
S1
N12
N4
S2
N18
O6
O5
O8
S2
O7
N12i
S1
Fe2
N10ii
Fe1ii
Fe1i
(a)
c
0
b
a
N29i
N26
N25
N27
N28
N31
N32
N30
N29
N26i
(b)